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Should statistical analysis of bond-strength data include or exclude cohesive failures?

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ABSTRACT

Objectives: The aim was to investigate shear bond strengths and failure modes of four self-etch bonding agents to bovine dentin and enamel and to compare evaluation of data sets with or without exclusion of cohesive failure specimens.

Methods: Composite-cylinders were bonded perpendicularly to bovine dentin and enamel surfaces. Shear-strengths were measured 24 h post-bonding of: Scotchbond Universal® (**SBU**, 3 M), OptiBond™ XTR (**OBXTR**, Kerr), OptiBond™ universal (**OBU**, KaVo-Kerr) and Prime & Bond active® (**PBA**, Dentsply-Sirona). Analysis of overall data was made via a linear mixed-model. This was repeated after exclusion of specimens associated with cohesive failures.

Results: When both adhesive and cohesive failures were considered, OBU and OBXTR showed comparable dentin and enamel bond strengths, whereas lower strengths were found on enamel for SBU ($p < 0.001$) and PBA ($p = 0.015$). For OBXTR higher shear strengths were measured for specimens associated with cohesive failures. When cohesive failures were excluded, the majority of shear bond strengths of adhesive failure specimens were only slightly different from overall results. However, uniquely with OBXTR dramatically lower shear bond strengths were found for dentin substrate.

Significance: After exclusion of cases with cohesive failures OBXTR adhesive fell behind other materials in the sequence of average shear strengths. This did not reflect the actual performance of the material. Therefore, in statistical analysis we do not recommend exclusion of data based on a specific fracture mode.

1. Introduction

Shear bond measurements, like all in-vitro experiments, should be performed under standardized conditions, as recommended in the literature, so that the results of different investigators can be compared with each other [1,2]. Regarding shear bond experiments it has long been debated if

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pre-test, mixed and cohesive failures should be included or excluded from the evaluation. In some studies, all shear bond strengths, whether resulting from adhesive, mixed or cohesive failures, were evaluated, e.g., by McLean et al. [3]; other authors recommend to exclude certain failure modes from the evaluation [4].

In 2009, the Academy of Dental Materials (ADM) held its annual meeting in Portland, Oregon, USA, concerning "Adhesion in Dentistry - Analyzing Bond Strength Testing Methods, Variables and Outcomes." The main objective of this meeting was to critically review different test methods used for dentin and enamel bonding studies and to identify important variables to be considered and reported in bond strength studies [5]. As a further consequence the ADM has published a series of guidance papers on adhesion science, listed in Roeder et al. [2] also including a recommendation concerning important study variables to be considered and reported in bond strength studies. We considered this recommendation in a macro-shear strength study [6]. By defining, describing and standardizing the study variables according to this recommendation, we were able to reduce pre-test failures to a minimum. In another guidance paper derived from the 2009 ADM conference it was proposed that cohesive failures in dentin should be excluded from the evaluation because "*these data are not representative of an interface bond strength, but rather reflect a mixture of mechanical properties of the different materials involved*." [4]. With reference to this recommendation, we have taken a closer look at the item "data reporting and analysis" and examined the effect of excluding test specimens with specific failure modes on the results.

Particular care was taken to document study variables, i.e. tooth substrate, restorative material, specimen preparation and pre-testing conditions, testing methods, data reporting and analyses, according to the recommendations by Roeder et al. [2].

The aim of this study was to investigate the following research hypotheses:

(1) Exclusion of shear bond strengths associated with cohesive failures from the statistical analysis significantly influences the outcome of bond strength data.
(2) New formulations of one step universal bonding agents show a similar bonding performance on enamel and dentin.

2. Materials and methods

2.1. *Number of tooth substrates*

Each bovine tooth was used only once, to determine the bond strengths to enamel or to dentin. At least 84 specimens were investigated for each material and dental hard tissue.

