Clinical Oral Investigations (2025) 29:141
https://doi.org/10.1007/s00784-025-06208-5

RESEARCH

Optimizing cleaning procedures for MEP residues: insights from a novel HPLC-MS analysis

Mei-Ling Zheng[1] · Jun-Yu Chen[2] · Bing Chen[3] · Chang-Yuan Zhang[4]

Received: 13 January 2025 / Accepted: 3 February 2025

Abstract

Objectives To develop a highly sensitive and reliable method for detecting Monobond Etch & Prime (MEP) residues on lithium disilicate glass ceramic (LDGC) surfaces treated with different cleaning procedures after MEP treating.

Materials and methods A sensitive and reliable high performance liquid chromatography-tandem triple quadrupole mass spectrometry (HPLC-TTQ/MS) method was developed for the accurate quantitative determination of tetrabutylammonium dihydrogen trifluoride (TDTF) in MEP. 100 rectangular LDGC specimens (8.0 × 5.0 × 2.0 mm) were fabricated using CAD/CAM and then treating with MEP. The residual amount of TDTF in the LDGC specimens surface was tested after different cleaning procedures with different experience dentists (Junior Dentist, Experienced Dentist), different rinsing times (3 s, 6 s, 9 s) and with or without ultrasonic cleaning (UC) ($n = 10$).

Results The developed method showed good linearity ($r^2 > 0.999$) over a wide concentration range. The limit of quantification of TDTF for MEP residue was less than 5 ng/mL. The method was then applied to a comparative study of MEP residues after various cleaning procedures of LDGC specimens. Residual TDTF was detected in all experimental groups with masses ranging from 62.42 ng to 74,611.74 ng. In the absence of UC, the Experienced Dentist group had lower TDTF residual levels (5,930.74 ng) than the Junior Dentist group (30,802.05 ng) ($P < 0.001$). The TDTF residue level was significantly lower in the rinsing 9 s group (3,102.89 ng) compared to the rinsing 3 s group (25,348.57 ng) ($P < 0.05$). After UC, for the same rinsing time there was no significant difference in TDTF residue between groups ($P \geq 0.062$). Notably, TDTF residue levels were substantially lower after UC with the same cleaning dentist and rinsing time ($P \leq 0.012$).

Conclusions No cleaning procedure can completely remove MEP residues, the dentist's clinical experience affects the degree of MEP cleaning, extended rinsing time improves cleaning efficiency, and ultrasonic cleaning is an effective method of removing MEP residues.

Clinical relevance No cleaning procedure can completely remove MEP, and dentists can increase the efficiency of cleaning by extending the rinsing time and using ultrasonic cleaning.

Keywords Cleaning procedures · MEP residues · TDTF · HPLC-MS

Mei-Ling Zheng, Jun-Yu Chen and Bing Chen have contributed equally to this work.

✉ Chang-Yuan Zhang
longsource@126.com

Mei-Ling Zheng
1454048010@qq.com

Jun-Yu Chen
18750871617@163.com

Bing Chen
BingChen_001@fjmu.edu.cn

[1] School and Hospital of Stomatology, Fujian Medical University, Fuzhou 350002, PR China

[2] School of Pharmacy, Fujian Medical University, Fuzhou 350122, PR China

[3] Key Laboratory of Nanomedical Technology, Education Department of Fujian Province, School of Pharmacy, Fujian Medical University, Fuzhou 350122, PR China

[4] Department of Prosthodontics, School and Hospital of Stomatology, Fujian Medical University, No.246 Yangqiao Zhong Road, Fuzhou 350002, Fujian Province, PR China

Published online: 19 February 2025

Introduction

Lithium Disilicate Glass-Ceramics (LDGC) is one of the most commonly used restorative materials in current dental clinics due to its excellent aesthetic, biocompatible and mechanical properties [1]. Debonding is one of the major clinical complications of LDGC [2]. The most common method for improving the bond strength of LDGC is etching with hydrofluoric acid (HF) followed by silanization [3, 4]. After etching with HF, the glass matrix of the LDGC is selectively removed and the crystal structure is exposed, thus the ceramic surface becomes roughened, which is expected to form micromechanical retention on the ceramic surface [5]. In addition, the roughened etched surface helps to provide more surface energy before binding with silane solution [6]. However, HF is hazardous because it is corrosive, reactive and toxic [7]. In case of direct contact, HF may cause tissue damage, even necrosis [8], so the use of HF as a ceramic etching gel is banned in some countries [9].

To avoid health hazards of HF in the dental office, a self-etching glass-ceramic primer Monobond etch & prime (MEP) is recommended to be used as a substitute for surface treatment of HF and silane [9, 10]. MEP not only reduces the processing steps, but also reduces the potential toxic effects associated with HF [11]. It was found that the average bond strength of LGDC treated with MEP was higher or not significantly different from HF acid etching and silanization [12–14]. Unfortunately, although MEP is much less harmful than HF, it is still slightly cytotoxic and should not be used directly in the mouth [15]. Because the etching component of MEP, tetrabutylammonium dihydrogen trifluoride (TDTF), is corrosive, acutely toxic and irritating [16]. So the manufacturer recommends thoroughly rinsing MEP off with water until the green colour has been removed. However, the exact rinsing method and time are not clear, and visual judgment alone may result in residual MEP. So it is necessary to investigate the effect of different cleaning procedures on the removal of MEP.

