Universal Adhesives' Bond Durability to Primary Enamel in Different Application-Modes

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Background and Purpose

The increased demand for simple and efficient adhesive systems made current multimode universal adhesives of great interest in pediatric dentistry [1]. These are mainly characterized by a reduction in the number of application steps, hence limiting the duration of treatment during restorative procedures, together with achieving good dentin adhesion [2]. However, application protocols for universal adhesives have not been defined precisely for primary enamel [3]. Furthermore, the effect of aging was ignored, which lead to reporting higher bond strength values than the real ones due to overlooking the effect of factors, such as thermal stresses and normal daily functions on the bond strength [4]. Thus, our aim was to evaluate the effect of phosphoric acid etching time on the microtensile bond strength (µTBS) of universal adhesives to primary enamel and its stability after aging.

Materials and Methods

Upon the approval of the Ethical Committee, Faculty of Medicine, Justus-Liebig-University Giessen (AZ 143/09), freshly extracted, at least one-surface caries-free, primary molars were collected and divided in mesiodistal direction to increase the sample size. Tooth halves (n=101) were then randomly assigned to three groups, depending on the type of universal adhesive used. Aprismatic enamel was removed with a fine diamond stone (diamond FG878EF, 012G, Busch) under water coolant (Fig. 1a) and groups were further subdivided according to the application time of phosphoric acid. Samples were then incubated in distilled water at +37°C for either 24h (Phase I), or 6 months (Phase II) after the addition of adhesives and composite resin (see Table 1).

Group / Subgroup	Scotchbond Universal Adhesive (3M™ Oral Care)	Clearfil Universal Bond Quick (Kuraray Noritake)	iBond Universal Adhesive (Heraeus Kulzer)
Phase I (24h aging)			
No enamel conditioning with 36 % H_3PO_4: 0 s	SU-SG1 (n=57)	CU-SG1 (n=58)	iBU-SG1 (n=67)
Enamel conditioning with 36 % H_3PO_4: 15 s	SU-SG2 (n=62)	CU-SG2 (n=61)	iBU-SG2 (n=62)
Enamel conditioning with 36 % H_3PO_4: 30 s	SU-SG3 (n=56)	CU-SG3 (n=62)	iBU-SG3 (n=62)
Phase II (6 months aging)			
No enamel conditioning with 36 % H_3PO_4: 0 s	SU-SG1 (n=79)	CU-SG1 (n=74)	iBU-SG1 (n=76)
Enamel conditioning with 36 % H_3PO_4: 15 s	SU-SG2 (n=75)	CU-SG2 (n=73)	iBU-SG2 (n=71)

Tab. 1: Universal adhesives used and their application modes. H_3PO_4: Phosphoric acid (DeTrey®Conditioner 36, Dentsply DeTrey GmbH), n: number of sticks/ group.

