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Effect of hydrofluoric acid and self-etch ceramic primers on the flexural strength and fatigue resistance of glass ceramics: A systematic review and meta-analysis of *in vitro* studies

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ABSTRACT

This systematic review evaluated the effect of different hydrofluoric acid (HF) etching regimens and a self-etch ceramic primer (SECP) on the flexural strength (FS) and fatigue failure load (FFL) of glass-ceramic materials.The identification of relevant studies was conducted by two authors in five databases: PubMED, Scopus, Web Of Science, LILACS and Virtual Health Library (BVS) until July 2022 with no year limit. The analysis was conducted in RevMan 5.4.1 Software (Cochrane Collaboration) using Random effect model at 5 %. The risk of bias of the included studies were assessed. From the 5349 articles identified, 34 were included for quantitative analysis. Meta-analysis showed that for predominantly glassy ceramics, etching with HF 5 % had no significant impact on FS, however, HF acid etching with concentrations greater than 5 % negatively impacted FS. For lithium disilicate glass-ceramics (LDGC) HF acid etching, negatively influenced FS, while increasing the FFL. HF etching negatively affected FS of hybrid ceramics. The self-etch ceramic primer and HF acid etching showed a similar impact on FFL and FS. This meta-analysis indicates that the impact of SECP and HF acid etching on the mechanical behavior of glass ceramics is material-dependent.

Scientific Field of Dental Science

Dental Materials; Dental restorative materials; Glass ceramics.

1. Introduction

Hydrofluoric acid (HF) etching has become the main method of increasing the surface energy of glass ceramics as it effectively enhances the bond strength of these restorations [1–3]. Hydrofluoric acid reacts with silicon dioxide, which after subsequent reactions, forms fluorosilicic acid, a product soluble in water [4,5]. Therefore, the application of HF in predominantly glassy ceramics can aggressively modify its tridimensional microstructure [6–8]. Cracks introduced by surface treatment methods are perpendicular to the cementation surface where there are the highest tensile stresses when associated with layer flexure. This makes the cementation surfaces highly prone to radial cracks and sensitive to pre-cementation procedures [9–12]. The material's ability to tolerate these defects and arrest cracks under complex stresses of the oral environment relies on the material microstructural features, strengthening mechanisms, and the acid dissolution pattern [4,12–14].

Until now the etching protocols described in the literature were based on the premise that the concentration and time application of HF acid should be proportional to the amount of glassy phase [7,15–17]. This comes from the fact that a great part of reinforcing crystals used in dental glass ceramics are more resistant than the amorphous phase to HF acid. Studies have shown the degree of susceptibility of ceramic components with or without crystallographic orientation varies according to acid concentration, application time, and presence of non-etchable elements [4,7,13,18].

An etching approach based on the ratio of amorphous and crystalline content leads to aggressive etching protocols that excessively modify the internal microstructure of glass ceramics and may not have a significant increase in bond strength that could justify such treatment [18–24]. Furthermore, a greater amount of glass matrix would require a milder etching regimen to increase surface free energy and wettability, thus yielding a suitable surface for the subsequent resin infiltration.

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According to Garfias et al. [6,24] and Murillo-Gómez et al. [7], the HF etching depth can even surpass the thickness of thin laminate restoration, which may influence not only mechanical properties but also optical characteristics [25–27].

Regarding the weakening effect caused by surface modification, the dissolution profile is a more valuable parameter than the extent of dissolution to determine the damaging potential of the introduced flaws. For instance, in two-phase materials whose components have similar solubility to acid, the dissolution pattern tends to be more homogeneous, which may explain the HF acid smoothening or protective effect in some glass-ceramics [13,26,27]. Other authors reported that the protective effect of 10 % HF might be caused by the inability of milder etching regimens of acid etching to soften residual manufacturing flaws that are also thought to be crack initiation sites that lead to slow crack growth and complete failure of the restoration [19,28]. Conversely, ceramic materials reinforced with less soluble interconnected crystalline structures tend to increase the peak-to-valley deviation with the deeper dissolution of the amorphous phase [21,29].

Thus, this systematic review aimed to evaluate the effect of different hydrofluoric acid (HF) etching protocols and a self-etch ceramic primer (SECP) on the flexural strength and fatigue failure load resistance of glass-ceramic materials. The null hypotheses of this review were that: (1) The HF etching would not affect the flexural strength compared to the control group; (2) The HF etching would not affect the fatigue failure resistance compared to the control group; (3) SECP would not affect the flexural strength and fatigue resistance compared to HF.

2. Material and methods

2.1. Protocol and registration

This systematic review was designed according to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) statement [30]. Previously to the database searching, a review protocol was prepared and registered at Open Science Framework (OSF) (registration doi: 10.17605/OSF.IO/ETB9J).

2.2. Sources of information and search strategies

The identification of relevant articles by title and abstract was conducted by two authors (PMM and GLMC) in five databases: PubMed, Scopus, Web of Science, LILACS, and Virtual Health Library (BVS). A manual search was conducted on Google Scholar, but no additional references that met the inclusion and exclusion criteria were found. The search strategy was composed of Medical Subject Headings terms (MeSH) and free keywords arranged by PICO strategy (Table 1). The following PICOT strategy was used: Population - “glass ceramics and hybrid ceramics”; Intervention - “hydrofluoric acid and self-etch ceramic primers”; Comparison - “non-etched glass-ceramics and hybrid ceramics”; Outcome - “flexural strength and fatigue testes”; Type of study: “*in vitro* studies”.

2.3. Eligibility criteria, study selection, and data extraction

The identification of eligible studies for full-text analysis was performed by two researchers (PMM and GLMC) according to the eligible criteria. Conflicted decisions were resolved by a third reviewer (CBA) and discussed for consensus among authors. The inclusion criteria were as follows: *in vitro* studies that assessed the effect of the hydrofluoric acid etch on the flexural strength in non-cemented specimens and the fatigue behavior of glass ceramics as well as hybrid materials (polymer-infiltrated ceramic network materials - PICN and CAD/CAM dispersed filler resin composites - DF-RCBs); fatigue tests with flat or full coverage specimens cemented on dentin analog materials. Regarding flexural strength, the tests considered for this study were the three-point bending test and the biaxial flexural test. The exclusion criteria were: no glass

Table 1
Search strategy for identification of relevant studies.

PubMed	("Ceramics" [MH] OR "Silicates" [MH] OR "Aluminum Silicates" [MH] OR "Aluminum Silicates"OR “Dental Porcelain” [MH] OR “Dental Porcelain” OR “Glass ceramic*” OR “Glass-ceramics” OR “Lithium disilicate” OR “Lithium silicate” OR “Leucite” OR “Feldspathic veneering” OR “Lithium disilicate glass-ceramic” OR “Lithium compound” OR “Ceramics” OR "Silicates" OR “Dental Porcelain” OR “Aluminum Silicates” OR “lithia disilicate” [Supplementary Concept] OR “lithia disilicate” OR “IPS-Empress ceramic” [Supplementary Concept] OR “IPS-Empress ceramic”OR “Cerec” OR “dental ceramic” [TIAB] OR “Veneer*” OR “ceramic veneer*” OR “vita Suprinity” OR “Dental Veneers” [MH]) AND (“Hydrofluoric acid” [MH] OR “Hydrofluoric acid” OR “Surface Treatment” OR “conditioning” OR “acid etching” OR "Dental Acid Etching" OR "Dental Acid Etching" [MH]) AND (“Flexural Strength” [MH] OR “Tensile Strength” [MH] OR "Stress, Mechanical" [MH] OR “Fractures, Stress” [MH] OR “Bending Strength” OR “Mechanical Properties” OR “Tensile Strength” OR “Biaxial flexural strength” OR “flexural strength” [MH] OR “flexural strength” OR “Roughness” OR “Fracture toughness” OR “Resistance values” OR “ceramic fracture” OR “fracture strength” OR "Fatigue" [MH] OR "Fatigue" OR “Weibull Distribution” OR “Weibull Statistics” OR “Weight-Bearing” [MH] OR “Weight-Bearing” OR "Surface Properties" [MH] OR "Surface Properties" OR “Three-Point Flexural Strength” OR "Dental Restoration Failure" [MH] OR "Dental Restoration Failure" OR "Dental Stress Analysis" [MH] OR "Dental Stress Analysis")
SCOPUS	(TITLE-ABS-KEY (ceramics OR silicates OR "Aluminum Silicates" OR "Dental Porcelain" OR "Glass ceramics" OR "Glass ceramic" OR “Glass-ceramic” OR "Lithium disilicate" OR "Lithium silicate" OR leucite OR "Feldspathic" OR "Feldspathic veneering" OR "Lithium disilicate glass-ceramic" OR ceramic OR "Dental Porcelain" OR "Aluminum Silicate" OR "lithia disilicate" OR "IPS-Empress ceramic" OR cerec OR "dental ceramic*" OR veneer* OR "ceramic veneer*" OR "VITA Suprinity" OR "Tooth Crown" OR "crowns") AND TITLE-ABS-KEY ("Hydrofluoric acid" OR "Surface Treatment" OR conditioning OR "acid etching" OR "Dental Acid Etching") AND TITLE-ABS-KEY ("Flexural Strength" OR "Tensile Strength" OR "Stress, Mechanical" OR "Fractures, Stress" OR "Bending Strength" OR "Biaxial flexural strength" OR "flexural strength" OR "Fracture toughness" OR "Resistance values" OR "ceramic fracture" OR "fracture strength" OR "Weibull Distribution" OR "Weibull Statistics" OR "Three-Point Flexural Strength" OR "Weight-Bearing" OR "Fatigue"))
Web of Science	ALL= ("Ceramics" [MH] OR "Computer-Aided Design" [MH] OR "Dental Porcelain"[MH] OR "Crowns" [MH] OR "Dental Prosthesis" [MH] OR "Glass ceramics" OR "Lithium disilicate" OR "Lithium silicate" OR "Lucite" OR "Feldspathic veneering ceramic" OR "Ceramics" OR "Dental Porcelain" OR "Crowns" OR "Dental Prosthesis" OR "Optimal Pressed Ceramic" OR "IPS-Empress ceramic" OR "Cerec" OR "Tooth Crown" OR "crowns" OR “Dental Veneers*” OR “Glass / chemistry*”) AND ALL= ("Hydrofluoric acid" [MH] OR "Surface Treatment" OR "Hydrofluoric acid etching" OR "Surface conditioning" OR "Acid etching" OR "Dental Acid Etching") AND ALL= ("Flexural Strength" [MH] OR "Fatigue" [MH] OR "Dental Stress Analysis" [MH] OR "Surface Properties" [MH] OR "Stress, Mechanical" [MH] OR "Dental Restoration Failure" [MH] OR "Fractures, Stress" [MH] OR "Bending Strength" OR "Mechanical Properties" OR "Tensile Strength" OR "Biaxial flexural strength" OR "Fracture toughness" OR "Weibull Distribution" OR "Weibull Statistics" OR "Three-Point Flexural Strength" OR "Weight-Bearing" OR "Fatigue" OR “Surface properties” OR “Survival Analysis” [MH] OR “Time Factors” [MH] OR “Materials Testing” [MH]) 1# ("Ceramics" [MH] OR "Computer-Aided Design" [MH] OR "Dental Porcelain"[MH] OR "Crowns" [MH] OR "Dental Prosthesis" [MH] OR "Glass ceramics" OR "Lithium disilicate" OR "Lithium silicate" OR "Lucite" OR "Feldspathic veneering ceramic" OR "Ceramics" OR "Dental Porcelain" OR "Crowns" OR "Dental Prosthesis" OR "Optimal Pressed Ceramic" OR "IPS-Empress ceramic" OR "Cerec" OR "Tooth Crown" OR "crowns" OR “Dental Veneers*” OR “Glass / chemistry*”) 2# ("Hydrofluoric acid" [MH] OR "Surface Treatment" OR "Hydrofluoric acid etching" OR "Surface conditioning" OR "Acid etching" OR "Dental Acid Etching") 3#("Flexural Strength" [MH] OR "Fatigue" [MH] OR "Dental Stress Analysis" [MH] OR "Surface Properties" [MH] OR "Stress, Mechanical" [MH] OR "Dental Restoration Failure" [MH] OR "Fractures, Stress" [MH] OR "Bending Strength" OR "Mechanical Properties" OR "Tensile Strength" OR "Biaxial flexural strength" OR "Fracture toughness" OR "Weibull Distribution" OR "Weibull

