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ORIGINAL RESEARCH

Effects of lactic acid etching on immediate and aged bond strength of resin-dentin bonding interface

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Abstract

To investigate the effects of lactic acid etching on the immediate and aged bond strength of the resin-dentin bonding interface, the resin-dentin bonding interface was evaluated 24 hours and 6 months later. A total of 42 isolated third molars were randomly divided into 6 experimental groups according to different lactate concentration (35%, 40%, 45%) and acid etching time (30 s, 45 s), with 37% phosphoric acid etching 15 s as a control. In each group, dentin samples were etched under different acidic conditions and bonded with Adper Single Bond 2 (3M ESPE) as directed. The immediate group was immediately stored in deionized water at 37 °C for 24 h, and the aging group was stored in artificial saliva at 37 °C for 6 months. Immediate and aged bond strengths were measured by a micro-tensile tester, and the specimen fracture surface was observed under a microscope. 14 isolated third molars were randomly divided into 7 groups, and each group was etched with acid. Collagen fibers morphology in dentin was examined after gradient dehydration with ethanol by scanning electron microscopy (SEM). Statistically, there was no difference between the resin-dentin immediate bonding strength of 35% lactic acid for 30 s and 37% phosphoric acid for 15 s, but the aged bond strength was greater than that of the phosphoric acid group. According to scanning electron microscope observations, the collagen fiber morphology in 35% and 40% lactate etching dentin 30 s groups was relatively intact compared with other groups. In conclusion, 35% lactic acid etching of dentin 30 s ensures both immediate and aged resin-dentin bond strength.

Keywords

Dentin; Resin; Micro-tensile bond strength; Stability

1. Introduction

Tooth decay is the most common dental disease, with about 2.3 billion people worldwide suffering from permanent tooth decay and 500 million children suffering from deciduous tooth decay in 2017 [1, 2]. Due to their favorable aesthetics and high operability, resin materials have become the most popular restorative materials. However, resin materials are constantly challenged by physical, chemical and biochemical conditions in the oral cavity [3]. Resin-dentin adhesive interfaces eventually fail to repair, resulting in secondary caries, so the average service life is about 5.7 years [4, 5]. Studies have shown that the collagen fiber network is exposed after acid erosion and demineralization of dentin, and partial collapse of the collagen fiber network as a resin penetration scaffold during the infiltration process of methacrylate resin monomer or the presence of water molecules will result in insufficient penetration of resin monomer, thereby causing the formed mixed layer to be a weak link in the resin-dentin bonding interface [6]. Moreover, the activation of endogenous matrix metalloproteinases (MMPs) during bonding will result in rapid degradation of collagen fibers over time [7]. Maintaining the structural integrity of the collagen web over time is crucial to the resin-dentine bonding interface durability. Therefore, to prevent collagen fiber network collapse during the bonding process, scholars [8] have conducted numerous studies and proposed the theory of selective demineralization of dentin.

Dentin can be selectively demineralized by reducing the etching agent concentration or shortening the etching time [9, 10]. Studies have shown that certain inorganic substances are retained in collagen fibers by reducing the phosphoric acid concentration or shortening the phosphoric acid etching time or using weakly acidic etching agents. Thus, immediate bonding improves [11–14]. However, there is no proof that aged bond strength is improved, and most previous studies have clinical operation difficulties and technical sensitivity, so it is difficult to apply in clinical practice. For this experiment, a weak acidity, low sensitization, high biosafety and non-toxic lactic acid is chosen. Although it can theoretically achieve selective demineralization, its application to resin-dentin bonding is limited. By studying different concentrations and times of acid etching, this experiment examines the effects of lactic acid on the immediate and aged bond strength of resin-dentin. The null hypothesis tested in this experiment is: ① There is no improvement in immediate or aged bond strength under different concentrations of lactic acid and different etching times. ② The use of lactic acid instead of phosphoric acid for acid etching does not preserve collagen fiber integrity.

2. Materials and methods

2.1 Material and solution configuration

2.1.1 Main materials and reagents

The composition, batch number, and manufacturer of the phosphoric acid, lactic acid stock solution, and other major materials used in this experiment are shown in Table 1.

2.1.2 Solution preparation

Directly diluting 85% of commercially available lactic acid with deionized water yields 35%, 40% and 45% of lactic acid. 37% phosphoric acid can be obtained by direct dilution of 98% commercially available phosphoric acid with deionized water. Storage at room temperature, away from light, and use within 1 month is recommended for the prepared solution.