1. Preparation of Samples

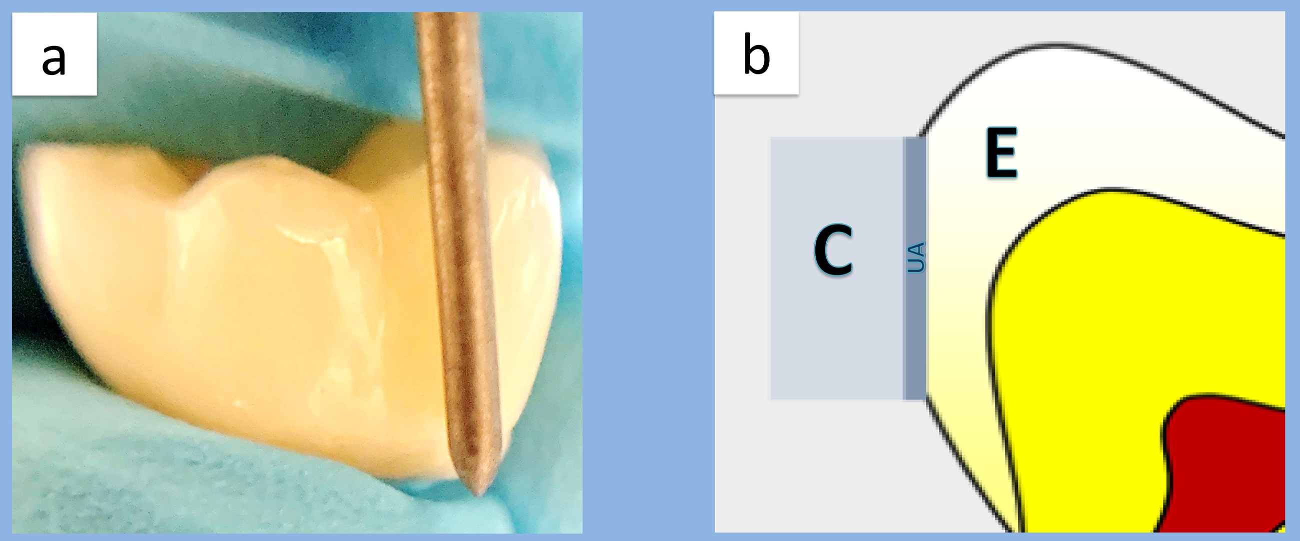

Fig. 1a: Removal of the aprismatic enamel layer with a fine diamond stone.
Fig. 1b: Schematic representation of the application of universal adhesives with subsequent build-up of tooth halves with resin composite (Filtek™ Z250, 3M™ Oral Care) in incremental technique (approx. 6 mm; E: enamel, UA: universal adhesive, C: composite resin). Specimens were then stored for 24h (Phase I) or 6 months (Phase II) at +37 °C in distilled water.

Materials and Methods (cont.)

2. Production of Sticks and µ-TBS Testing

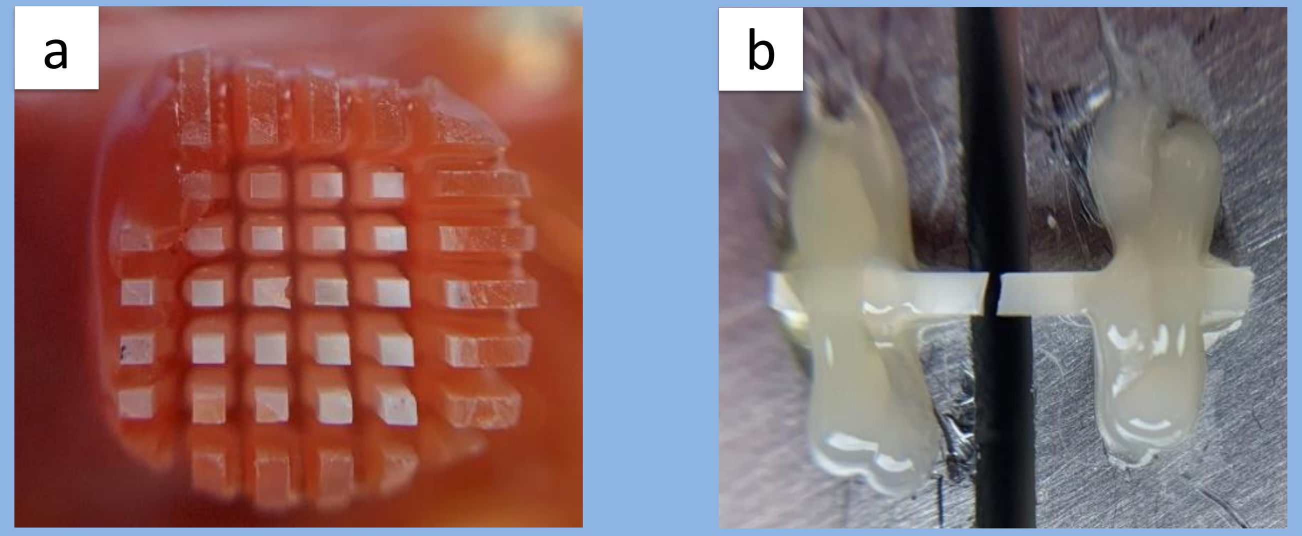

Fig. 2a: Sawing of tooth halves in mesiodistal and bucco-oral directions (IsoMet Highspeed Pro, Buehler; sawed sticks were 0.7 mm × 0.7 mm in dimension).
Fig. 2b: Fixation of sticks with Dyract Flow (Dyract Flow, Dentsply DeTrey GmbH) to the holder of the testing machine (TC-550, Syndicad; µ-TBS; testing parameters: maximum force = 50 N, crosshead speed = 1 mm / min).