(*continued on next page*)

Table 1 (*continued*)

	Statistics" OR "Three-Point Flexural Strength" OR "Weight-Bearing" OR "Fatigue" OR "Surface properties"OR "Survival Analysis" [MH] OR "Time Factors" [MH] OR "Materials Testing" [MH])
LILACS	((ceramics OR silicates OR "Aluminum Silicates" OR "Dental Porcelain" OR "Glass ceramics" OR "Glass ceramic" OR "Glass-ceramic" OR "Lithium disilicate" OR "Lithium silicate" OR leucite OR "Feldspathic" OR "Feldspathic veneering" OR "Lithium disilicate glass-ceramic" OR ceramic OR "Dental Porcelain" OR "Aluminum Silicate" OR "lithia disilicate" OR "IPS-Empress ceramic" OR cerec OR "dental ceramic*" OR veneer* OR "ceramic veneer*" OR "VITA Suprinity" OR "Tooth Crown" OR "crowns") AND ("Hydrofluoric acid" OR "Surface Treatment" OR conditioning OR "acid etching" OR "Dental Acid Etching") AND ("Flexural Strength" OR "Tensile Strength" OR "Stress, Mechanical" OR "Fractures, Stress" OR "Bending Strength" OR "Biaxial flexural strength" OR "flexural strength" OR "Fracture toughness" OR "Resistance values" OR "ceramic fracture" OR "fracture strength" OR "Weibull Distribution" OR "Weibull Statistics" OR "Three-Point Flexural Strength" OR "Weight-Bearing" OR "Fatigue"))

ceramics; no control group; no intervention related to hydrofluoric acid etching; fatigue tests in dry conditions; etched experimental groups treated with other surface treatment methods such as sandblasting or silica coating.

Study Information, data for the meta-analysis, and descriptive analysis were extracted by two reviewers (PMM and GLMC). One parameter of the multi-step cyclic fatigue test (fatigue failure load - FFL) and one for the flexural test (flexural strength - MPa) were included in the data extraction form. The fatigue test parameters of the included studies are presented in Table 3.

2.4. Risk of bias assessment

The quality assessment of the included studies comprehended seven parameters: Sample size calculation; Randomization of ceramic specimens; Clear description of specimen preparation and dimensions according to standard rules (ISO); Intervention (hydrofluoric acid or self-etch ceramic primer application protocol); Description of Finishing and polishing protocols; Specimens prepared by a single operator; Description flexural and fatigue test Parameters. For each of them, values were attributed from 0 to 2: 0 (green) if the study was clearly

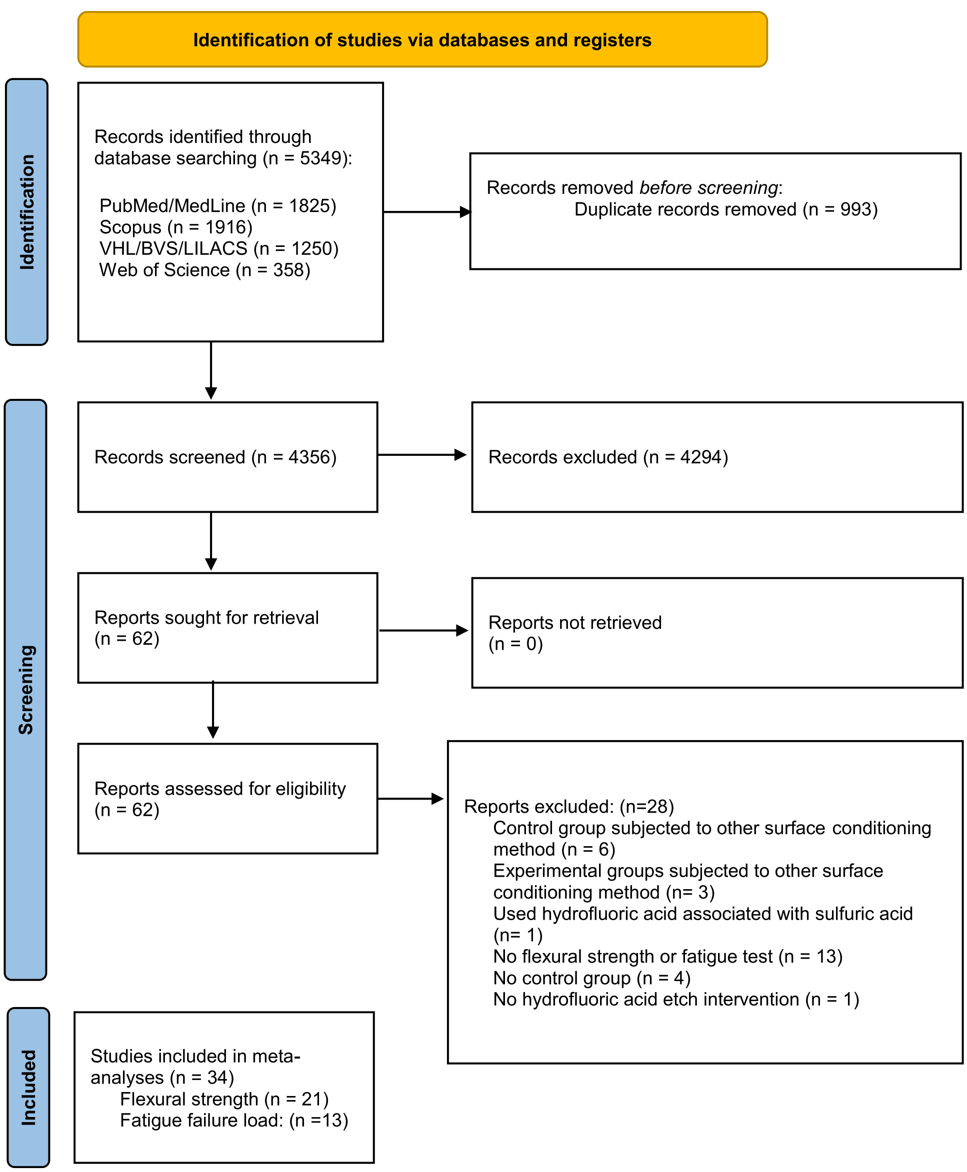

Fig. 1. PRISMA flow-chart.

described; 1 (yellow) if the parameters were described but the execution was unclear; 2 (red) if the parameters were not described. The risk of bias was classified as low if its value ranged from 0 to 4, medium if from 5 to 9, and high from 10 to 14. Each parameter was assessed separately as shown in Fig. 2.