2.2. *Bonding substances and composite*

The following dentin adhesive systems were investigated: Scotchbond Universal® (SBU), OptiBond™ XTR (OBXTR), OptiBond™ universal (OBU) and Prime & Bond active® (PBA). For all experiments ceram.x® universal was used as composite material. All materials are listed in Table 1. Details of specimen preparation and analysis of data are listed in Table S1.

2.3. *Application, air drying and light curing of materials*

Enamel or dentin was prepared with abrasive paper (see Table S1). Enamel and dentin surfaces and adhesives were air dried with a multifunctional syringe applying a moderate flow rate (2 m/s) as described earlier [6]. OBXTR primer was applied for 20 s, whereas adhesives were applied for 15 s or 20 s depending on the manufacturer`s instructions with disposable micro-applicators using a light scrubbing motion (Microbrush, Grafton, USA). Light curing was performed with three Bluephase 20i curing lights (Ivoclar Vivadent, Schaan, Liechtenstein, irradiance 1200 mW/cm^2 [high power mode]). All adhesives were cured for 10 s, composites 20 s from three sides each. Irradiances of all curing lights were verified using a MARC-RC™ device (Blue Light Analytics, Halifax, NS, Canada).

2.4. *Measurement technique*

After completion of bonding, positioning and curing of composite cylinders specimens were stored in an incubator (Heraeus incubator B6200, Thermo Fisher Scientific Inc., Waltham, Massachusetts) at 37 °C and kept under 100 % relative humidity for 24 h in order to create testing conditions similar to an oral environment. Further on three specimens at a time were fixed vertically in plaster in a plastic tray. Care was taken to ensure that the composite cylinders were aligned parallel to the surface of the plaster. After embedding of teeth, hard plaster was allowed to set for 1 h before testing. Subsequently, each specimen was manually positioned precisely below the bevelled chisel. In this way, the force could be applied as close as possible to the junction between composite and dental hard tissue. A Universal testing machine (1446602010/TND, Zwick/Roell, Ulm, Germany) was used for measurements. The applied pre-load was 5 N with a subsequent cross head speed of 0.8 mm/min. During testing, the fracture load was recorded and nominal stress at failure was calculated by dividing the fracture load by the bonding area.

2.5. *Fracture analysis*

As a basis for the fracture analysis of failure modes in adhesive resin bonding to dentin, the classification according to Scherrer et al. was used, simplified and additionally applied to enamel [4]. The failure modes were visually inspected without magnification and divided into cohesive or adhesive failures only, defined as follows: All failures were grouped together as adhesive failures which were designated by Scherrer et al. as "adhesive concerning the interface dentin-adhesive" or "adhesive concerning the interface resin-adhesive" [4]. All failures were grouped together as cohesive failures which were "cohesive in dentin" or "mixed cohesive in dentin - adhesive". No failures of the category "cohesive in resin" were observed.

Table 1 – Materials investigated: primer, adhesive materials, and composite.

Code	Material	Manufacturer	Lot Numbers	Formulation
SBU	Scotchbond Universal® One-Component Universal Self-Etching Dental Adhesive	3 M ESPE, Neuss, Germany	594754 71130	MDP phosphate monomer, demethacrylate resins, HEMA, Vitrebond™ copolymer, filler, ethanol, water, initiators, silane
OBXTR	OptiBond™ XTR Two-component, Self-etch Universal Adhesive	Kerr, Orange, CA, USA	Primer: 540462 Adhesive: 542703	**Primer:** Acetone, ethyl alcohol, hydroxy ethyl methacrylate (HEMA) **Adhesive**: Ethyl alcohol, alkyl dimethacrylate resins, Barium aluminoborosilicate glass, fumed silica (silicon dioxide), Sodium hexafluorosilicate
OBU	OptiBond™ universal One-Component Self-Etching Dental Adhesive	Kerr, Orange, CA, USA	701659	Acetone, 2-hydroxyethyl methacrylate, ethanol, glyceryl dimethacrylate
PBA	Prime & Bond active® One-Component Self-Etching Dental Adhesive	Dentsply Sirona, Bensheim, Germany	181000013	Phosphoric acid modified acrylate resin, multifunctional acrylate, bifunctional acrylate, acidic acrylate, isopropanol, water, initiator, stabilizer
	Ceram X Universal Universal Nano-Ceramic Restorative	Dentsply Sirona, Bensheim, Germany	1508000827 150800061 150300091 150800028	Methacrylate modified polysiloxane (organically modified ceramic), dimethacrylate resins, fluorescent pigment, UV stabilizer, stabilizer, camphorquinone, ethyl-4(dimethylamino) benzoate, Bbis(4-methyl-phenyl)iodonium hexafluorophosphate, barium-aluminum-borosilicate glass, ytterbium fluoride, iron oxide pigments and titanium oxide pigments according to shade