3. Analysis of Fracture Modes under Light Microscope

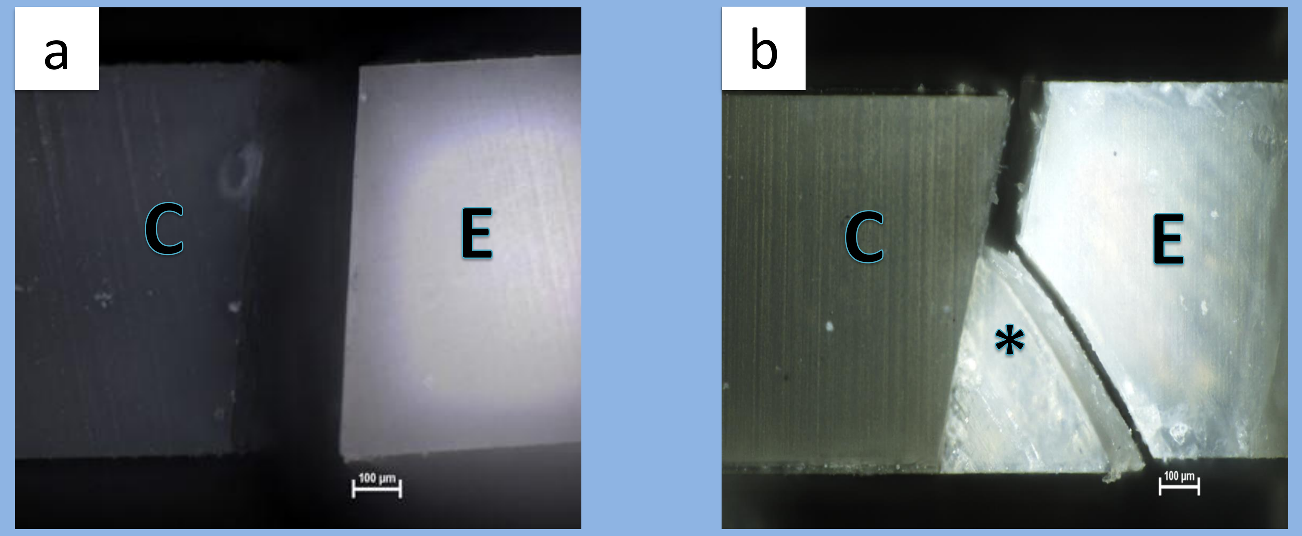

Fig. 3: Analysis of fracture modes with Light Microscope (AZ100M, Nikon) at 24x magnification.
Fig. 3a: Adhesive fracture (C: composite resin, E: enamel).
Fig. 3b: Mixed fracture (enamel-adhesive; star: fractured enamel fragment).