2.5. Data analysis

The analysis was conducted in RevMan 5.4.1 Software (Copenhagen, Nordic Cochrane Center, Cochrane Collaboration) using a random effect model. The heterogeneity of the studies was assessed with the Cochrane Q test ($p < 0.1$) and the inconsistency with the I^2 test indicates that values higher than 50 % have substantial heterogeneity [31]. A significance level was set at 5 % (Z test).

3. Results

3.1. Selection of studies

Through the search strategy used, a total of 5349 articles were identified. After duplicate removal, 4356 articles remained for the title and abstract analysis, of which 4294 articles were excluded, leaving 62 articles for full-text analysis. Thirty-four articles were included for quantitative analysis and 28 were excluded for not meeting the inclusion criteria. The identification of studies is shown in the PRISMA flow diagram (Fig. 1).

For flexural strength, the comparison between hydrofluoric acid etching and the control group was performed for each ceramic material. Flexural strength data were divided according to material composition as follows: feldspathic, leucite, and fluorapatite-based ceramics; lithium disilicate glass-ceramics and hybrid ceramics. Subgroups were defined according to HF acid concentration and application time. A general analysis of all ceramic materials was performed for fatigue failure load. The comparison between self-etch ceramic primer and hydrofluoric acid for flexural strength and fatigue failure load was analyzed separately. Characteristics of included studies that evaluated flexural strength and fatigue are described in Tables 2 and 3, respectively.

3.2. Risk of bias in individual studies

Of the 34 studies included, 22 (64.70 %) showed a low risk of bias, and 12 (35.29 %) showed a medium risk of bias. None of the studies presented a high risk of bias. The least reported parameters in the studies were “sample size calculation” and “specimens prepared by a single operator”.

3.3. Meta-analyses and synthesis of results

3.3.1. Flexural Strength

3.3.1.1. Predominantly glassy ceramics. Seven studies [15,18,26,32–34,56] were analyzed for the flexural strength of feldspathic, leucite, and fluorapatite-based glass ceramics as shown in Fig. 3. The first subgroup, 5 % HF acid etching, did not show a significant difference from the control group ($p = 0.43$; $I^2 = 97$ %). In subgroup 1.1.2, etching with 10 % HF acid for 60 s favored the control group, indicating that HF significantly reduced flexural strength ($p = 0.01$; $I^2 = 91$ %). Etching with 10 % for less than 60 s significantly reduced flexural strength ($p = 0.0002$; $I^2 = 0$ %). However, a single article showed that 10 % HF acid etching for more than 60 s had no significant difference with the control group ($p = 0.06$; $I^2 = 0$ %). A single article showed that etching with 20 % HF significantly reduced flexural strength ($p < 0.0001$; $I^2 = 0$ %). Regarding the robustness of the results, it is essential to emphasize that a single article was included in subgroups 1.1.4 and 1.1.5 to analyze different interventions discretely. Thus, these results

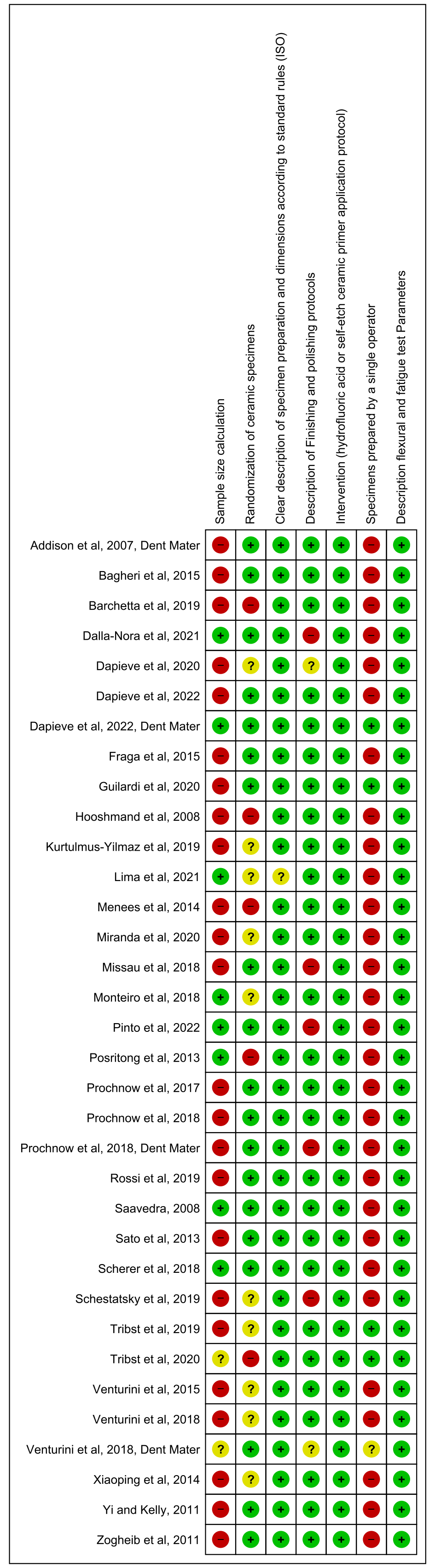

Fig. 2. Risk of bias summary.

Table 2
Characteristics of included studies that evaluated flexural strength.

Study/Y	Material	Brand	Polishing Protocol	Flexural Test	Sample Size	Sample Thickness	HF (%)	Ecthing Time (s)
Venturini et al. [18]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Wet ground with 400-, 600- and 1200-grit SiC paper	Three-point bending test	30	1.2 mm	1 % 3 % 5 % 10 %	60 s
Addison et al. [13]	Feldspathic ceramic	Vitadur Alpha dentinpowder (VITA Zahnfabrik)	Not reported	Biaxial Flexural Test	3	0.9 mm	5 % 10 % 20 %	45 s 90 s 180 s
Bagheri et al. [32]	Lithium disilicate glass-ceramic Feldspathic ceramic	IPS e.max CAD (Ivoclar Vivadent AG) VitaBlocks Mark II (VITA Zahnfabrik)	Wet ground with 800- to 1200-grit SiC paper	Biaxial Flexural Test	15	1.3 mm	9.5 %	20 s 60 s
Yi and Kelly [15]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Wet ground with 600-grit SiC paper	Biaxial Flexural Test	10	1.1-1.2 mm	9 %	60 s
Saavedra [33]	Feldspathic ceramic	VITA VM®7 Feldspathic Ceramics (VITA Zahnfabrik)	Ground with 1000-grit sandpaper	Three-point bending test	10	1.2 mm	10 %	20 s
Posritong et al. [26]	Fluorapatite-based glass-ceramic	IPS e.max ZirPress (Ivoclar Vivadent AG)	Wet ground with 400- and 600-grit SiC paper	Biaxial Flexural Test	24	0.8 ± 0.1 mm	5 %	30 s 60 s 90 s 120 s
Fraga et al. [34]	Leucite-reinforced glass-ceramic	IPS Empress CAD (Ivoclar Vivadent AG)	Ground with 400-, 600- and 1200-grit SiC paper	Biaxial Flexural Test	24	1.3 mm	10 %	60 s
Tribst et al. [35]	Lithium disilicate glass ceramic Polymer-infiltrated ceramic-network Leucite-reinforced glass-ceramic	E.max CAD (Ivoclar Vivadent AG) VITA Enamic (VITA Zahnfabrik) Empress CAD (Ivoclar Vivadent AG)	Ground with 600- and 1200-grit SiC paper	Biaxial Flexural Test	10	1.2 mm	10 % and Monobond Etch & Prime (Ivoclar Vivadent)	60 s
Hooshmand et al. [36]	Leucite-reinforced glass-ceramic Lithium disilicate glass-ceramic	IPS Empress press (Ivoclar Vivadent AG) IPS Empress II press (Ivoclar Vivadent AG)	Wet ground with 400-, 600- and 800-grit SiC paper	Biaxial Flexural Test	10	2 mm	9 %	120 s
Zogheib et al. [37]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Ground with 1000-grit SiC paper	Three-point bending test	15	2 mm	4.9 %	20 s 60 s 90 s 180 s
Xiaoping et al. [21]	Lithium disilicate glass-ceramic	IPS e.max press (Ivoclar Vivadent AG)	Ground with 400-, 600-, and 800-grit SiC paper	Three-point bending test	42	2 mm	9.5 %	20s40s60s 120 s
Prochnow et al. [38]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Ground with 400-, 600-, and 1200-grit SiC paper	Three-point bending test	23	1.2 mm	1 % 3 % 5 % 10 %	20 s
Menees et al. [39]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Ground with 180-, 320-, and 600-grit abrasive paper	Three-point bending test	10	2.5 mm	5 % 9.5 %	20 s 120 s
Rossi et al. [40]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Ground with 280-, 800- and 1200-grit SiC paper	Three-point bending test	10	2 mm	5 %	20 s
Sato et al. [41]	Lithium disilicate glass-ceramic	IPS e.max press (Ivoclar Vivadent AG)	Ground with 800- and 1200-grit diamond papers	Three-point bending test	10	2.2 mm	5 %	20 s
Lima et al. [42]	Lithium disilicate glass-ceramic Zirconia-reinforced lithium silicate Zirconia-reinforced lithium silicate	IPS e.max CAD (Ivoclar Vivadent AG) Vita Suprinity (VITA Zahnfabrik) Celtra Duo (Dentsply Sirona)	Ground with 240-, 400-, 600- and 1200-grit SiC paper	Biaxial Flexural Test	15	1.2 mm	10 % and Monobond Etch & Prime (Ivoclar Vivadent)	20 s
Kurtulmus-Yilmaz et al. [25]	Lithium disilicate glass-ceramic Resin nanoceramic Resin nanoceramic Polymer-infiltrated ceramic	IPS e.max CAD (Ivoclar Vivadent AG) Lava Ultimate (3 M ESPE, St Paul, MN) Cerasmart (GC Dental Products) VITA Enamic (VITA Zahnfabrik)	Wet ground with 600-, 1000- and 1200-grit SiC paper	Three-point bending test	10	1.2 mm	5 %	20 s 60 s