2.2 Main instruments and equipment

The lot numbers and manufacturers of the main instruments used in this experiment are shown in Table 2.

2.3 Preparation and grouping of micro-tensile test specimens

Periodontal tissues were removed and stored at 4 °C in normal saline containing 0.02% sodium azide used within 1 month. On 42 of the third molars, enamel and superficial dentin were removed using a hard tissue microtome under running water. After exposing the middle dentin, 320, 600, 800, 1000, 1200 grit silicon carbide sandpaper was applied to polish the dentin surface for 20 s with flowing water. Shaked in an ultrasonic cleaner for 5 min, stored in normal saline, and used within 1 week. There were 6 experimental groups divided according to lactic acid concentration (35%, 40%, 45%) and acid etching time (30 s, 45 s), with 37% phosphoric acid etched for 15 s as the control group, with 6 dentin specimens per group. The experimental groupings are shown in Table 3. Different acid etching treatments were performed on dentin according to the experimental design. Rinse for 30 s after acid etching, then place on absorbent paper until there is no running water. Once the tooth surface has dried, apply 2–3 layers of adhesive to the dentin surface immediately with a small cotton swab fully impregnated with adhesive, rub repeatedly for 15 seconds, gently dry with an air gun for 5 seconds, then light cure for 10 seconds. Stack resin blocks by layer curing, light cure for 20 s, and the overall resin block thickness is 4 mm. Samples were fixed to a hard tissue microtome, and specimens with a cross-sectional area of 1 mm^2 and a length of 8 mm were cut. There are 20 test pieces in each group, of which 10 test pieces are used for the immediate bond strength test (groups A–G), and

TABLE 1. Materials used in this study.

Material	Composition	Lot no.	Manufacturer
Phosphoric acid	Phosphoric acid (98 wt.% in H_2O)	20210506	Xilong Science Co., Ltd., China
Lactic acid	Lactic acid (85 wt.% in H_2O)	20210618	Maikelin Company, China
Artificial saliva	Potassium chloride magnesium chloride polyvinyl alcohol	200611	Xinheng Co., Ltd., China
Filtek Z350 XT	Bis-GMA, UDMA, TEGDMA, bis-EMA, silica/zirconia nanoparticles, nanoclusters of silica/zirconia nanoparticles in a total of 78.5% wt	NE52111	3M ESPE, St. Paul, MN, USA
Adper Single Bond 2	HEMA, Bis-GMA, Glycerol 1,3-dimetharcylate, Diurethane dimethacrylate, water ethanol, photoinitiators, silanized silica, polyacrylic and itaconic acid copolymer	NC83276	3M ESPE, St. Paul, MN, USA

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; Bis-EMA, ethoxylated-bisphenol A glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

TABLE 2. Instruments used in this study.

Instrument	Lot no.	Manufacturer
Microtensile strength tester	-	BISCO, USA
SmartLite Focus Pen-Style LED Curing Light	-	Dentsply, USA
Hard tissue slicer	SM2600	EXAKT, Germany
Scanning electron microscope	-	Hitachi, Japan
Stereomicroscope	-	MOTIC CHINA GROUP CO., LTD., China

Abbreviations: LED, light emitting diode.

10 test pieces are used for the aged bond strength test (groups A1–G1).

TABLE 3. Lactic acid concentration and acid etching time in Group.

Group		Description of acid etching concentration and time
24 h	6 mon	
Group A	Group A1	37% phosphoric acid 15 s
Group B	Group B1	35% lactic acid 30 s
Group C	Group C1	35% lactic acid 45 s
Group D	Group D1	40% lactic acid 30 s
Group E	Group E1	40% lactic acid 45 s
Group F	Group F1	45% lactic acid 30 s
Group G	Group G1	45% lactic acid 45 s

For the immediate bond strength test, specimens in the immediate group (groups A–G) were stored in deionized water at 37 °C for 24 hours and kept at a constant temperature. For the aged group (groups A1–G1), specimens were stored in artificial saliva at 37 °C at a constant temperature, replaced once a week and stored in a glass bottle for 6 months in the dark for aged bond strength test.The equipment used in the experiment is shown in Fig. 1.

FIGURE 1. The experimental process of the study. (a) Hard tissue slicer. (b,c) Micro-tensile strength test.