Results

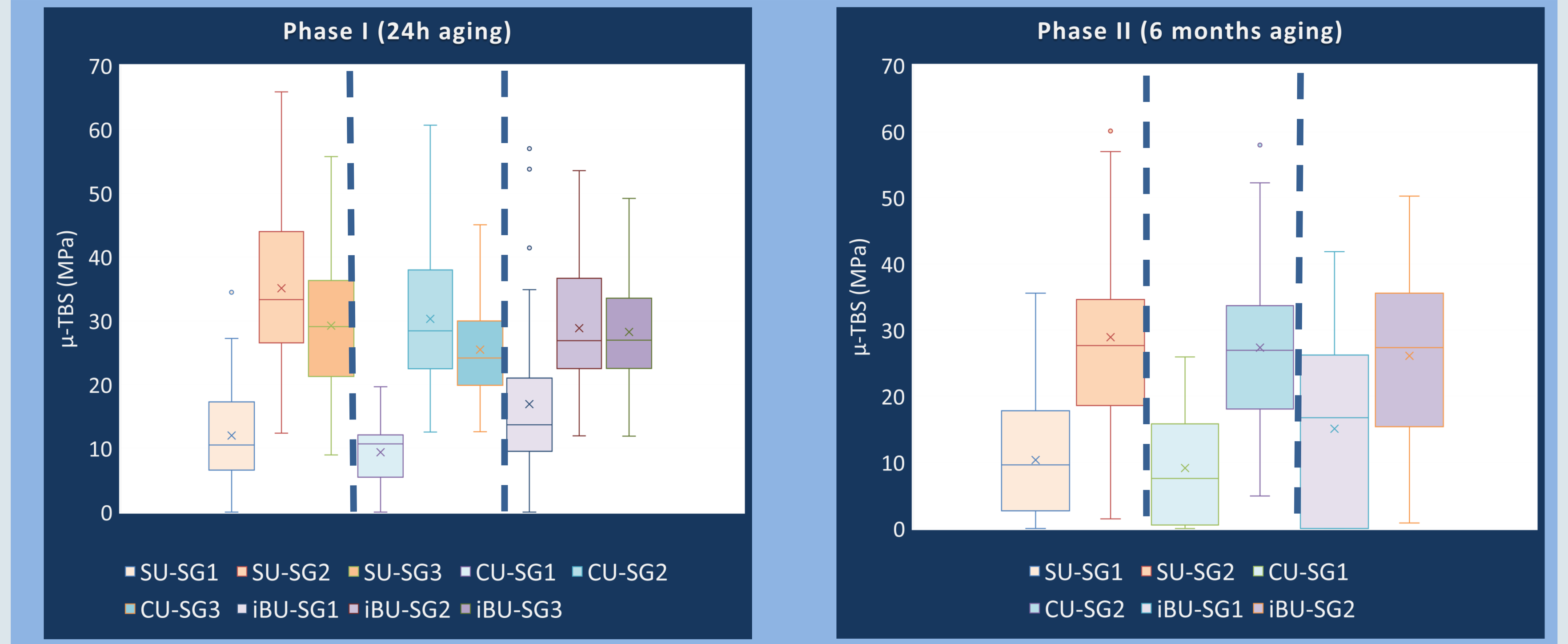

Fig. 4: Boxplot representation of the statistical analysis of µ-TBS data (SPSS 26.0, IBM Statistics, Armonk, NY, USA) using linear mixed model (restricted maximum likelihood, REML) and SIDAK post hoc tests (p<0.05). Left: Phase I (24h aging), right: Phase II (6 months aging).

Phosphoric acid conditioning improved the bond strength of composite resin to enamel for all universal adhesives used significantly ($p<0.001$). No significant difference was observed between groups etched for 15 s and 30 s for all adhesives used ($p>0.05$). Significant differences were determined only between the groups CU (µ-TBS value in MPa: 9.3; 95% CI [7, 11.7]) and iBU (16.9; [13.5, 20.3]) in SG1 ($p=0.029$) and between the groups SU in SG2 (34.9; [31.4, 38.5]) and CU in SG3 (25.5; [22.7, 28.3]) ($p=0.005$). µ-TBS values of all subgroups were decreased after aging, yet the decline was only significant in SU-SG2 (ANOVA, $p=0.014$).

Results (cont.)