Therefore, the aim of this study was to develop a highly sensitive and reliable method for detecting MEP residues and to use it for a comparative study of the effect of different cleaning procedures on MEP residues on LDGC surfaces after MEP treatment. The null hypotheses were: (1) There was no significant difference in MEP residue after cleaning by dentists with different experience; (2) There was no difference in MEP residue by different cleaning time with water; (3) There was no difference in MEP residue before and after ultrasonic cleaning.

Materials and methods

Reagents and materials

Methanol and ethanol of mass spectrometry-grade was acquired from Merck (Darmstadt). Tetra-N-Butylammonium Dihydrogentrifluoride and tetrapropylammonium Iodide were purchased from Adamas-beta (Benchmade). Ultrapure water was prepared by a Milli-Q purification system (Mill-pore). Ethanol of analytical grade and other chemical reagents were purchased from Sinopharm Chemical Reagent Corporation.

MEP marker calibration

TDTF (Adamas-beta) was used as the standard, while tetrapropylammonium iodide (TPAI; Adamas-beta) served as the internal standard (IS)(Fig. 1). The standard working solution concentrations were 10, 25, 50, 100, 250, 500, and 1000 ng/mL, with the final IS concentration in the samples set at 50 ng/mL. Based on quantitative detection via Liquid Chromatograph-Mass Spectrometer (LC-MS, LCMS-8040; Shimadzu), the standard curve for TDTF and the lower limit of quantification (LLOQ) were determined through regression analysis using weighted least squares ($W = 1/X^2$). The equation for the standard curve was $y = 12182x - 9163.3$ ($r^2 = 0.999$), indicating good linearity for TDTF within the range of 10 to 1000 ng/mL (Table 1). Method validation was performed using three sets of standardized working solution concentrations: low (20 ng/mL), medium (400 ng/mL), and high (800 ng/mL). The validation results demonstrated that the quantitative detection of TDTF by high performance liquid chromatography-mass spectrometry (HPLC-MS) was specific (Fig. 2), exhibiting excellent accuracy, precision (Table 1), and stability (Table 2).

LDGC specimen fabrication

A rectangular stereolithography (STL) file was designed using Three-dimension design software (Solidworks, V2023; Solidworks Corp). The rectangle is 8.0 mm long, 5.0 mm wide, and 2.0 mm thick. A cylindrical bar with a diameter of 1.5 mm and a length of 5 mm is centered on one surface of the rectangle (Fig. 3).

The STL file was entered into the computer and 110 identical wax patterns were produced using CAD/CDM (Wieland). 110 LDGC specimens (IPS e.max Press; Ivoclar Vivadent) were sintered and fabricated using wax patterns according to the manufacturer's instructions. All LDGC specimens were checked for dimensional consistency, three of the failed specimens were discarded, and 100 specimens were randomly selected for experimental study.

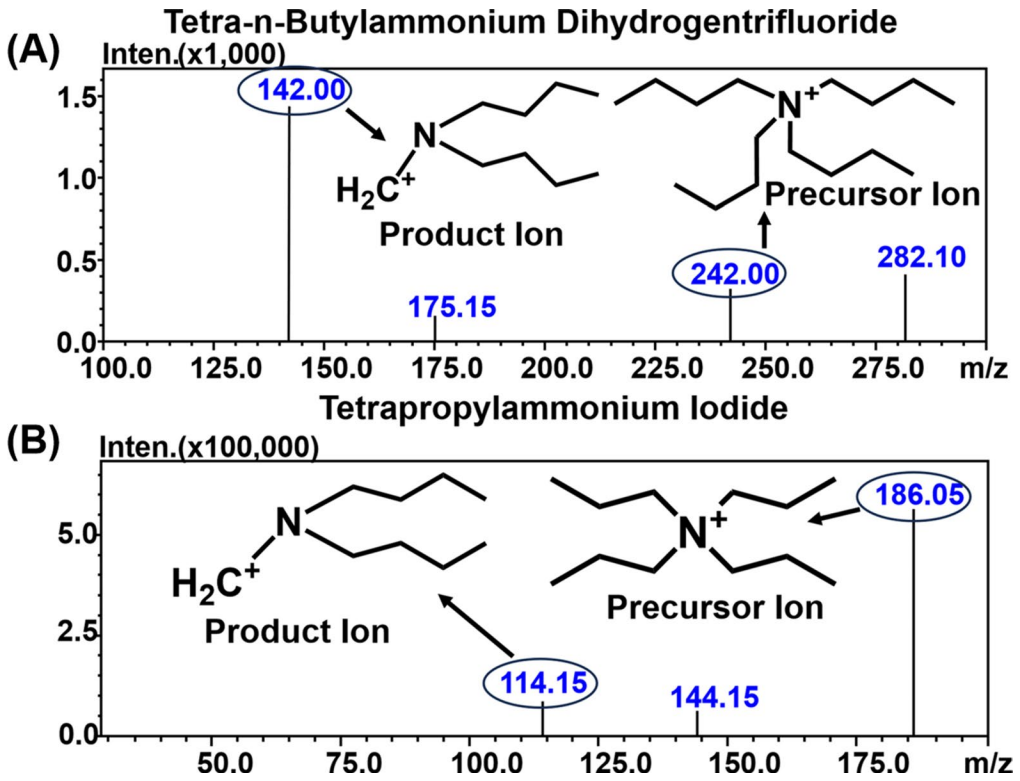

Fig. 1 The structures and product ion mass spectra of organic cations of (**A**) tetrabutylammonium dihydrogen trifluoride (TDTF) and (**B**) tetrapropylammonium iodide (TPAI, IS)