2.4 Immediate and aged bond strength test

The specimens of each immediate group were placed on the microtensile tester, loaded at a fixed speed, broken, and the maximum micro-tensile bond strength (μTBS) was recorded. The bonding area was calculated using an electronic vernier caliper. After 6 months, each aged group was tested using the same method. Using the formula: bond strength (MPa) = maximum microtensile strength (N)/bonding area of the test piece (mm^2), the bonding strength of the test piece is calculated.

2.5 Statistical analysis

For statistical analysis, SPSS 25.0 software package (SPSS Software, version 25.0, IBM Corp., New York, USA) was used. Experimental data were submitted to a normality test and presented a normal distribution. SNK-q (Student-Newman-Keul) were used for multiple comparisons. The t-test was performed to compare whether the mean difference between the groups before and after aging was statistically significant. The test level was $\alpha = 0.05$, and $p < 0.05$ indicated statistical significance.

2.6 Observation and analysis of the fracture mode of the specimen

After the μTBS test, the two fractured ends were examined with a stereomicroscope at 35 magnification. Fracture modes on the dentin sides of the specimens were considered and classified into the following categories: adhesive failure, cohesive failure in dentin, cohesive failure in composite resin, and mixed failure [15].

2.7 SEM observation of demineralized dentin

14 selected and collected teeth were placed on a hard tissue microtome. After exposure to the middle dentin, they were divided into 7 groups (A–G groups, n = 2) according to different acid etching methods. Each group received the same acid etching concentration and acid etching time as the immediate microtensile test set. The treated specimens were dehydrated in ethanol gradients and stored in hexamethyldisilazane for 20 min. After drying for 24 h, a scanning electron microscope at 5000 magnification and 20,000 magnification was used to examine the morphology of dentinal tubules and collagen fibers.

3. Results

3.1 Immediate and aged micro-tensile bond strength results

As shown in Fig. 2 and Table 4 there was no significant difference in the bond strength between the 35% lactic acid 30 s group and the 37% phosphoric acid 15 s group (control group) ($p > 0.05$) in the immediate group. All other experimental groups had a lower immediate bond strength than the control group, there is a significant statistical difference ($p < 0.05$), however, all of them are greater than 20 MPa, which meets clinical resin bond strength requirements. Compared to the immediate groups, the aged bond strength of each aged group was significantly decreased, and the results were statistically different ($p < 0.05$). Among them, the 35% lactic acid 30 s group and the 40% lactic acid 30 s group were significantly higher than the control group ($p < 0.05$). There were no statistical differences between the 35% lactic acid 30 s group and the 40% lactic acid 30 s group ($p > 0.05$). The aged bond strength of the other experimental groups was significantly lower than that of the control group ($p < 0.05$).