Analysis of Fracture Modes

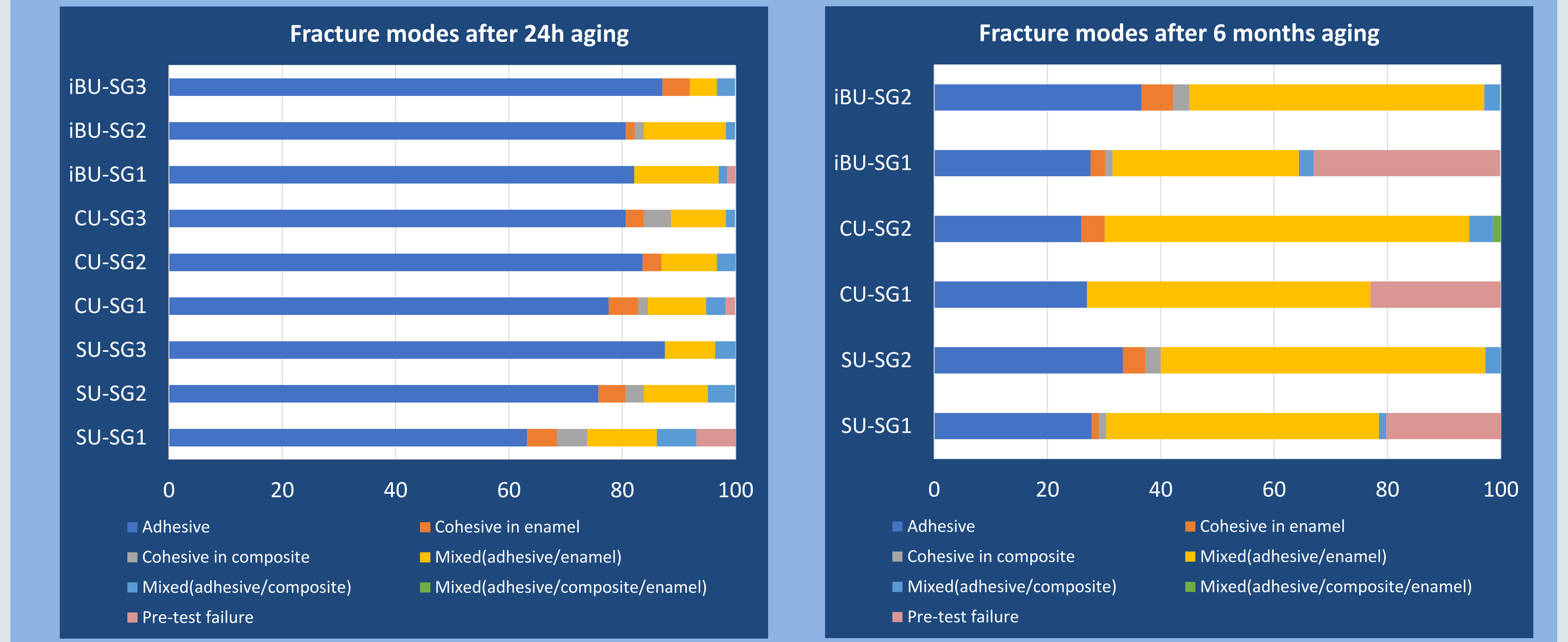

Fig. 5: A graphic representation of the fracture modes in Phase I (left; 24h aging) versus Phase II (right; 6 months aging) in distilled water. Different types of fractures are presented in % and are differentiated through color coding.

In Phase I, the most common fracture mode for all universal adhesives was the adhesive fracture (78.7 %), followed by mixed fracture (enamel-adhesive; 10.6 %). While in Phase II, the most common fracture mode for all universal adhesives was the mixed fracture (enamel-adhesive; 50.7 %), followed by the adhesive fracture (29.7 %).

Evaluation under the Scanning Electron Microscope (SEM)