Table 1 Linearity of the calibration curves, LLOQ, precision and accuracy of HPLC-MS method for detection of TDTF

Analyte	Linear range (ng/mL)	LLOQ (ng/mL)	Regression equation ($w=1/x^2$)	Precision (RSD, %)			Accuracy	
				Conc. (ng/mL)	Intraday	Interday	Conc. (ng/mL)	RE (%)
TDTF	10 ~ 1000	3	y = 12182x -9163.3	20	7.17	8.94	20	-2.65
				400	5.61	5.43	400	9.89
				800	1.61	9.26	800	9.34

RSD, relative standard deviation; Conc., concentration; RE, relative error

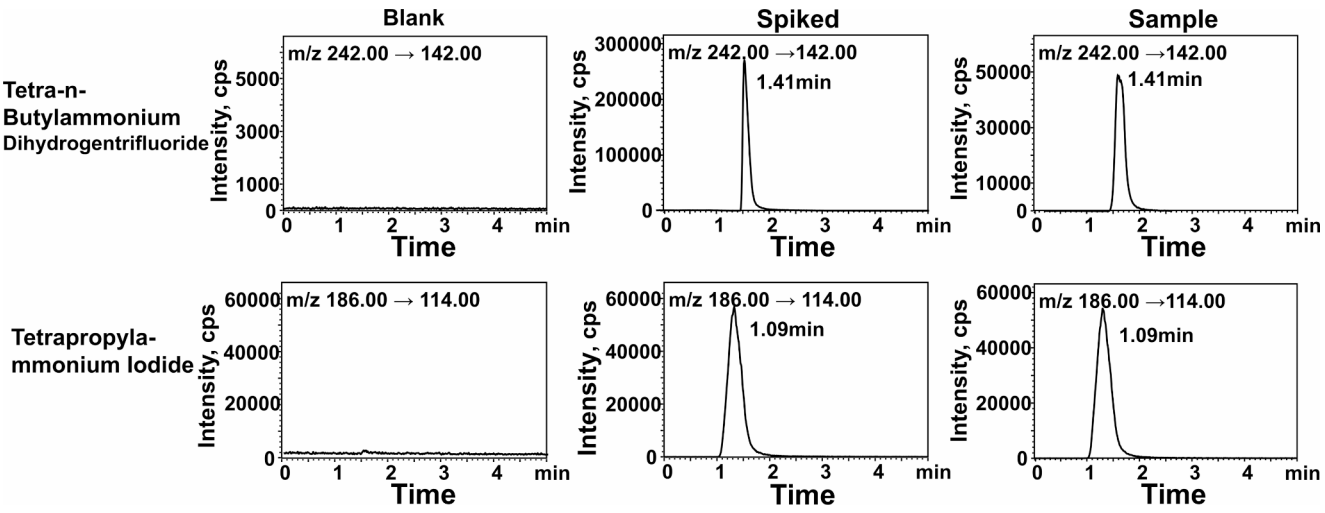

Fig. 2 Representative chromatograms of sample: blank solvent sample, blank solvent spiked with quality control (QC) and IS (50 ng/mL of TDTF and TPAI), sample collected from the MEP residuals after cleaning procedures

Table 2 Bench-top, short-term, freeze-thaw and long-term stability of TDTF detection

Analyte	Conc. (ng/mL)	Bench-top stability (37°C, 8 h)		Short-term stability (4°C, 12 h)		Freeze-thaw stability (three cycles)		Long-term stability (-80°C, 30 days)	
		Bias (%)	RSD (%)	Bias (%)	RSD (%)	Bias (%)	RSD (%)	Bias (%)	RSD (%)
TDTF	20	-7.77	2.09	0.25	12.53	0.86	10.64	5.31	11.19
	400	-6.00	0.20	2.00	4.14	5.51	3.95	6.42	3.13
	800	-2.68	13.88	0.82	2.22	4.94	2.24	5.79	2.43