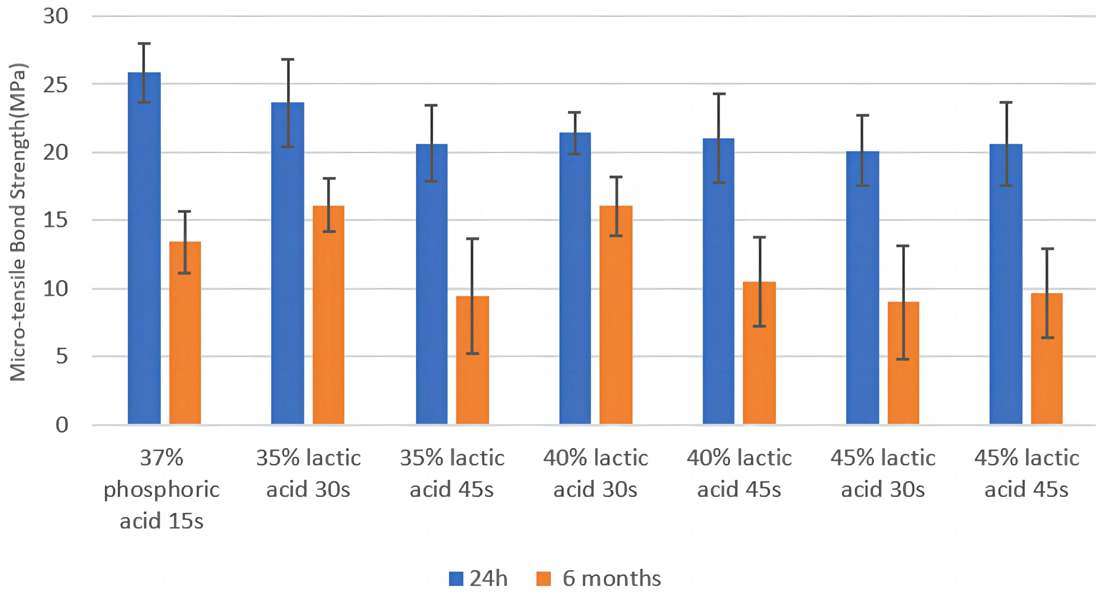

FIGURE 2. Statistic graph of immediate and aged micro-tensile bond strength.

TABLE 4. Statistic table of immediate and aged micro-tensile bond strength.

Groups	Micro-tensile Bond Strength (MPa; n = 10, $\bar{x} \pm s$)	
	24 h	6 mon
37% phosphoric acid 15 s	25.88 ± 2.15^{Aa}	13.44 ± 2.29^{Ab}
35% lactic acid 30 s	23.63 ± 3.16^{Aa}	16.12 ± 1.96^{Bb}
35% lactic acid 45 s	20.66 ± 2.77^{Ba}	9.46 ± 4.25^{Cb}
40% lactic acid 30 s	21.45 ± 1.53^{Ba}	16.08 ± 2.14^{Bb}
40% lactic acid 45 s	21.08 ± 3.25^{Ba}	10.52 ± 3.26^{Cb}
45% lactic acid 30 s	20.13 ± 2.60^{Ba}	9.00 ± 4.16^{Cb}
45% lactic acid 45 s	20.62 ± 3.08^{Ba}	9.68 ± 3.30^{Cb}

At a test level of $\alpha = 0.05$, $p < 0.05$, different lowercase letters in rows and different majuscule in columns were statistically significant.

3.2 Analysis results of fracture mode

The immediate and aged fracture modes of the 7 groups of specimens are dominated by interface fracture, with less cohesive fracture and mixed fracture (Figs. 3,4).

3.3 Analysis of scanning electron microscope results

The first set of arrows indicates the stain covering the dentin tubule surface without acid treatment. As shown in Fig. 5, scanning electron microscope images were taken of the dentin surface of each group (groups A to G). When magnified 5000 times, the stain layer of groups A to G is completely removed, and the dentin tubules (pointed by the arrow) are open. At 20,000 times, collagen fibers were completely exposed in the group A. The morphology and integrity of collagen fibers in the group B and group D were better preserved. Several collagen fiber ends split in the rest of the group, as indicated by arrow points.

4. Discussion

4.1 Influence of selective demineralization of dentin on bond strength

Resin restorations fail due to a lack of integrity in the mixed layer at the dentin-resin adhesive interface, which is primarily due to matrix metalloproteinases (MMPs) and cysteine cathepsin degrading the bare collagen fibers in the dentin. Collagen fiber networks as a result of water evaporation [16–18], thus preventing resin monomer penetration and resulting in reduced resin aged bond strength. To support the network structure and prevent collapse of collagen fibers during acid etching, inorganic substances must be retained in collagen fibers. Previous study found that 17% ethylenediaminetetraacetic acid (EDTA), 2% doxycycline (DO), and 2% chlorhexidine (CHX) could inhibit MMPs [19]. Since dentin of deciduous teeth have less inorganic matter and more organic matrix than that of permanent teeth, MMP inhibitors have obvious advantages in deciduous teeth. To investigate whether different MMPs inhibitors can improve the aging bonding strength of dentin-resin, Parsaie Z [20] took deciduous teeth dentin as the research object and concluded that CHX was applied to the dentin after acid etching in aging samples after 6 months, the bond strength