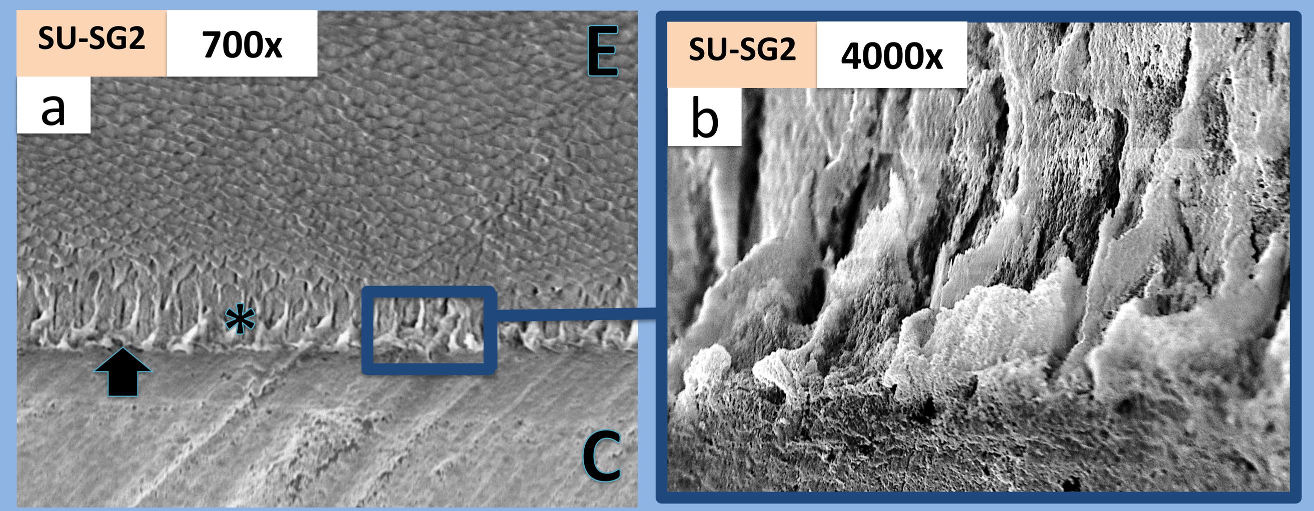

Fig. 6: Exemplary SEM images of the adhesive zone of a specimen with enamel conditioning. For preparing samples for SEM images, the enamel of all specimens was demineralized with 36 % H_3PO_4, dried and sputtered with gold.
Fig. 6a: The adhesive layer is clearly visible without noticeable damage, and numerous resin tags can be seen (C: composite resin, E: enamel, star: resin tags, arrow: adhesive joint).
Fig. 6b: Resin tags are well formed and run into the clearly visible enamel prisms.

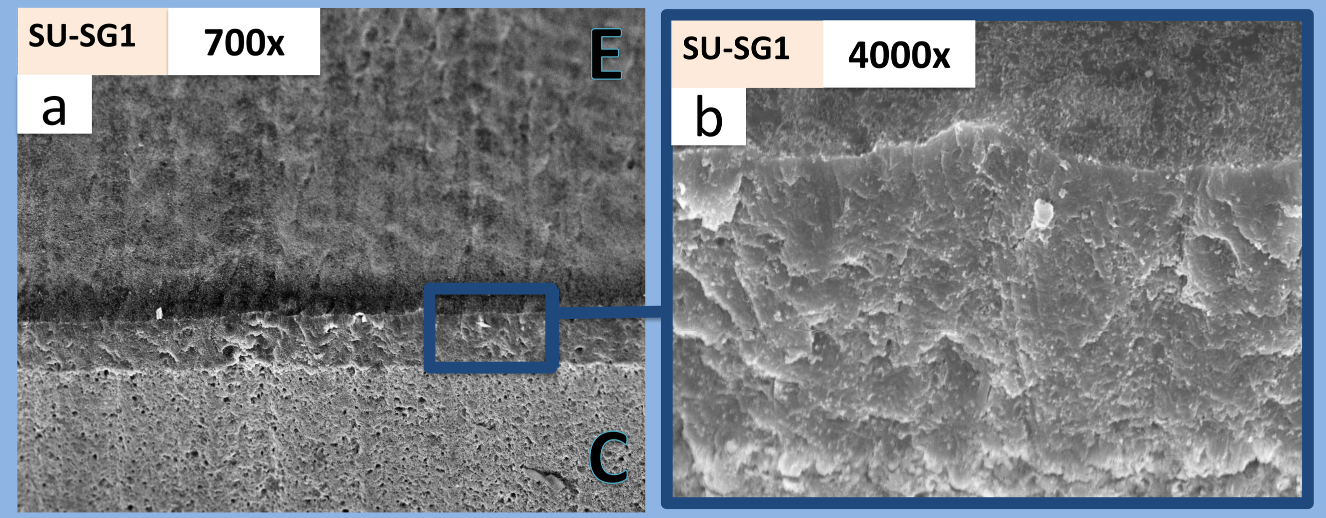

Fig. 7: Exemplary SEM images of the adhesive zone without enamel conditioning.
Fig. 7a: The adhesive layer is clearly visible with several cracks and voids (C: composite resin, E: enamel, arrow: adhesive joint).
Fig. 7b: Resin tags appear to be rudimentary.

Conclusions

1. Etching with phosphoric acid remains the gold standard for bonding universal adhesives to primary enamel.
2. Etching time showed no significant effect on µ-TBS.
3. Aging for 6 months in distilled water can affect the µ-TBS values when SU applied in 15s etching mode.

References

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