Conc., concentration;RSD, relative standard deviation

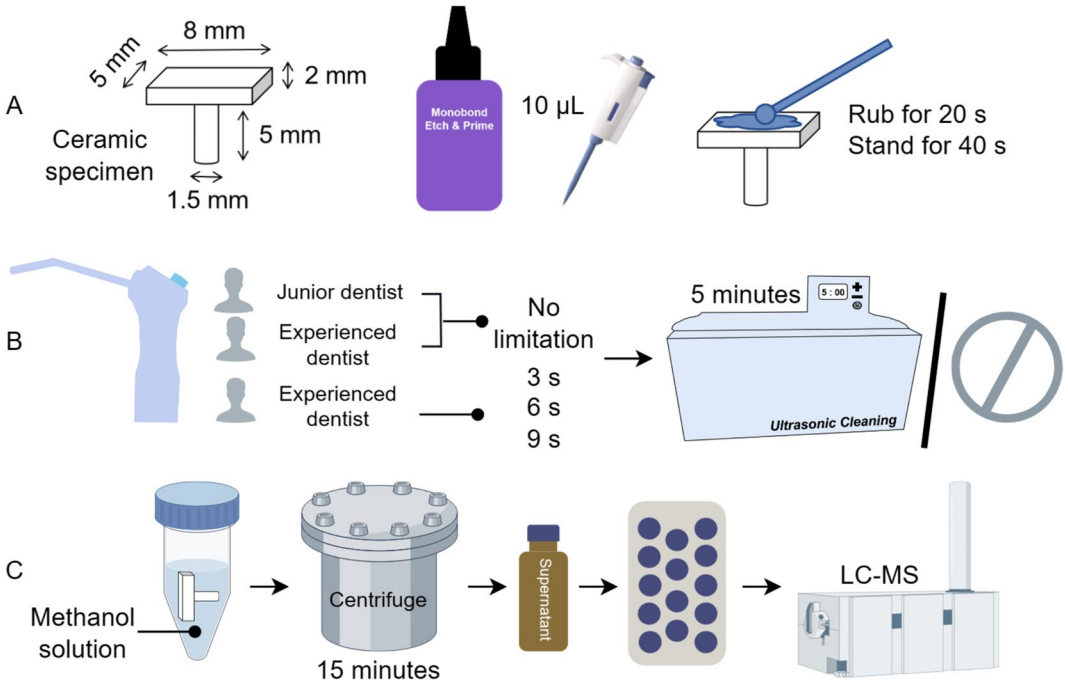

Fig. 3 Experimental design of the study. (**A**, Etching. **B**, Flushing. **C**, Testing)

Treatment of LDGC specimens with MEP

100 LDGC specimens were ultrasonically (BioSonic UC125; COLTENE) cleaned with distilled water for 5 min and then air dried. 10 uL of MEP (Ivoclar Vivadent) solution was uniformly applied to the test surface of each specimen using a pipette (Single Channel Digital Variable Pipette; Shanghai Zhouhui Biochemical Instrument Co., Ltd.). Rub the MEP on the surface of the specimen with a miniature brush for 20 s, then let it stand for 40 s.

Cleaning procedures

The LDGC specimens were divided into 10 groups ($n=10$) according to the cleaning procedure, showed in Table 3. The junior dentist was a dentist with less than 2 years of clinical practice, and the experienced dentist had more than 15 years of dental clinical practice. The air pressure of air-water spray in present study was 2.5 MPa.

After rinsing with water, each specimen was immersed in an EP (Eppendorf AG) tube containing 600 µL of prepared aqueous methanol solution (methanol: purified water = 1: 1) (Methanol; Concord Technology) for 24 h.

HPLC-MS/MS analysis of MEP

Stock solutions, quality control and calibration standard samples

Accurately weigh TDTF, TPAI, and use purified water to make up to a final concentration of 1 mg/mL for the stock solutions, which were then stored in a refrigerator at 4°C. Dilute the stock solution of TDTF with purified water, vortex mix thoroughly to prepare three sets of quality control working solutions at low, medium, and high concentrations. The concentrations for TDTF (QC) working solutions were 20, 400, and 800 ng/mL, with the final concentration for TPAI maintained at 50 ng/mL. A certain volume of the TDTF and TPAI stock solutions was diluted using a dilution

Table 3 Cleaning procedures of different experimental groups

Group	Cleaning operator	Cleaning condition	Cleaning time	Ultrasonic cleaning 5 min
JD	Junior dentist	Unrestricted, flushing the LDGC specimen etched surface with an air-water spray in accordance with individual clinical practice. The duration, mode, and distance of the cleaning procedure were unlimited.	unrestricted	No
JDUC				Yes
ED	Experienced dentist			No
EDUC				Yes
T3	Experienced dentist	Flushing the LDGC specimen etched surface vertically with an air-water spray at a distance of 1.0 cm.	3 s	No
T3UC				Yes
T6			6 s	No
T6UC				Yes
T9			9 s	No
T9UC				Yes

JD: Rinse under running water by Junior dentist; JDUC: Rinse under running water by Junior dentist + Ultrasonic Cleaning; ED: Rinse under running water by Experienced dentist; EDUC: Rinse under running water by Experienced dentist + Ultrasonic Cleaning; T3: Rinse under running water for 3 s; T3UC: Rinse under running water for 3 s + Ultrasonic Cleaning; T6: Rinse under running water for 6 s; T6UC: Rinse under running water for 6 s + Ultrasonic Cleaning; T9: Rinse under running water for 9 s; T9UC: Rinse under running water for 9 s + Ultrasonic Cleaning

factor with purified water and mixed thoroughly to prepare a series of standard working solutions for the calibration curve. The concentrations in the working solutions for TDTF were 5, 10, 25, 50, 100, 250, 500, 800, and 1000 ng/mL, while the final concentration for TPAI was maintained at 50 ng/mL. Regression analysis was performed using $1/X^2$ weighting to obtain standard curves and linear ranges for each component, with the lowest concentration point in each standard curve serving as the LLOQ.

Quantitative analysis of MEP

Chromatographic separation was performed using an Ultimate® XB-C18 column (50 × 2.1 mm, 3.5 µm) with a mobile phase consisting of 0.01% acetic acid in water (A) and methanol (B). The flow rate and column temperature were maintained at 0.2 mL/min and 40 °C, respectively. A gradient elution program was applied as follows: 0–1 min: B (%) 35–40; 1–3 min: B (%) 40–80; 3–5 min: B (%) 80–90; 5–7 min: B (%) 95; 7–13 min: B (%) 35; with 35% B used for equilibration.