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Table 2 (*continued*)

Study/Y	Material	Brand	Polishing Protocol	Flexural Test	Sample Size	Sample Thickness	HF (%)	Ecthing Time (s)
Miranda et al. [43]	Polymer-infiltrated ceramic-network	Vita Enamic (VITA Zahnfabrik)	Wet ground with 400-, 600-, and 1200-grit SiC paper	Biaxial Flexural Test	13	1.2 mm	5 % 10 %	30 s 60 s 90 s
Barchetta et al. [28]	Zirconia-reinforced lithium silicate	Vita Suprinity (VITA Zahnfabrik)	Wet ground with 600-, 800-, and 1200-grit SiC paper	Biaxial Flexural Test	15	1.2 mm	10 %	20 s 40 s 60 s
Pinto et al. [44]	Polymer-infiltrated ceramic-network Resin nanoceramic	Vita Enamic (VITA Zahnfabrik) Lava Ultimate (3 M ESPE, St Paul, MN)	Wet ground with 600-, 800-, and 1200-grit SiC paper	Biaxial Flexural Test	15	1.2 mm	10 % and Monobond Etch & Prime (Ivoclar Vivadent)	60 s

Table 3
Characteristics of included studies that evaluated the fatigue failure load.

Study/Y	Material	Brand	Polishing Protocol	Sample Size	HF (%)	Ecthing Time (s)	FFL Test Parameters
Venturini et al. [45]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Ground with 600-grit SiC paper	20	1 % HF 5 % HF 10 % HF	60 s	500,000 cycles at 20 Hz; Initial load = 180 N; Step size = 20 N; Under water
Venturini et al. [46]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Ground with 600-grit SiC paper	20	1 % HF 5 % HF 10 % HF	60 s	500,000 cycles at 20 Hz; Initial load = 290 N; Step size = 30 N; Under water
Missau et al. [47]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Not reported	20	1 % HF 5 % HF 10 % HF	60 s	100,000 cycles at 10 Hz; Initial load = 585,5 N; Step size = 58.5 N; Under water
Guilardi et al. [48]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Ground with 400-,600-, 800-,1200- and 2000-grit SiC paper	5	10 % HF	60 s	250,000 cycles at 20 Hz; Initil load = 505 N; Step size = 25 N; Under water
Yi and Kelly [15]	Feldspathic ceramic	VitaBlocks Mark II for CEREC/inLab (VITA Zahnfabrik)	Wet ground with 600-grit SiC paper	10	9 % HF	60 s	500,000 cycles at 15 Hz; Initial load = 230 N; Step size = 20 N; Under water
Dapieve et al. [49]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Wet ground with 400-, 600- and 1200-grit SiC paper	15	5 % HF	20 s	10,000 cycles at 20 Hz; Initial load = 200 N; Step size = 50 N; Under water
Prochnow et al. [29]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Wet ground with 600- and 1200-grit SiC paper	20	3 % HF 5 % HF 10 % HF	20 s	500,000 cycles at 20 Hz; Initial load = 720 N; Step size = 70 N; Under water
Schestatsky et al. [50]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Not reported	10	5 % HF and Monobond Etch & Prime (Ivoclar Vivadent)	20 s	15,000 cycles at 20 Hz; Initial load = 400 N; Step size = 100 N; Under water
Prochnow et al. [51]	Lithium disilicate glass-ceramic	IPS e.Max CAD, (Ivoclar Vivadent AG)	Not reported	18	3 % HF 5 % HF 10 % HF	20 s	500,000 cycles at 20 Hz; Initial load = 720 N; Step size = 70 N; Under water
Scherer et al. [52]	Lithium disilicate glass-ceramic	IPS e.Max CAD, (Ivoclar Vivadent AG)	Ground with 120-, 400- and 1200-grit SiC paper	15	5 % HF and Monobond Etch & Prime (Ivoclar Vivadent)	20 s	250,000 cycles at 20 Hz; Initial load = 1050 N; Step size = 52,5 N;
Dapieve et al. [53]	Lithium disilicate glass-ceramic	IPS e.Max CAD, (Ivoclar Vivadent AG)	Ground with 120-, 400- and 1200-grit SiC paper	15	5 % HF and Monobond Etch & Prime (Ivoclar Vivadent)	20 s	10,000 cycles at 20 Hz; Initial load = 200 N; Step size = 50 N; Under water
Tribst et al. [54]	Lithium disilicate glass-ceramic	IPS e.max CAD (Ivoclar Vivadent AG)	Ground with 600- and 1200-grit SiC paper	20	10 % HF	20 s	100,000 cycles at 20 Hz; Initial load = 1435 N; Step size = 72 N
Monteiro et al. [19]	Zirconia-reinforced lithium silicate glass	Vita Suprinity (VITA Zahnfabrik)	Ground with 600-, 800- and 1200-grit SiC paper	15	5 % HF 10 % HF	30 s 60 s 90 s	500,000 cycles at 20 Hz; Initial load = 925 N; Step size = 45 N; Under water.
Dapieve et al. [55]	Lithium disilicate glass-ceramic	IPS e.Max CAD (Ivoclar Vivadent AG)	Ground with 1200- and 2400-grit SiC paper	10	10 % HF and Monobond Etch & Prime (Ivoclar Vivadent)	60 s	10,000 cycles/step at 20 Hz; initial load of 200 N, Step-size = 50 N; Under water.
Dalla-Nora et al. [27]	Zirconia-reinforced lithium silicate	Vita Suprinity (VITA Zahnfabrik)	Wet ground with 400-, 600- and 1200-grit SiC paper	15	5 % HF and Monobond Etch & Prime (Ivoclar Vivadent)	20 s	10,000 cycles/step at 20 Hz; Initial load = 200 N, Step-size = 100 N; Under water.

Study or Subgroup	Control Mean [MPa]	Control SD [MPa]	Control Total	Hydrofluoric acid etch Mean [MPa]	Hydrofluoric acid etch SD [MPa]	Hydrofluoric acid etch Total	Weight	Mean Difference IV, Random, 95% CI
1.1.1 5%								
Addison et al, 2007 5% for 45s Felds	94.4	9.9	3	83.4	11.4	3	4.8%	11.00 [-6.09, 28.09]
Venturini et al., 2015 HF5% 60 s Felds	143.3	12.1	30	102.02	5.3	30	6.0%	41.28 [36.55, 46.01]
Addison et al, 2007 5% for 90s Felds	94.4	9.9	3	83.1	12.3	3	4.7%	11.30 [-6.57, 29.17]
Addison et al, 2007 5% for 180s Felds	94.4	9.9	3	83.6	14.2	3	4.5%	10.80 [-8.79, 30.39]
Posritong et al,2013 5% for 30s Fluo	98.4	14.9	24	98.4	8	24	5.8%	0.00 [-6.77, 6.77]
Posritong et al,2013 5% for 60s Fluo	98.4	14.9	24	103.6	12	24	5.8%	-5.20 [-12.85, 2.45]
Posritong et al,2013 5% for 90s Fluo	98.4	14.9	24	106.8	21.7	24	5.5%	-8.40 [-18.93, 2.13]
Posritong et al,2013 5% for 120s Fluo	98.4	14	24	103.4	17.9	24	5.7%	-5.00 [-14.09, 4.09]
Subtotal (95% CI)			**135**			**135**	**42.8%**	**6.93 [-10.37, 24.23]**
Heterogeneity: Tau² = 582.35; Chi² = 202.47, df = 7 (P < 0.00001); I² = 97%								
Test for overall effect: Z = 0.79 (P = 0.43)								
1.1.2 10% for 60s								
Fraga et al, 2015 HF10% for 60s Leu	177.2	26.5238	24	169.1	17.7614	24	5.3%	8.10 [-4.67, 20.87]
Bagheri et. al, 2015 HF9,5% for 60s Felds	68.52	11.36	15	57.2	4.54	15	5.9%	11.32 [5.13, 17.51]
Venturini et al., 2015 HF10% 60 s Felds	143.3	12.1	10	102.64	8.7	10	5.6%	40.66 [31.42, 49.90]
Yi-J and Kelly, 2011 HF9% for 60s Felds	91.59	11.48	10	81.36	4.45	10	5.8%	10.23 [2.60, 17.86]
Subtotal (95% CI)			**59**			**59**	**22.6%**	**17.63 [3.50, 31.75]**
Heterogeneity: Tau² = 186.01; Chi² = 33.22, df = 3 (P < 0.00001); I² = 91%								
Test for overall effect: Z = 2.45 (P = 0.01)								
1.1.3 10% for less than 60s								
Saavedra, 2008 HF%10 for 20s Felds	73.79	5.83	10	60.65	10.07	10	5.8%	13.14 [5.93, 20.35]
Addison et al, 2007 10% for 45s Felds	94.4	9.9	3	84.9	13.8	3	4.6%	9.50 [-9.72, 28.72]
Subtotal (95% CI)			**13**			**13**	**10.4%**	**12.69 [5.94, 19.44]**
Heterogeneity: Tau² = 0.00; Chi² = 0.12, df = 1 (P = 0.73); I² = 0%								
Test for overall effect: Z = 3.68 (P = 0.0002)								
1.1.4 10% for more than 60s								
Addison et al, 2007 10% por 90s Felds	94.4	9.9	3	82.2	10	3	5.0%	12.20 [-3.72, 28.12]
Addison et al, 2007 10% por 180s Felds	94.4	9.9	3	83.3	13.9	3	4.6%	11.10 [-8.21, 30.41]
Subtotal (95% CI)			**6**			**6**	**9.5%**	**11.75 [-0.53, 24.04]**
Heterogeneity: Tau² = 0.00; Chi² = 0.01, df = 1 (P = 0.93); I² = 0%								
Test for overall effect: Z = 1.88 (P = 0.06)								
1.1.5 20%								
Addison et al, 2007 20% por 45s Felds	94.4	9.9	3	72.9	11.2	3	4.8%	21.50 [4.58, 38.42]
Addison et al, 2007 20% for 90s Felds	94.4	9.9	3	75.8	7.8	3	5.1%	18.60 [4.34, 32.86]
Addison et al, 2007 20% for 180s Felds	94.4	9.9	3	79.2	12.8	3	4.7%	15.20 [-3.11, 33.51]
Subtotal (95% CI)			**9**			**9**	**14.7%**	**18.60 [9.23, 27.97]**
Heterogeneity: Tau² = 0.00; Chi² = 0.25, df = 2 (P = 0.88); I² = 0%								
Test for overall effect: Z = 3.89 (P < 0.0001)								
Total (95% CI)			**222**			**222**	**100.0%**	**11.96 [3.66, 20.26]**
Heterogeneity: Tau² = 295.53; Chi² = 237.69, df = 18 (P < 0.00001); I² = 92%								
Test for overall effect: Z = 2.83 (P = 0.005)								
Test for subgroup differences: Chi² = 2.09, df = 4 (P = 0.72), I² = 0%								