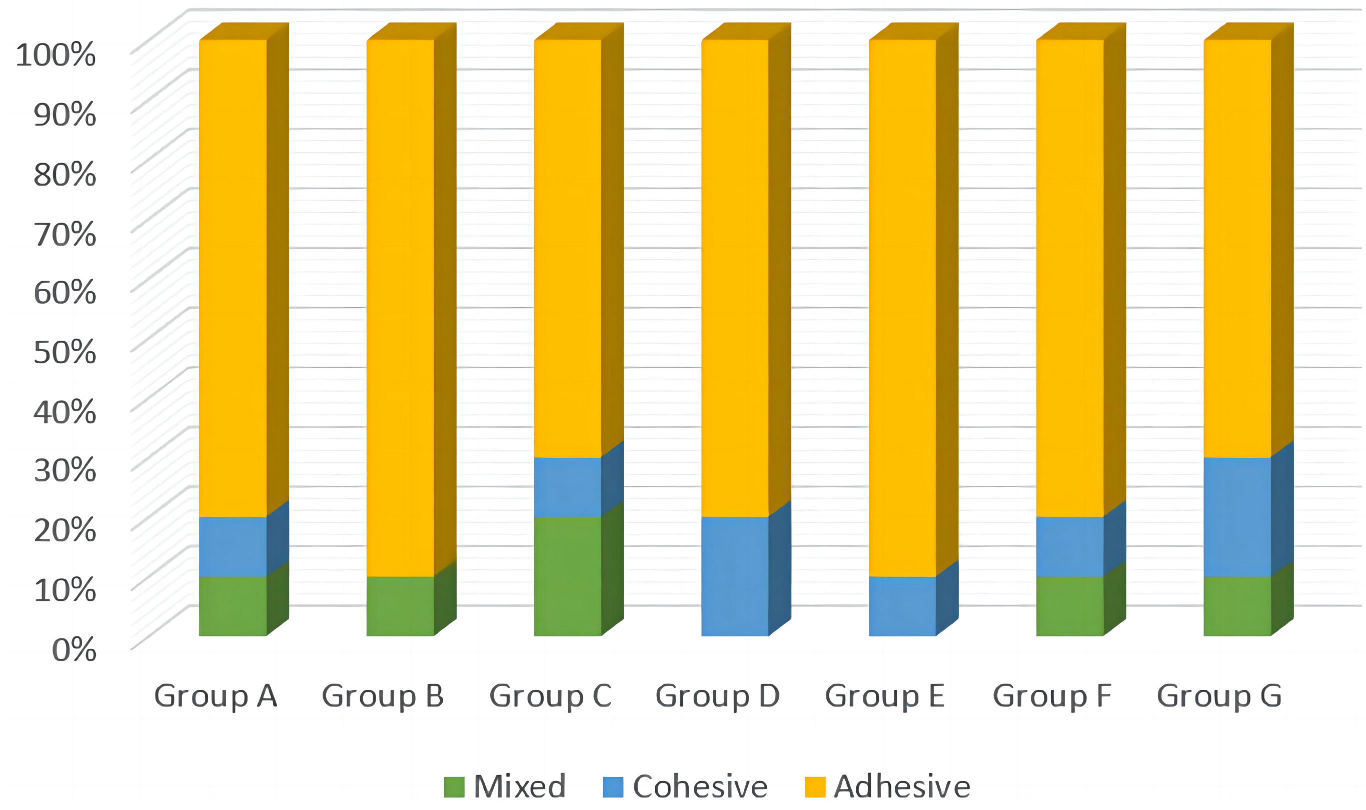

FIGURE 3. Statistic graph of the immediate fracture mode of each group of specimens (Example (%)).