Analysis was conducted using the Shimadzu HPLC-MS 8040 triple quadrupole mass spectrometer in Multiple Reaction Monitoring (MRM) mode. The MRM transition parameters for positive ionization mode, as shown in Fig. 1, indicate that for tetrabutylammonium the selected precursor and productions were m/z 242.00 → 142.00 with a collision energy (CV) of -24.0 V, and for tetrabutylammonium iodide, they were m/z 186.00→114.00 with a collision energy of -24 V. The electrospray voltage was set to 6000 V, nebulizer gas flow was 3 L/min, drying gas flow was 15 L/min, DL temperature was maintained at 250°C, and heat block temperature was set at 400°C.

Samples preparation

EP tubes impregnated with LDGC specimens were processed as follows: sonicated in 0°C water for 5 min, vortexed for 3 min (vortex mixer, NY-1; Enyi Instrument Manufacturing Co., Ltd.), and centrifuged at 1000 g/min for 15 min (centrifuge 5430 R; Eppendorf AG). 200 µL of supernatant from each EP tube was transferred to individual vials, and 5 µL of IS was added to each vial for quantitative analysis by HPLC-MS [17].

Statistical analysis

Data were performed using GraphPad Prism software (v8.0.2; GraphPad Software Corp). Tests of normality, independent Two-Sample T-Tests, and one-way ANOVA tests ($\alpha = 0.05$) were used to assess the significance of the results.

Results

Method development and validation

In the validation process, typical MRM chromatograms of the analytes and IS were analyzed in three different scenarios: (A) drug-free solvent, (B) solvent spiked with QC and IS, and (C) samples collected from LDGC specimens that had been treated with MEP and subsequently cleaned. As depicted in Fig. 2, no interfering endogenous peaks were observed in the blank solvent samples obtained from the subjects.

The method for quantifying MEP in rinsed plasma demonstrated excellent accuracy and precision, with calibration curves showing a linearity of $r^2 \geq 0.999$ across concentrations of 5-1000 ng/mL, and intra- and inter-day precision (RSD) for quality control samples remaining below 9.26%, while the accuracy ranged from -2.65 to 9.89%. After investigating specificity, linearity, precision, stability, matrix effects, extraction recovery, and dilution effects, the method for detecting MEP was confirmed to be accurate, stable, and reliable.

Table 4 Amount of TDTF for different experimental group

Group	*N*	Mean (ng)	SD	*P*
JD	10	30805.48	16152.79	<0.001****
JDUC	10	264.77	123.75	
ED	10	5827.63	2602.18	<0.001****
EDUC	10	229.44	36.40	
T3	10	25348.57	24587.13	0.002**
T3UC	10	400.14	111.87	
T6	10	8304.97	6697.79	<0.001****
T6UC	10	443.88	184.66	
T9	10	3102.89	2358.14	0.012*
T9UC	10	316.17	89.93	

N, number of specimens; SD, standard deviation; *, **, **** means statistically significant differences

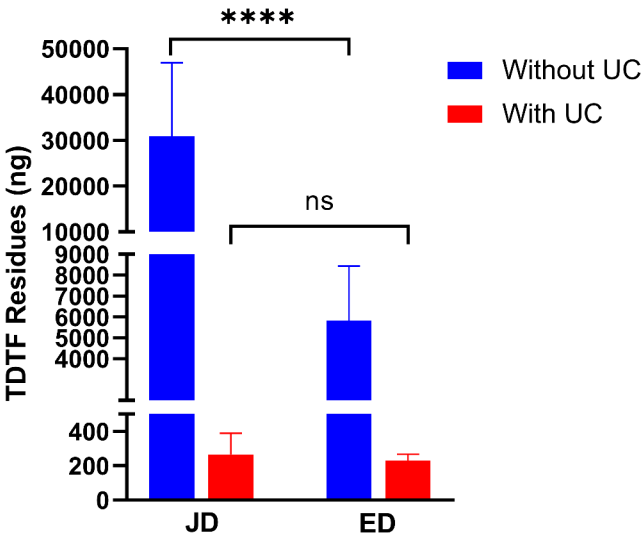

Fig. 4 Bar chart of mean TDTF residue values between the groups with different operator. ns means not significant; and **** means $P<0.001$ between the marked groups

Table 5 Brown-Forsythe and Welch ANOVA tests between groups with different time and without ultrasonic cleaning

Groups	T3	T6	T9
T3	/	0.135	0.045*
T6	0.135	/	0.098
T9	0.045*	0.098	/

*Statistically significant differences ($P<0.05$)

The test results of TDTF for each experimental groups

The test results of TDTF for each experimental groups were shown in Table 4. TDTF were found in all groups, with mass ranging from 62.42 to 74611.74 ng. Without limiting the cleaning time, ultrasonic cleaning had a statistically significant effect on the difference in TDTF for the same cleaning dentist ($P < 0.05$, Table 4). In the absence of ultrasonic cleaning, the amount of TDTF after cleaning by experienced dentist (5827.63 ng) was significantly lower ($P <$ 0.001, Fig. 4) than the result of cleaning by junior dentist (30805.48 ng).