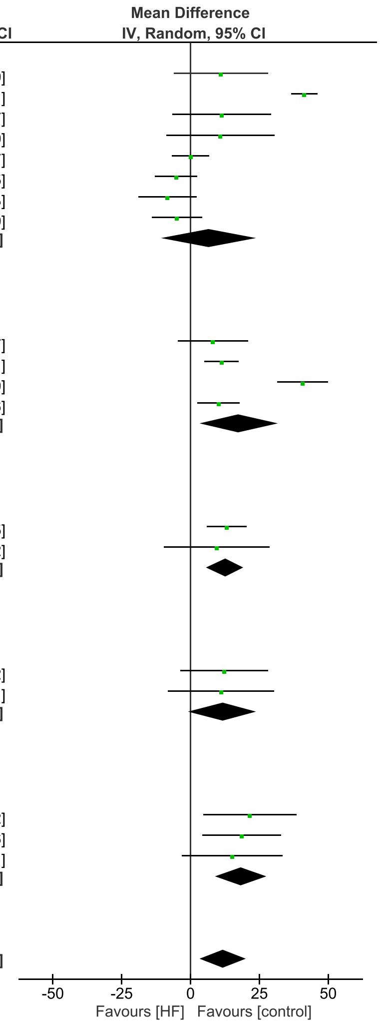

Fig. 3. Subgroup analysis comparing flexural strength of hydrofluoric acid etching and control of predominantly glassy ceramics. Felds: feldspathic glass-ceramic; Fluo: fluorapatite-based glass-ceramic; Leu: leucite-based glass-ceramic.

should be interpreted with caution. Overall results indicate a negative impact of hydrofluoric acid on the flexural strength of predominantly glassy ceramics.

3.3.1.2. Lithium disilicate glass-ceramic. For lithium disilicate glass-ceramic, 8 articles [21,25,29,39–41,37,57] were included in the meta-analysis (Fig. 4). The global result indicates a negative effect of HF acid etching ($p < 0,00001$) with substantial heterogeneity. In some subgroups, a single article was included (1.2.2 and 1.2.6) to analyze the effect of each application time and HF concentration separately. Therefore, this must be considered when interpreting the results.

3.3.1.3. Hybrid ceramics. Four studies that assessed PICN or/and DF-RCB materials were included in a meta-analysis [25,43,58,44]. As shown in Fig. 5, the application of HF significantly reduced the flexural strength of these materials ($p < 0,00001$) with substantial heterogeneity (I^2 =89 %). It is worth mentioning that 10 % HF provided the lowest values of FS.

3.3.1.4. SECP and HF. Three studies compared FFL between groups etched with SECP and HF [44,42,35]. When HF acid etching was compared with SECP, there was no significant difference in the flexural strength ($p = 0.34$) (Fig. 6).

3.3.2. Fatigue Failure Load

3.3.2.1. Glass-ceramic materials. The meta-analysis for fatigue failure load included eleven studies [15,19,29,38,48,53,52,59,45,51,46]. Lithium disilicate glass-ceramic showed favorable results for the etched group ($p = 0.00001$; i2 = 88 %). In one article the HF acid etching of zirconia-reinforced lithium silicate (ZLS) favored the etched group (Fig. 7). For feldspathic glass ceramic, HF acid etching favored the control group ($p = 0.02$; $I^2 = 96$ %). Regarding global result, acid application increased the fatigue failure load of glass ceramics ($p < 0.00001$; I^2 =100 %).

3.3.2.2. Self-etch ceramic primer. The comparison between SECP and HF for fatigue failure load six articles [27,53,52,55,50,54] were included and presented no significant difference ($p = 0.56$) (Fig. 8).

4. Discussion

This systematic review assessed the effect of HF application regimen on the flexural strength and fatigue failure load of glass ceramics. Since the overall analysis showed a significant influence of hydrofluoric acid application on the flexural strength and fatigue failure load on the materials included in this study, the first two null hypotheses were rejected. Whereas no significant difference was found between HF acid and SECP,

Study or Subgroup	Control Mean [MPa]	Control SD [MPa]	Control Total	Hydrofluoric acid etch Mean [MPa]	Hydrofluoric acid etch SD [MPa]	Hydrofluoric acid etch Total	Weight	Mean Difference IV, Random, 95% CI
1.2.1 5% for 20s								
Kurtulmus-Yilmaz et al, 2019 5% 20s LD CAD	218.1	13.8	10	184.3	9.5	10	9.9%	33.80 [23.42, 44.18]
Menees et al., 2014 5% 20s LD CAD	376.5	79.13	10	343.7	65.94	10	3.0%	32.80 [-31.04, 96.64]
Prochnow et al, 2017 5% 20s LD Press	321.88	57.6	23	308.36	59.1	23	6.1%	13.52 [-20.21, 47.25]
Rossi et al, 2019 5% 20s LD CAD	318.11	56.63	10	298.69	37.02	10	5.0%	19.42 [-22.51, 61.35]
Sato et al, 2013 5% 20s LD Press	25.672	71.32	10	2.648	33.99	10	4.2%	23.02 [-25.94, 71.99]
Zogheib et al, 2011 4,9% 20s LD CAD	417	55	15	367	68	15	4.7%	50.00 [5.74, 94.26]
Subtotal (95% CI)			**78**			**78**	**32.9%**	**31.91 [22.74, 41.08]**
Heterogeneity: Tau² = 0.00; Chi² = 2.38, df = 5 (P = 0.79); I² = 0%								
Test for overall effect: Z = 6.82 (P < 0.00001)								
1.2.2 5% for 60s								
Zogheib et al, 2011 4,9% 60s LD CAD	417	55	15	363	84	15	4.0%	54.00 [3.19, 104.81]
Subtotal (95% CI)			**15**			**15**	**4.0%**	**54.00 [3.19, 104.81]**
Heterogeneity: Not applicable								
Test for overall effect: Z = 2.08 (P = 0.04)								
1.2.3 5% for 120								
Menees et al., 2014 5% 120s LD CAD	376.5	79.13	10	333.8	49.16	10	3.4%	42.70 [-15.04, 100.44]
Zogheib et al, 2011 4,9% 180s LD CAD	417	55	15	314	62	15	5.0%	103.00 [61.06, 144.94]
Subtotal (95% CI)			**25**			**25**	**8.4%**	**76.25 [17.53, 134.97]**
Heterogeneity: Tau² = 1155.17; Chi² = 2.74, df = 1 (P = 0.10); I² = 64%								
Test for overall effect: Z = 2.55 (P = 0.01)								
1.2.4 10% for 20s								
Bagheri et. al, 2015 9,5% 20s LD CAD	89.56	12.77	15	84.39	19.74	15	9.7%	5.17 [-6.73, 17.07]
Menees et al., 2014 9,5% 20s LD CAD	376.5	79.13	10	304.5	43.16	10	3.6%	72.00 [16.13, 127.87]
Prochnow et al, 2017 10% 20s LD Press	321.88	57.6	23	291.48	40.7	23	6.9%	30.40 [1.58, 59.22]
Xiaoping et al., 2014 9.5% 20s LD Press	384	33	42	347	43	42	9.0%	37.00 [20.61, 53.39]
Subtotal (95% CI)			**90**			**90**	**29.2%**	**29.07 [5.31, 52.82]**
Heterogeneity: Tau² = 404.37; Chi² = 13.88, df = 3 (P = 0.003); I² = 78%								
Test for overall effect: Z = 2.40 (P = 0.02)								
1.2.5 10% for 120s								
Hooshmand et. al, 2008 9% 120s LD Press	283.97	48.52	10	250.56	34.61	10	5.7%	33.41 [-3.53, 70.35]
Menees et al., 2014 9,5% 120s LD CAD	376.5	79.13	10	353.5	69.54	10	2.9%	23.00 [-42.29, 88.29]
Xiaoping et al, 2014 9.5% 120s LD Press	384	33	42	317	41	42	9.1%	67.00 [51.08, 82.92]
Subtotal (95% CI)			**62**			**62**	**17.6%**	**50.19 [21.95, 78.43]**
Heterogeneity: Tau² = 315.28; Chi² = 3.97, df = 2 (P = 0.14); I² = 50%								
Test for overall effect: Z = 3.48 (P = 0.0005)								
1.2.6 10% for 60s								
Xiaoping et al., 2014 9.5% 60s LD Press	384	33	42	327	67	42	8.0%	57.00 [34.41, 79.59]
Subtotal (95% CI)			**42**			**42**	**8.0%**	**57.00 [34.41, 79.59]**
Heterogeneity: Not applicable								
Test for overall effect: Z = 4.95 (P < 0.00001)								
Total (95% CI)			**312**			**312**	**100.0%**	**39.86 [26.94, 52.79]**
Heterogeneity: Tau² = 412.79; Chi² = 58.77, df = 16 (P < 0.00001); I² = 73%								
Test for overall effect: Z = 6.04 (P < 0.00001)								
Test for subgroup differences: Chi² = 7.61, df = 5 (P = 0.18), I² = 34.3%								