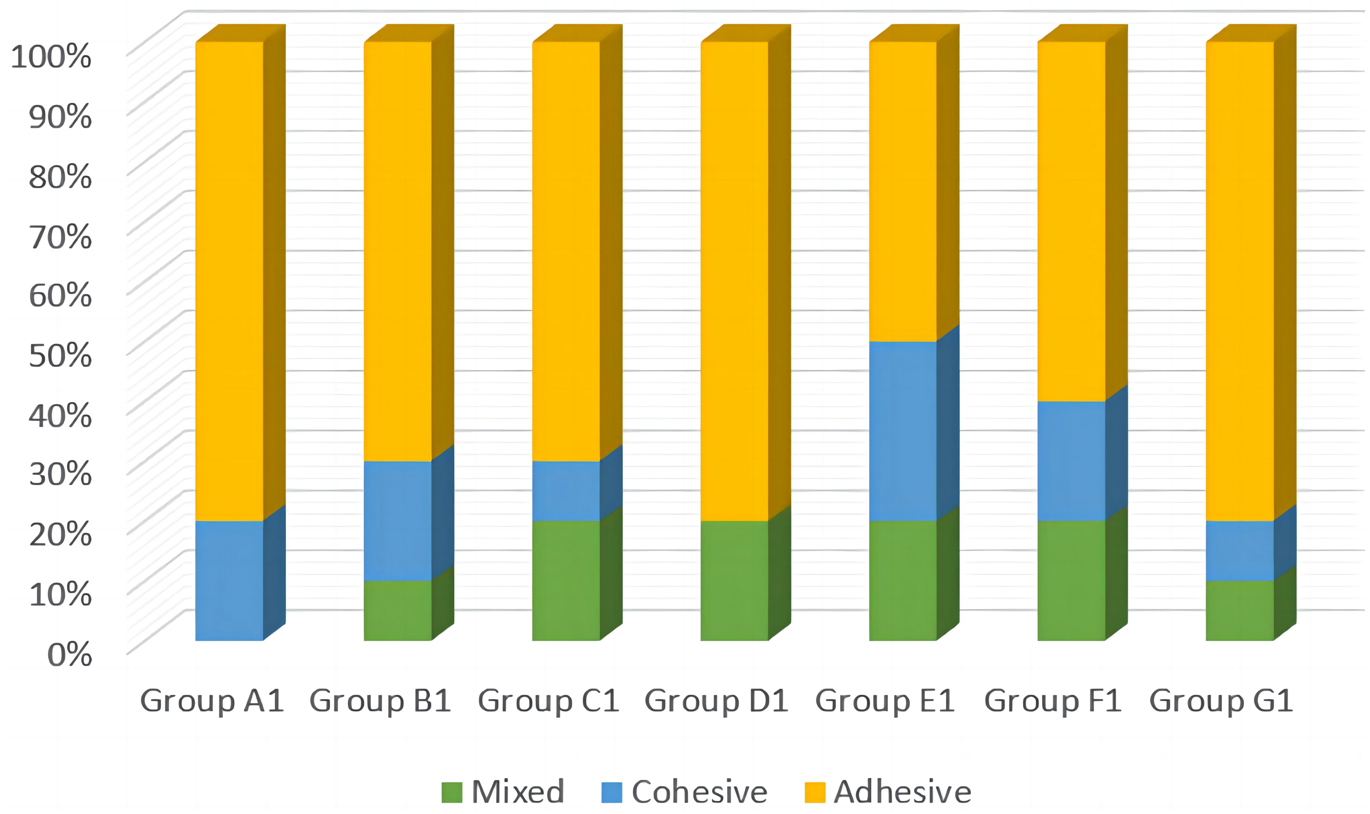

FIGURE 4. Statistic graph of aged fracture mode results of each group of specimens (Example (%)).