Table 6 Duncan multiple comparisons analysis between groups with different time and with ultrasonic cleaning

Groups	T3UC	T6UC	T9UC
T3UC	/	>0.999	0.188
T6UC	>0.999	/	0.062
T9UC	0.188	0.062	/

In the absence of ultrasonic cleaning, by the same experienced dentist, the amount of TDTF was significantly less in the 9-second cleaning group than in the 3-second cleaning group ($P<0.05$, Table 5). And the differences between the cleaning 6-second group and both the 3-second ($P=0.135$) and 9-second groups ($P=0.098$) were not statistically significant. However, after ultrasonic cleaning, there was no difference in the amount of TDTF for different cleaning times ($P\geq0.062$, Fig. 4; Table 6).

Discussion

The aim of this study was to develop a highly sensitive and reliable method for detecting MEP residues on LDGC surfaces after MEP treatment and to compare the effects of different cleaning procedures on MEP residues.

HPLC-MS is an invaluable method for the detection of residues because of its high sensitivity for trace detection, excellent separation efficiency for compound identification and quantification, versatility in analyzing a wide range of chemical substances, quantitative analytical capability to ensure compliance with safety standards, and automation features that minimize errors and enable high-throughput detection [18, 19]. Method validation was an essential step in ensuring that the HPLC-MS analytical procedure for monitoring trace amounts of MEP residues in dental settings produces reliable and reproducible results. This study focuses on the validation of an LC-MS/MS method for the detection and quantification of MEP. After investigating specificity, linearity, precision, stability, matrix effects, extraction recovery, and dilution effects, the method for detecting MEP was confirmed to be accurate, stable, and reliable. Therefore, a highly sensitive and reliable method was developed to detect MEP residues in this study.

According to the results, without ultrasonic cleaning, the amount of TDTF after cleaning by experienced dentist (5827.63 ng) was significantly lower ($P < 0.001$) than that of cleaning by junior dentist (30805.48 ng). Extending the cleaning time reduced the amount of TDTF and there was a significant difference between cleaning for 3 s and 9 s. After ultrasonic cleaning, there was no difference in the amount of

TDTF for different cleaning times ($P \geq 0.062$). So all the null hypotheses were rejected.

Bonding between glass-ceramic and resin cement is dependent on the physical and chemical interaction between them, which is considered to be one of the key factors for long-term clinical success [20]. HF etching and subsequent silane application was considered the gold standard for bonding glass-ceramics [4]. However, HF is highly toxic and exposure to dentin produces amorphous fluoride deposits [21]. Therefore, the contact of HF to dentin should be avoided [22]. MEP is one of the substitutes to HF that is currently commonly used in clinical dentistry to achieve similar acid etching effects as HF [10, 11], but with much less toxic side effects.

MEP aims to eliminate the possible toxic effects and related unfavorable outcomes of using hydrofluoric acid [23]. MEP contains an etching-agent for ceramic, a silane, and a priming agent in a bottle, which enables surface etching coupled with silanization in one step [24]. Several studies have compared the differences in bond strength between treatments with MEP and HF. A number of studies have found that the bond strength of LDGCs treated with MEP was not significantly different [9, 11, 13, 14, 25] or even higher [12, 26] than that treated with HF. So MEP was introduced as a revolutionary self-etching ceramic primer [27]. However, although MEP is much safer than HF, it cannot be used in the oral cavity due to its slight cytotoxicity [15]. Therefore, after treatment with MEP, it needs to be thoroughly cleaned.

The instruction for MEP calls for rinsing with water and determining complete removal based on color, but do not specify the time, distance, or manner of running water rinsing, which depends heavily on the dentist's clinical experience. The TDTF amount in the experienced dentist group was significantly lower than that of junior dentist group without ultrasonic cleaning ($P < 0.001$), suggesting that clinically experienced dentists have advantage in better removal of MEP residues. However, the inability of both experienced and junior dentists to completely remove MEP suggests that visual color recognition alone is not completely effective in removing MEP, which should be of great concern to clinical dentists. Clinical experience and color recognition are not truly effective in removing MEP.

Cleaning methods for removing acid etchant residues from ceramic surfaces included the use of ultrasonic baths [28, 29], 37% phosphoric acid [30], and running water rinsing [31], whereas fewer residues were observed when ultrasonic cleaning was used [32, 33]. The use of ultrasonic cleaning has been shown to be an effective procedure for the removal of residual fluorosilicates [34]. Similar results were found in this study, where the addition of ultrasonic cleaning to running water rinse was the most effective method for removing MEP residues. In present study, the use of ultrasonic cleaning resulted in a significant reduction of MEP residue ($P \leq 0.012$) when the operator or running water cleaning time was same. So the use of running water rinsing followed by ultrasonic cleaning is highly recommended for clinical use of MEP.

The principle of ultrasonic cleaning is to remove particles through the cavitation effect produced by the liquid medium [35], and cavitation caused by changes in temperature and pressure will also cause chemical changes that accelerate the cleaning efficiency [36]. It should be noted, however, that the ultrasonic cleaning did not completely eliminate the TDTF either, suggesting that further refinement of the cleaning procedure may be required.