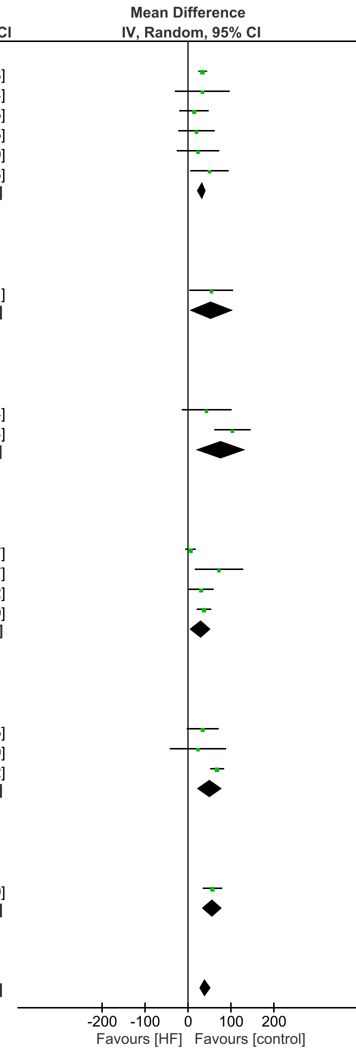

Fig. 4. Subgroup analysis for flexural strength between hydrofluoric acid etching and control of lithium disilicate glass-ceramics (LD).

Study or Subgroup	Control Mean [MPa]	Control SD [MPa]	Control Total	Hydrofluoric acid etch Mean [MPa]	Hydrofluoric acid etch SD [MPa]	Hydrofluoric acid etch Total	Weight	Mean Difference IV, Random, 95% CI
1.3.1 General analysis								
Miranda et al, 2020 5% 30s PICN	129.1	12.29	13	120.68	11.59	13	12.2%	8.42 [-0.76, 17.60]
Miranda et al, 2020 5% 60s PICN	129.1	12.29	13	123.39	12.73	13	12.0%	5.71 [-3.91, 15.33]
Kurtulmus-Yilmaz et al, 2019 5% 60s DF-RCB	125.9	10.2	10	87.66	7.8	10	12.6%	38.24 [30.28, 46.20]
Kurtulmus-Yilmaz et al, 2019 5% 60s PICN	116.4	9.5	10	107.6	8	10	12.7%	8.80 [1.10, 16.50]
Miranda et al, 2020 5% 90s PICN	129.1	12.29	13	118.92	10.53	13	12.3%	10.18 [1.38, 18.98]
Miranda et al, 2020 10% 30s PICN	129.1	12.29	13	108.71	4.86	13	12.9%	20.39 [13.21, 27.57]
Miranda et al, 2020 10% 60s PICN	129.1	12.29	13	102.94	9.61	13	12.4%	26.16 [17.68, 34.64]
Miranda et al, 2020 10% 90s PICN	129.1	12.29	13	96.02	4.12	13	12.9%	33.08 [26.03, 40.13]
Subtotal (95% CI)			**98**			**98**	**100.0%**	**19.05 [10.41, 27.68]**
Heterogeneity: Tau² = 137.60; Chi² = 62.92, df = 7 (P < 0.00001); I² = 89%								
Test for overall effect: Z = 4.32 (P < 0.0001)								
Total (95% CI)			**98**			**98**	**100.0%**	**19.05 [10.41, 27.68]**
Heterogeneity: Tau² = 137.60; Chi² = 62.92, df = 7 (P < 0.00001); I² = 89%								
Test for overall effect: Z = 4.32 (P < 0.0001)								
Test for subgroup differences: Not applicable								

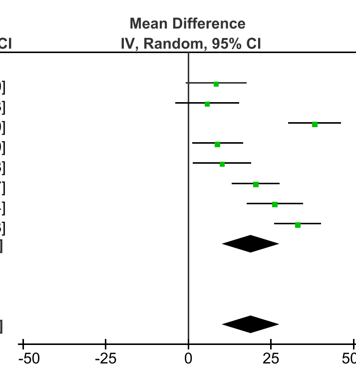

Fig. 5. Forest plot for the analysis of flexural strength between hydrofluoric acid etching and control of hybrid ceramics.

therefore the third null hypothesis was accepted. When interpreting the results presented in this study, it's crucial to note that this systematic review exclusively encompasses *in vitro* studies. Therefore, caution must be taken to avoid extrapolating these findings to directly guide clinical decisions.

The oral environment imposes a great challenge for laboratory testing as it promotes a complex combination of factors such as temperature variation, humidity, restoration design, and cyclic loads that contribute to the damage accumulation process [60]. Different flexural testing methods can provide reliable data regarding the mechanical characterization of brittle materials, but as others, fast fracture monotonic tests are unable to assess strength degradation and simulate oral environment conditions [60,61]. Therefore, cyclic methods, such as Boundary, staircase technique, and step-stress, can precisely predict the

Study or Subgroup	Self-etch ceramic primer Mean [MPa]	SD [MPa]	Total	Hydrofluoric acid etch Mean [MPa]	SD [MPa]	Total	Weight	Mean Difference IV, Random, 95% CI
Lima et al, 2021 10% 20 ZLS CAD (Suprinity)	207.45	28.63	15	269.58	27.07	15	15.5%	-62.13 [-82.07, -42.19]
Lima et al, 2021 10% 20s ZLS CAD (Celtra Duo)	165.41	33.86	15	195.51	42.12	15	14.4%	-30.10 [-57.45, -2.75]
Lima et al, 2021 10% 20s LD CAD	298	53.29	15	289.3	40.11	15	13.4%	8.70 [-25.05, 42.45]
Tribst et al, 2020 10% 60s LD CAD	445.3	39	10	490.3	19	10	14.5%	-45.00 [-71.89, -18.11]
Tribst et al, 2020 10% 60s Leu CAD	439.3	29	10	436.8	21	10	15.2%	2.50 [-19.69, 24.69]
Pinto et al, 2022 10% 60s PICN	144	15.1	15	130.87	13.61	15	16.5%	13.13 [2.84, 23.42]
Tribst et al, 2020 10% 60s PICN	459.8	68	10	424.3	48	10	10.5%	35.50 [-16.09, 87.09]
Total (95% CI)			**90**			**90**	**100.0%**	**-13.06 [-40.11, 13.98]**

Heterogeneity: Tau² = 1125.23; Chi² = 58.57, df = 6 (P < 0.00001); I² = 90%
Test for overall effect: Z = 0.95 (P = 0.34)

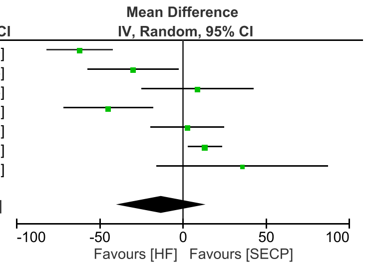

Fig. 6. Forest plot for the analysis of flexural strength between hydrofluoric acid etching and self-etch ceramic primer (SECP).

failure of glass ceramics and assess forms of long-term degradation in service conditions [62–64].

4.1. Predominantly glassy ceramics

As shown in Fig. 3, there was a significant reduction in FS of predominantly glassy ceramics when 10 % HF was used. This may be explained by the preferential increase of pre-existing flaws when 10 % HF acid is used for 60 s, as reported by Addison et al. [13] and Della Bona et al. [65]. In contrast, an increase in application time for more than 60 s has not reduced FS which suggests a polishing effect that brought the reduction of high amplitude defects or stabilization of preexisting sharp cracks by blunting [56,46,66]. It is well known that the distribution, size, and geometry of reinforcing crystals have a detrimental impact on the etching pattern and the formation of potential crack initiation sites. In conventional feldspathic ceramics, it has been shown that leucite crystals are preferably dissolved relative to the amorphous phase due to its high silica content, and the absence of interlocking between fine leucite crystals [12,56,65]. However, dissolution selectivity depends upon acid concentration and application time, thus resulting in a wide variety of topographic changes, yielding partial or even complete dissolution of leucite crystals [12,13,67,68].