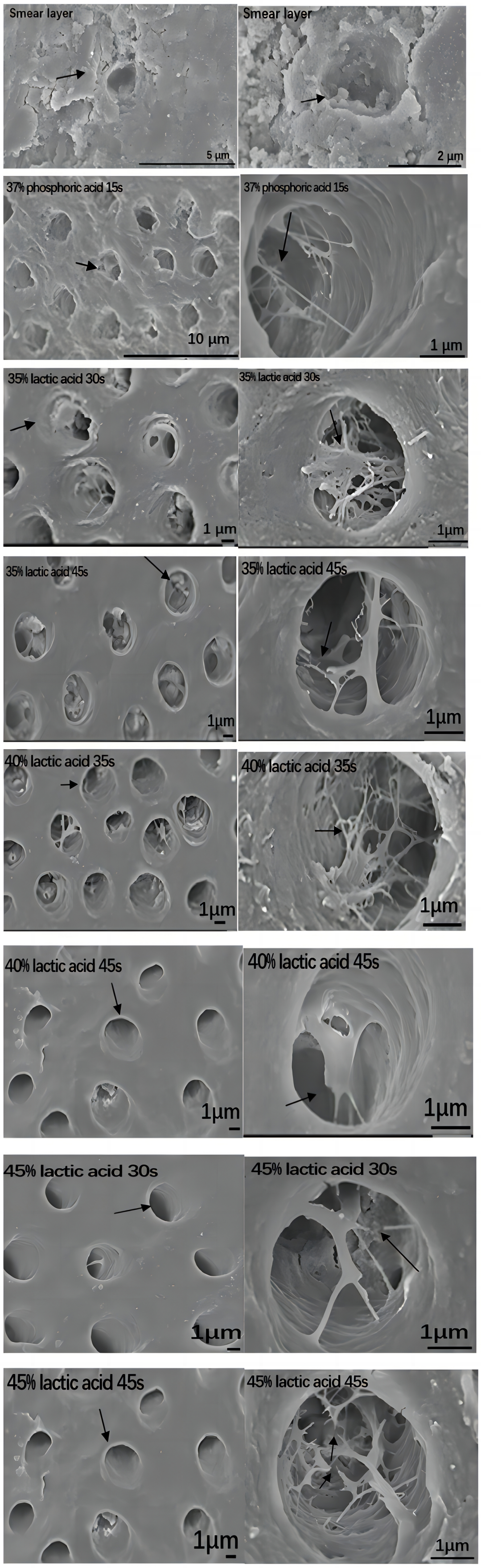

FIGURE 5. Scanning electron microscopic images of dentin interface in each group.

of dentin was significantly higher than that of control group, but there was no statistical difference between EDTA group and DO group. Galo's findings indicate that the shear bond strength of the aged samples after 1 month and 6 months after CHX treatment was lower than that of aged samples without CHX treatment [21]. The author proposed that the possible reason for this result was that CHX was hydrophilic and easily exudated from dentin. Moreover, the penetration of dentine tubules by hydrophilic monomers will be adversely affected, affecting the stability of the mixed layer. More studies are required to confirm whether CHX's long-term effect as an MMPs inhibitor is sufficient for clinical use. To maintain the integrity of the spatial structure of collagen fiber, scholars proposed the use of a collagen fiber crosslinking agent. proanthocyanidin (PA) can destroy the polymerization of resin monomers, resulting in low bonding strength and bonding failure, so PA is not suitable to be added to an adhesive. Rey [22] added PA to phosphoric acid on this theoretical basis. According to *in situ* zymogram, MMPs in all groups showed higher activity after acid etching, but there was no difference among all groups, indicating PA addition did not reduce MMP activity. As a result of the crosslinking activity of PA, the diameter of the tubes at the depth of the dentin tubules decreased in the PA group. It was suggested, however, that further studies were needed to determine whether the use of PA increased resin-dentin adhesive interface stability. Both MMPs inhibitors and crosslinking agents have technical sensitivity and effect instability, which need to be verified by further experiments.

Scholars proposed selective demineralization to improve adhesive interface, scholars stability [19, 23]. To verify the feasibility of selective demineralization of dentin, previous scholars have made various attempts. Li Bingqing [12] changed the concentration of phosphoric acid etching and found that the 5% phosphoric acid group had the highest bonding strength, which was significantly different from other groups. At the same time, energy dispersive X-ray analysis (EDX) showed that the calcium content on the surface of dentin were significantly higher after acid etching with low concentration phosphoric acid than after acid etching with high concentration phosphoric acid group. Consequently, the author concluded that reducing the concentration of phosphoric acid to a certain extent can result in selective demineralization of dentin. However, this experiment only proves that using 5% phosphoric acid enhances the immediate bond strength, but it has not been evaluated whether the aged bond strength has improved. By reducing the time that phosphoric acid acts on dentin, Thiago [13] found that after 24 hours and 6 months, the bond strength of the acid etched 3 s group was significantly higher than that of the other groups. Therefore, the authors concluded that 32% phosphoric acid etching for 3 s improved the immediate and aged bond strength of resin bonding. At the same time, there was no statistical difference in the residual calcium ion content between the non-etched group and the acid-etched 3 s group measured by EDX. This result may be explained by the short phosphoric acid etching time and intact inorganic substances in collagen fibers. However, the short phosphoric acid etching makes clinical operation difficult. Mohammed [14] found that 30% lactic acid etching for 10 s completely removed the smear layer from the scanning electron microscope results from different concentrations of lactic acid etching dentin. As the lactic acid concentration increases, the diameter of dentinal tubules increases. Also, the shear strength obtained from different concentrations of lactic acid acting on the dentin surface for 10 s is all less than 20 MPa, which is consistent with the pre-experiment results of this experiment: 20%, 30%, 40% and 50% of lactic acid etched the dentin surface for 15 s, and the immediate bond strength was less than 20 MPa. The bond strength of each group after acid etching for 30 s increased significantly, and the immediate bond strength reached more than 20 MPa in the 40% lactic acid etching group for 30 s. According to most scholars, clinical resin bonding requires bond strength greater than 20 MPa [24, 25], while fourth and fifth generation adhesives can reach more than 20 MPa [26]. Therefore, in this experiment, 20 MPa is used as the boundary value, and the lactic acid concentration is 35%, 40%, 45% and the acid etching time is 30 s and 45 s. To investigate the changes in immediate and aged bond strength after different concentrations of lactic acid were acted on dentin at different times. Microtensile bond strength test is considered to be the most suitable method for testing bond strength because of standard operating procedures, wide application and versatility [27]. This study showed that 35% lactic acid etched dentin for 30 s not only guaranteed the immediate bonding strength, but also improved aged bond strength, proving the first null hypothesis incorrect. Scanning electron microscope results showed a relatively intact collagen fiber structure in 35% and 40% lactate eroded dentin 30 s group, possibly due to the partial retention of inorganic substances in the collagen fiber, so the second null hypothesis was not valid.