Cleaning time is another critical factor. Unlike inlays, onlays, crowns and other restorations that have a variety of irregular anatomical structures on the surface, in this study, the tested specimens had flat surfaces, regular structures, and small surface areas. Therefore, three relatively short running water rinse times of 3 s, 6 s and 9 s were set, with the main purpose of testing the effect of rinsing time on the cleaning situation. The results that the amount of the MEP residue decreases with the increase in cleaning time, with a significant difference between the results of rinsing for 3 s and rinsing for 9 s ($P = 0.045$). So extending the rinsing time is therefore an effective way to clean the etchant. And after ultrasonic cleaning, there was no longer a statistically significant difference ($P = 0.188$) in the amount of TDTF between 3 s and 9 s of rinsing time, which again demonstrates the importance of ultrasonic cleaning.

Gels do not flow easily and can be used for small area applications, whereas solvents are not easily contained within a small fixed area and tend to spill over the target etching surface. The etchant chosen for this study was a solvent that tends to spill over the etching surface during the study and cannot be completely controlled on the acid etching surface, which may be another reason why MEP residues were measured in all experimental groups. However, in dental clinical work, there are also cases of MEP overflowing the edge of the acid etching surface. Therefore, the setup of this study was consistent with the clinical reality, which suggests that clinical acid etching should be done with gel-based acid etchers rather than fluid solvents whenever possible.

In addition, although MEP residues were measured in all experimental groups, it is unknown whether MEP residues affect the bond strength and whether they have toxic effects on the dentin and surrounding tissues, which will be further studied in future.

Conclusions

Withing the limitation of present study, the follow conclusions can be drawn: (1) No cleaning procedure can completely remove MEP residues. (2) The dentist's clinical experience affects the degree of MEP cleaning. (3) Extended flushing time improves cleaning efficiency of removing MEP residues. (4) Ultrasonic cleaning is an effective method of removing MEP residues.

Author contributions Z.M.L performed experiments, analyzed the data, wrote the manuscript; C.J.Y. performed experiments; C.B. designed the research, revised the manuscript. Z.C.Y. conceived the idea, oversaw the research, revised the manuscript, and provided funding.

Funding This work was supported by Fujian Province Natural Science Foundation of China (No. 2023J01713).

Data availability No datasets were generated or analysed during the current study.

Declarations

Ethical approval Not Applicable.

Conflict of interest The authors declare that there are no conflict of interests and thank Figdraw for their support in drawing.

References

1. Zarone F, Di Mauro MI, Ausiello P, Ruggiero G, Sorrentino R (2019) Current status on lithium disilicate and zirconia: a narrative review. BMC Oral Health 19(1):134
2. Morimoto S, Rebello de Sampaio FB, Braga MM, Sesma N, Özcan M (2016) Survival rate of Resin and Ceramic Inlays, Onlays, and overlays: a systematic review and Meta-analysis. J Dent Res 95(9):985–994
3. Grewal Bach GK, Torrealba Y, Lagravère MO (2014) Orthodontic bonding to porcelain: a systematic review. Angle Orthod 84(3):555–560
4. El-Damanhoury HM, Gaintantzopoulou MD (2018) Self-etching ceramic primer versus hydrofluoric acid etching: etching efficacy and bonding performance. J Prosthodont Res 62(1):75–83
5. Borges GA, Sophr AM, De Goes MF, Sobrinho LC, Chan DCN (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthet Dent 89(5):479–488
6. Torres SM, Borges GA, Spohr AM, Cury AA, Yadav S, Platt JA (2009) The effect of surface treatments on the micro-shear bond strength of a resin luting agent and four all-ceramic systems. Oper Dent 34(4):399–407
7. Wille S, Lehmann F, Kern M (2017) Durability of resin bonding to lithium disilicate and zirconia ceramic using a self-etching primer. J Adhes Dent 19(6):491–496
8. Özcan M, Allahbeickaraghi A, Dündar M (2012) Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. Clin Oral Investig 16(1):15–23
9. Vichi A, Fabian Fonzar R, Carrabba M, Louca C, Scotti N, Mazzitelli C (2021) Comparison between Hydrofluoric Acid and single-component primer as conditioners on Resin Cement Adhesion to Lithium Silicate and Lithium Disilicate Glass Ceramics. Mater (Basel) 14(22):6776
10. Icochea AEL, Fernandes-Neto C, Bisaia A, Mondelli RF, Rizzante FAP, Furuse AY (2024) Evaluation of single-step self-etching ceramic primer and zirconia primer for bonding to zirconia-reinforced lithium silicate ceramic. Gen Dent 72(6):20–24
11. Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U (2019) New approaches in Bonding to Glass-Ceramic: self-etch glass-ceramic primer and Universal adhesives. J Adhes Dent 21(3):209–217
12. Çokakoğlu S, Nalçacı R, Altıntaş SH, Atıcı F (2024) Can a self-etching primer be effective in Bonding Aligner attachments to different types of ceramics? Turk J Orthod 37(1):30–35
13. Miranda LM, Caldas ATL, Moura DMD, Souza KB, Assunção IV, Özcan M (2024) Recycling CAD/CAM Lithium Disilicate Ceramic: Effect of Surface Treatment and Aging on the bond strength to Resin Cement. Int J Prosthodont 37(6):675–685
14. Vila-Nova TEL, Moura DMD, Araújo GM, Pinto RAS, Leite FPP, Melo RM (2022) Effect of Adhesive Resin Application on the durability of adhesion to CAD/CAM Glass-ceramics after either hydrofluoric acid etching or self-etch primer application. J Adhes Dent 24:279–289
15. Scientific Documentation Monobond Etch &, Prime https://www.ivoclar.com/de_li/eifu?brand=Monobond+Etch+%26+Prime[Accessed 1 January 2025]
16. National Center for Biotechnology Information (2024) PubChem Compound Summary for CID 11748636, Tetrabutylammonium fluoride dihydrofluoride
17. Chen B, Xu D, Li Z, Jing Y, Lin L, Li S (2022) Tissue distribution, excretion, and Interaction with Human serum albumin of total Bioflavonoid Extract from Selaginella Doederleinii. Front Pharmacol 13:849110
18. Chen J, Li T, Huang D, Gong W, Tian J, Gao X (2023) Integrating UHPLC-MS/MS quantitative analysis and exogenous purine supplementation to elucidate the antidepressant mechanism of Chaigui granules by regulating purine metabolism. J Pharm Anal 13(12):1562–1576
19. Chen B, Wang X, Zou Y, Chen W, Wang G, Yao W (2018) Simultaneous quantification of five biflavonoids in rat plasma by LC-ESI-MS/MS and its application to a comparatively pharmacokinetic study of Selaginella Doederleinii Hieron extract in rats. J Pharm Biomed Anal 149:80–88
20. Tian T, Tsoi JK, Matinlinna JP, Burrow MF (2014) Aspects of bonding between resin luting cements and glass ceramic materials. Dent Mater 30(7):e147–e162
21. Szep S, Gerhardt T, Gockel HW, Ruppel M, Metzeltin D, Heidemann D (2000) In vitro dentinal surface reaction of 9.5% buffered hydrofluoric acid in repair of ceramic restorations: a scanning electron microscopy investigation. J Prosthet Dent 83(6):668–674
22. Loomans BA, Mine A, Roeters FJ, Opdam NJ, De Munck J, Huysmans MC (2010) Hydrofluoric acid on dentin should be avoided. Dent Mater 26(7):643–649
23. Dönmez MB, Yucel MT, Kilic I, Okutan Y (2018) Novel ceramic primer vs conventional treatment methods: effects on roughness and bond strength of all-ceramic restorations. Am J Dent 31(5):249–252
24. Prado M, Prochnow C, Marchionatti AME, Baldissara P, Valandro LF, Wandscher VF (2018) Ceramic surface treatment with a single-component primer: Resin adhesion to glass ceramics. J Adhes Dent 20(2):99–105
25. Yu P, Wang XY (2021) Effects of Surface Treatment procedures on Bond Strength of Lithium Disilicate Glass Ceramic. Chin J Dent Res 24(2):119–124
26. Román-Rodríguez JL, Perez-Barquero JA, Gonzalez-AnguloE, Fons-Font A, Bustos-Salvador JL (2017) Bonding to silicate