It has been demonstrated that the weakening or strengthening effect of HF acid etching can happen synchronically [17,56,69] while preexisting defects are blunted [18,46,70], new voids are introduced by complete dislodgements of leucite crystals or other aluminum-silicate crystals [7]. In this sense, an inadequately polished control group impedes a precise assessment of the HF effect on roughness profile and mechanical behavior, as HF application for 20 s is unable to remove remaining milling scratches [28] and manufacturing defects that can reduce flexural strength of glass ceramics [34,71].

The damaging effect of preferential crystal dissolution or crystal pullout on the formation of initial flaws tends to be aggravated in the presence of larger sharp leucite non-interlocked crystals or clusters that can be found in some of the conventional feldspathic ceramics [72]. This characteristic tends to result in a heterogeneous dissolution and higher profile amplitude along the sampling length. In contrast, evenly distributed small crystals such as in fluorapatite-based ceramic [26,30,72,73] and leucite glass ceramics with small, rounded crystals [34,72] seem to provide a less damaging dissolution profile according to results shown in Fig. 3. Furthermore, Posritong et al. [26] demonstrated that surfaces etched with 5 % HF acid for 30 and 60 s provided a smoother surface with shallower pits, which might indicate similar acid dissolution susceptibility within the ceramic components.

In contrast to the flexural strength analysis for feldspathic ceramics, fatigue failure load was only reduced by acid concentrations lower than 10 %. According to Guilardi et al. [48] even though etched specimens showed a significantly more irregular surface with deeper pits and grooves, 10 % acid etching was not able to reduce fatigue resistance. This corroborates with Venturini et al. [46], who reported a higher cement filling potential on specimens etched with 10 % HF acid.

Cementation can improve the mechanical behavior of brittle materials by diminishing flexure, improving stress distribution, and reducing tensile stresses on the intaglio surface opposite to the cusp contact [27,48,46,55,47]. Therefore, reducing radial flexural fractures. Additionally, the crack closure phenomenon occurs by the resin cement infiltration into the crack that reduces the roughness effect caused by the acid etching. Some studies have theorized that the polymerization shrinkage of the cement at the crack tip can strengthen the crack bridging by opposing the tensile opening mode [21,26,74–76]. The ability to completely fill cracks also relies on the cement viscosity and silanization efficiency. According to Guilardi et al. [48], self-adhesive resin cement has a higher viscosity and, thus, is less able to fill surface flaws. To overcome this limitation the application of an adhesive layer on the ceramic was proposed as it can increase ceramics fatigue load-bearing capacity [77]. However, this method does not contribute to bond strength according to Passos et al. [78] and Nogueira et al. [79].

Furthermore, the effectiveness of silane in forming siloxane bonds with the ceramic surface has a detrimental effect on the spreading ability of the cementing agent [8,48,68,80]. A hydrophobic surface is formed by the reduction of hydroxyl groups that reacted with silanol groups and the presence of organofunctional groups of silane methacrylates [80]. This increases the affinity to the cement hydrophobic monomers and might explain the effect of silane on improving the fatigue resistance of glass ceramics [8,78,80].

4.2. Lithium disilicate glass-ceramic and zirconia-reinforced lithium silicate glass-ceramic

Lithium disilicate glass-ceramic (LD) has interlocked needle-like crystals that sustain the resistance of its surface even after the extensive dissolution of the glassy matrix [7,55]. The aggressive dissolution of the glassy phase within lithium disilicate crystals produces a deeper and irregular dissolution pattern which improves cement infiltration [7,8,68]. The topographic analysis of the studies included in subgroup 2.1.1 in the FFL analysis (Fig. 7), showed that the pits introduced by etching were completely filled with cement [51,55,54]. The hybrid layer formed by the cement within the crystals of the LD yields a network able to hinder crack initiation at the intaglio surface. This cementation protective effect may explain the increase in fatigue failure load (Fig. 7), although flexural load values decreased (Fig. 4). Even though aggressive etching protocols have not negatively impacted LD in terms of fatigue, the dissolution depth still is a concerning factor, especially for thin laminate veneers, that should be further evaluated [6,24].

Three studies that evaluated zirconia-reinforced lithium silicate glass-ceramic showed contrasting dissolution patterns with those of lithium disilicate glass-ceramic, given their difference in their microstructure and crystal size. ZLS is composed of three main Silica structures as follows: glassy phase (SiO2), lithium silicate crystals (Li2SiO3), and Quartz (SiO2 crystalline form) resulting from partial crystallization with ZrO_2 as a nucleating agent. ZLS presents finer equiaxial crystals (0.5 µm) with 10 % (in weight) of zirconia crystals dispersed in the glass matrix, while LD features needle-like crystals (1.5 - 3.13 µm) [4,19,68,72]. According to Monteiro et al. [19] 5 % HF etching for 20 s clearly shows a selective dissolution of the glass matrix by the exposure of Li_2SiO_3 and zirconia crystals, but it was insufficient to remove bur

Study or Subgroup	Control Mean [ffL (N)]	Control SD [ffL (N)]	Control Total	Hydrofluoric acid etch Mean [ffL (N)]	Hydrofluoric acid etch SD [ffL (N)]	Hydrofluoric acid etch Total	Weight	Mean Difference IV, Random, 95% CI
2.1.1 Lithium Disilicate glass-ceramic								
Prochnow et al, 2018(2) 3% 20s LD CAD	965	197.6435	20	1,355	47.2208	20	4.7%	-390.00 [-479.06, -300.94]
Dapieve et al, 2020 5% 20s LD CAD	786.67	213.36	15	830	127.9	15	4.5%	-43.33 [-169.22, 82.56]
Dapieve et al, 2022 5% 20s LD CAD	639	100.0538	6	817	60.0323	6	4.6%	-178.00 [-271.36, -84.64]
Prochnow et al, 2018(2) 5% 20s LD CAD	965	197.6435	20	1,335	86.5358	20	4.6%	-370.00 [-464.56, -275.44]
Scherer, et al 2018 5% 20s LD CAD	876.7	96.5	15	1,305.9	66.1	15	4.8%	-429.20 [-488.39, -370.01]
Prochnow et al, 2018(2) 10% 20s LD CAD	965	197.6435	20	1,175	201.9169	20	4.5%	-210.00 [-333.83, -86.17]
Tribst et al, 2019 10% 20s LD CAD	1,210	60	20	1,430	265	20	4.5%	-220.00 [-339.08, -100.92]
Subtotal (95% CI)			**116**			**116**	**32.2%**	**-268.85 [-372.49, -165.21]**
Heterogeneity: Tau² = 16866.40; Chi² = 49.68, df = 6 (P < 0.00001); I² = 88%								
Test for overall effect: Z = 5.08 (P < 0.00001)								
2.1.2 Feldspathic glass-ceramic								
Venturini et al, 2017 1% 60s Felds	245	15.1	20	242.5	24.7	20	4.9%	2.50 [-10.19, 15.19]
Venturini et al, 2018 1% 60s Felds	351.7	13.4	20	301.7	71	20	4.8%	50.00 [18.33, 81.67]
Venturini et al, 2017 5% 60s Felds	245	15.1	20	216.7	22.5	20	4.9%	28.30 [16.42, 40.18]
Venturini et al, 2018 5% 60s Felds	351.7	13.4	20	255	23	20	4.9%	96.70 [85.03, 108.37]
Yi-J and Kelly, 2011 HF9% for 60s Felds	251.43	10.6	10	222.86	14.16	10	4.9%	28.57 [17.61, 39.53]
Venturini et al, 2017 10% 60s Felds	245	15.1	20	255.7	53.8	20	4.8%	-10.70 [-35.19, 13.79]
Venturini et al, 2018 10% 60s Felds	351.7	13.4	20	341.7	20.6	20	4.9%	10.00 [-0.77, 20.77]
Guilardi et al, 2020 10% 60s Felds	544.47	28.03	5	506.84	42.79	5	4.8%	37.63 [-7.21, 82.47]
Subtotal (95% CI)			**135**			**135**	**38.8%**	**30.20 [4.51, 55.88]**
Heterogeneity: Tau² = 1251.61; Chi² = 168.68, df = 7 (P < 0.00001); I² = 96%								
Test for overall effect: Z = 2.30 (P = 0.02)								
2.1.3 Zirconia-reinforced lithium silicate glass-ceramic								
Monteiro et al, 2018 5% 30s ZLS	257.5	23.9	15	857.5	43.8	15	4.8%	-600.00 [-625.25, -574.75]
Monteiro et al, 2018 5% 60s ZLS	257.5	23.9	15	857.5	99.3	15	4.8%	-600.00 [-651.69, -548.31]
Monteiro et al, 2018 5% 90s ZLS	257.5	23.9	15	795.6	37.4	15	4.9%	-538.10 [-560.56, -515.64]
Monteiro et al, 2018 10% 30s ZLS	257.5	23.9	15	610	23.9	15	4.9%	-352.50 [-369.60, -335.40]
Monteiro et al, 2018 10% 60s ZLS	257.5	23.9	15	837.5	84.9	15	4.8%	-580.00 [-624.63, -535.37]
Monteiro et al, 2018 10% 90s ZLS	257.5	23.9	15	1,052.5	99.3	15	4.8%	-795.00 [-846.69, -743.31]
Subtotal (95% CI)			**90**			**90**	**29.0%**	**-576.48 [-695.85, -457.11]**
Heterogeneity: Tau² = 21878.14; Chi² = 505.54, df = 5 (P < 0.00001); I² = 99%								
Test for overall effect: Z = 9.47 (P < 0.00001)								
Total (95% CI)			**341**			**341**	**100.0%**	**-240.82 [-335.73, -145.91]**
Heterogeneity: Tau² = 48192.98; Chi² = 8092.28, df = 20 (P < 0.00001); I² = 100%								
Test for overall effect: Z = 4.97 (P < 0.00001)								
Test for subgroup differences: Chi² = 119.86, df = 2 (P < 0.00001), I² = 98.3%								