4.2 Analysis of fracture modes of immediate and aged specimens under different acid etching conditions

The fracture modes are divided into adhesive fracture, cohesive fracture and mixed fracture. Bonding surface fracture means that the fracture occurs between the resin and the adhesive or between the dentin and the adhesive, which indicates that the adhesive force is less than the cohesive force of the dentin and the cohesive force of the resin, and it is the most reflective of the actual adhesive strength of the dentin fracture mode [28]. Sano [29] found that almost all interface failures occurred when the bonding area was less than 2 mm^2. This is due to the fact that the strength of the resin and dentin themselves is much higher than the bond strength between the two, and because of the small bonding area of the micro-tensile test samples, the vast majority of the sample fracture occurs at the bond interface, which minimizes the effect of cohesive destruction of the dentin and resin on the test results. This is consistent with the results obtained in this experiment. In this experiment, the fracture surfaces of the fractured specimens in the immediate group and the aging group were analyzed by stereomicroscopy, and it was concluded that the fracture patterns of the immediate and aging specimens in each experimental group were dominated by the fracture of the adhesive surface, which indicated that the weak area of the resin-dentin bond was located at the adhesive interface. Although the microtensile test results concluded that 35% lactic acid etching 30 s can ensure the immediate bond strength while improving the aging bond strength, but this group of immediate and aging specimen fracture is still dominated by the adhesive surface fracture, this is because the depth of dentin acid etching and demineralization is greater than the depth of penetration of the adhesive resin, the bottom of the mixed layer is not wrapped in the adhesive resin and exposed collagen fibers exposed, the exposed collagen fibers are susceptible to degradation by water, enzymes, bacteria, temperature and stress, the free water molecules can promote the hydrolysis of the mixed layer, which will lead to the failure of the resin bonding repair [30]. Therefore, although 35% lactic acid etching for 30 s can relatively retain the integrity of the collagen fiber network, these unfavorable factors affecting the degradation of collagen fibers are still present, and the adhesive interface is still a weak area of the resin-dentin adhesive bonding. Therefore, maintaining the integrity of collagen fibers for a long time and prolonging the service life of the resin are still the difficulties in the current research.

Since this study is a laboratory study, it remains to be further investigated whether lactic acid etching applied to clinical manipulation will yield the same results. Also whether the inorganic material is partially retained in each experimental group has not been further investigated. Therefore, EDX will be used to further explore the determination of the amount of residual calcium ions in dentin after lactic acid is applied to dentin at different concentrations and times. This will be combined with transmission electron microscopy to observe the distribution of inorganic substances in collagen fibers. Ultimately, it will be verified whether lactic acid can achieve dentin selectivity and whether the inorganic material is partially retained during the acid etching process.

5. Conclusions

According to this study, 35% lactic acid etched dentin for 30 s improved resin-dentin aged bond strength while ensuring immediate bond strength. However, this study was conducted *in vitro*, to verify whether lactic acid can extend service life in the oral cavity, further clinical studies are necessary.

AVAILABILITY OF DATA AND MATERIALS

The data are contained within this article.

AUTHOR CONTRIBUTIONS

ZGZ, YYM—conception and design. QH—provision of study materials, data analysis and interpretation, manuscript writing. ZGZ—final approval of manuscript. All authors contributed to editorial changes in the manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Informed consent was obtained from each patient. The study received ethical exemptions from the Ethics Committee of the

Affiliated Stomatological Hospital of Nanchang University.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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