ceramics: conventional technique compared with a simplified technique. J Clin Exp Dent 9(3):e384–e386
27. Dapieve KS, Aragonez GC, Prochnow C, Burgo TAL, Rippe MP, Pereira GKR et al (2021) Different etching times of a one-step ceramic primer: effect on the resin bond strength durability to a CAD/CAM lithium-disilicate glass-ceramic. J Adhes Dent 23(2):133–143
28. Poulon-Quintin A, Ogden E, Large A, Vaudescal M, Labrugère C, Bartala M et al (2021) Chemical surface modification of lithium disilicate needles of a silica-based ceramic after HF-etching and ultrasonic bath cleaning: impact on the chemical bonding with silane. Dent Mater 37(5):832–839
29. Elraggal A, Afifi R, Abdelraheem I (2022) Effect of erosive media on microhardness and fracture toughness of CAD-CAM dental materials. BMC Oral Health 22(1):191
30. Komagata Y, Ikeda H, Fujio Y, Nagamatsu Y, Shimizu H (2023) Effect of phosphoric acid and sodium hydroxide on cleaning and bonding of saliva-contaminated feldspar porcelain. J Prosthodont Res 67(1):132–137
31. Kukiattrakoon B, Thammasitboon K (2012) Optimal acidulated feldspathic porcelain: on shear bond strength to resin composite. Eur J Dent 6(1):63–69
32. Steinhauser HC, Turssi CP, Franca FM, Amaral FL, Basting RT (2014) Micro-shear bond strength and surface micromorphology of a feldspathic ceramic treated with different cleaning methods after hydrofluoric acid etching. J Appl Oral Sci 22(2):85–90
33. Martins ME, Leite FP, Queiroz JR, Vanderlei AD, Reskalla HN, Ozcan M (2012) Does the ultrasonic cleaning medium affect the adhesion of resin cement to feldspathic ceramic. J Adhes Dent 14(6):507–509
34. Dos Santos DM, Bitencourt SB, da Silva EV, Matos AO, Benez GC, Rangel EC et al (2020) Bond strength of lithium disilicate after cleaning methods of the remaining hydrofluoric acid. J Clin Exp Dent 12(2):e103–e107
35. Zhou W, Sarpong F, Zhou C (2022) Use of Ultrasonic Cleaning Technology in the whole process of Fruit and Vegetable Processing. Foods 11(18):2874
36. Kentish S, Ashokkumar M (2011) Ultrasound technologies for Food and Bioprocessing. Springer, New York, pp 1–12