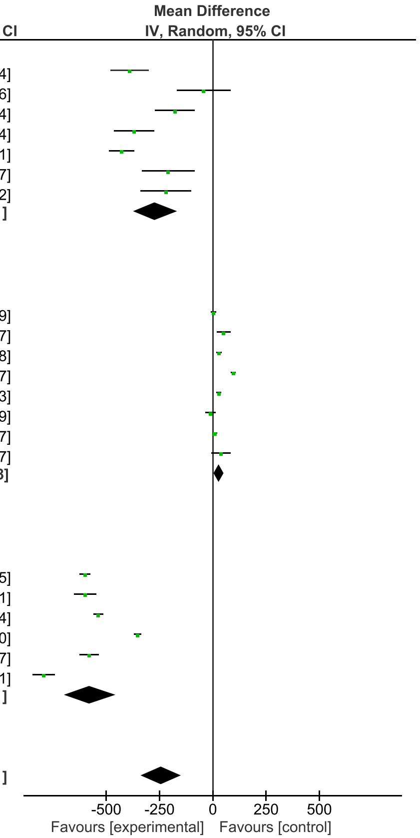

Fig. 7. Subgroup analysis for fatigue failure load between hydrofluoric acid etching and control of lithium disilicate glass-ceramic, feldspathic ceramics, and zirconia-reinforced lithium disilicate.

Study or Subgroup	Self-etch ceramic primer Mean [ffl (N)]	SD [ffl (N)]	Total	Hydrofluoric acid etch Mean [ffl (N)]	SD [ffl (N)]	Total	Weight	Mean Difference IV, Random, 95% CI
Scherer, et al 2018 5% 20s LD CAD	1,193.7	65.1	15	1,305.9	66.1	15	14.4%	-112.20 [-159.15, -65.25]
Dapieve et al, 2022 10% 60s LD CAD HV	875	112.3216	10	955	137.6795	10	13.0%	-80.00 [-190.13, 30.13]
Dapieve et al, 2022 10% 60s LD CAD LV	1,090	196.727	10	815	89.1303	10	12.3%	275.00 [141.14, 408.86]
Dalla-Nora et al, 2021 5% 20s ZLS	1,026.67	152.8942	15	1,206.67	217.9018	15	12.3%	-180.00 [-314.71, -45.29]
Schestatsky et al, 2019 5% 20s LD CAD	1,200	92.2616	10	1,220	199.9002	10	12.3%	-20.00 [-156.46, 116.46]
Schestatsky et al, 2019 5% 20s LD Press	1,460	184.5233	10	1,400	294.9577	10	9.8%	60.00 [-155.64, 275.64]
Dapieve et al, 2020 5% 20s LD CAD	880	106.57	15	830	127.9	15	13.7%	50.00 [-34.25, 134.25]
Tribst et al, 2019 10% 20s LD CAD	1,750	162	20	1,430	265	20	12.3%	320.00 [183.88, 456.12]
Total (95% CI)			**105**			**105**	**100.0%**	**34.70 [-81.72, 151.12]**

Heterogeneity: Tau² = 23955.22; Chi² = 66.82, df = 7 (P < 0.00001); I² = 90%
Test for overall effect: Z = 0.58 (P = 0.56)

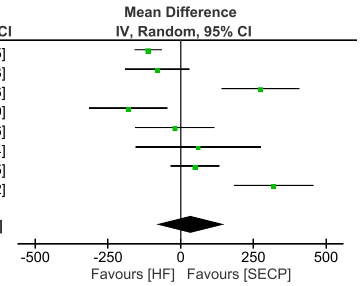

Fig. 8. Forest plot for the analysis of fatigue failure load between hydrofluoric acid etching and self-etch ceramic primer (SECP).

scratches and grooves that were introduced in the manufacturing process. According to Barchetta et al. [28] the inability of shorter HF application times (10 % HF for 20 s) to remove pre-existing flaws was the reason for the lower values in flexural strength compared to 10 % HF etching for 60 s. With the increase in etching time Monteiro et al. [19] reported an increase in cement infiltration that can promote protective interactions such as crack bridging, [15,26,81] which leads to a reduction of flexural cracks at the tensile surface [12].

4.3. Hybrid ceramics

The application of HF acid was suggested as surface treatment for CAD/CAM hybrid ceramic materials due to the high filler content and a positive correlation for bond strength [82–84]. However, the microstructural differences between CAD/CAM dispersed filler resin composites (DF-RCBs) and a polymer-infiltrated ceramic network (PICN) material lead to contrasting dissolution patterns. Acid etching on composite materials with dispersed fillers caused the complete removal of silica compounds, hindering the silane methyl group from reacting with the substrate Si-OH group [8,43,80]. Therefore, aluminum oxide sandblasting is more effective due to being a non-selective surface treatment [7,85].

The role of stress-strain properties and particle toughening mechanisms should be considered to correlate fractographic events in composite materials to surface profile modification by the acid dissolution of the inorganic phase [86]. The infiltration of a polymer in a continuous ceramic structure rather than filler dispersal into a resin matrix led to a material with higher hardness than conventional resins and with higher strain to fracture and damage tolerance than feldspathic ceramic materials [43,71]. The polymer phase increases toughness by a crack bridging mechanism and by limiting crack propagation to the ceramic structure [71]. This is based on the “principle of combined action”, in which the combination of materials of distinct mechanical characteristics can overcome the inherent deficiencies of each component individually. Nevertheless, the results of this study suggest that surface modification by acid etching still is a concerning factor in terms of flexural strength.

For all the HF application regimens there were a significant decrease in flexural strength. Fractographic analysis of the studies from Fig. 5 showed that all fractures emanated from the etched surface, which may imply the introduction of crack initiation defects by the acid etching. Results suggest that for PICN materials, etching with a 5 % HF is less damaging, while for DF-RCBs the HF acid should not be used as there is no significant improvement in bond strength that could justify the lessening in its flexural strength caused by HF acid etching.

4.4. Self-etch ceramic primer

As an alternative for the gold standard hydrofluoric acid etching followed by 3-MPS silane application, a novel self-etch ceramic primer combines a milder acidity etchant (tetrabutylammonium dihydrogen trifluoride) and a silane monomer (trimethoxysilylpropyl methacrylate) in a single bottle. According to the manufacturers, a single step etching and silanization intend to reduce application time, and technique sensitivity and avoid exposure to hydrofluoric acid, a highly poisonous solution. Goes et al. [7] demonstrated that SECP-treated specimens had no significant difference in etching depth compared to untreated groups, corroborating with other studies [55,65] in which SECP produced a less pronounced etching pattern compared to HF. This might be explained by the lower acidity of this fluoride compound and due to the formation of insoluble reaction products that may prevent the excessive dissolution of the glassy matrix mainly in pre-existing flaws [13,65]. However, the current study showed no significant difference between SECP and HF regarding FS and FFL. Regarding chemical interaction and wettability, SECP showed the highest contact angle and hydrophobicity when compared with HF [8,37,54]. This is expected from an efficiently silanized surface [8,87] as it indicates the formation of covalent bonds (Siloxane, Si-O-Si) with Si atoms from the ceramic surface and the presence of methacrylate end groups to further copolymerization with resin cement monomers [2,8,51,49].

4.5. Limitations

The high heterogeneity seen in most figures could be caused by the variability of ceramic materials (type and composition within brands), silane primers (composition and lot number), the polishing protocol used (differences in time, brand of polisher, type of polisher and force applied), and the machine used for testing these materials (brand, cell load, and the velocity of applied force). The inability of the polishing protocol to remove previous surface defects implies that the assessment of the HF acid variable is not isolated from other factors, such as milling and grinding. High heterogeneity is also expected due to differences in fatigue and flexural test setups, as shown in Tables 2 and 3, respectively. Since no material, polishing protocol, or test setups from the included results can be considered as the ’gold standard,’ a sensitivity analysis was not performed. Regardless that most studies had a low risk of bias, only 27 % of the studies reported the sample size calculation, which has a great impact on the results and is often defined based on studies with similar methodologies. Also, sample preparation and allocation are often poorly reported, and are usually performed by a single operator. The results should be interpreted with caution, taking into consideration that some subgroup analyses had only one or a few studies and the limitations of *in vitro* testing to simulate clinical conditions. For this reason, this review should not dictate protocols for clinical use but can guide the development of clinical research aimed at reducing the concentration of hydrofluoric acid or exploring conditioning materials that are free from this component, considering each type of ceramic reported.

5. Conclusion

Based on the findings of this systematic review and meta-analysis of *in vitro* studies, the following conclusions were drawn:

1. For predominantly glassy ceramics, 10 % HF acid etching for 60 s or less, reduced the flexural strength, while 5 % HF acid had no impact. Acid concentration and application time did not influence the fatigue failure load of these materials.
2. HF acid etching had a negative impact on lithium disilicate glass ceramics. However, all HF acid etching regimens significantly increase the fatigue failure load which indicates the effect of combined action with the resin cement.
3. HF acid etching reduced the flexural strength of hybrid ceramics.
4. The self-etch ceramic primer and HF acid etching had similar fatigue failure load and flexural strength values.

Conflict of 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.

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