2.6. *Statistical methods*

We provide descriptive statistics (mean and standard deviation) of shear bond strenghts per material and enamel/ dentin, both including cohesive failures and excluding them. Corresponding boxplots are shown, as well as a bar plot for the percentage of failures.

For inference, we used a linear mixed model [7] to predict shear bond strength based on fixed effects technique, material, dental hard tissue and a random effect for the individual user. Diagnostics were checked visually. For tests based on this model we used t-Tests and the Kenward-Roger approximation for degrees of freedom [8].

All computations were done using R version 4.2.0 [9].

3. Results

Dentin and enamel bond strengths were comparable for OBU and OBXTR while lower values were measured on enamel for SBU ($p < 0.001$) and PBA ($p = 0.015$). The rank orders of shear strengths for dentin/enamel pooled, enamel only and dentin only are depicted in Table 2.

All materials showed more cohesive failures in dentin than in enamel (Fig. 1). Most cohesive failures in dentin were observed for SBU and OBXTR followed by OBU and PBA.

Fig. 2 shows shear strengths of specimens with adhesive failures only (cohesive failures excluded) or all data (cohesive failures included). Medians of specimens with adhesive failures only are only slightly different from overall results with the exception of OBXTR with a dramatically lower median for dentin when cohesive failures are excluded.

4. Discussion

SBU and PBA were found to have lower bond strengths in enamel than in dentin, while no difference was observed for OBXTR and OBU. Accordingly, the research- hypothesis [2] was accepted.

There is no clear consensus in the literature regarding the interpretation of failure modes in terms of their classification. Nor are failure modes routinely determined and defined with the same methods e.g. by optical or electron microscopy [4,10]. The results of a meta-analysis showed a strong correlation between the failure mode and the mean bond strength and in consequence that high bond strengths are often associated with cohesive failures [1]. This led some authors to conclude that cohesive failures either in dentin or resin should be completely excluded from the evaluation, as they are more related to the brittleness of tooth structure or resin-composite and to the test geometry rather than to the actual bond (strength) between resin and dental hard tissues [4,11,12]. However, there might be a risk that products showing almost only cohesive failures, even with a very high sample size, will fail to give a fair impression compared to other products, since fewer values could be included in the evaluation in comparison to another product tested in parallel with predominantly adhesive failures. Therefore, in the present work the data were evaluated both with and without cohesive failures of specimens in dentin and enamel.

Table 2 – Enamel-dentin comparisons and rank orders of shear strengths for dentin/enamel pooled, enamel only and dentin only.

Dentin/enamel pooled			Enamel only			Dentin only		
Material	Mean	SD	Material	Mean	SD	Material	Mean	SD
PBA	11.6	5.9	PBA[A]	9.1	3.4	PBA[A]	11.6	5.9
OBU	17.2	5.6	SBU[B]	15.2	4.3	OBU[C]	17.2	5.6
SBU	18.4	5.1	OBU[C]	18.9	4.4	SBU[B]	18.4	5.1
OBXTR	21.1	8.9	OBXTR[D]	20.1	3.9	OBXTR[D]	21.1	8.9

A: $p = 0.015$
B: $p < 0.001$
C: $p = 1.000$
D: $p = 1.000$

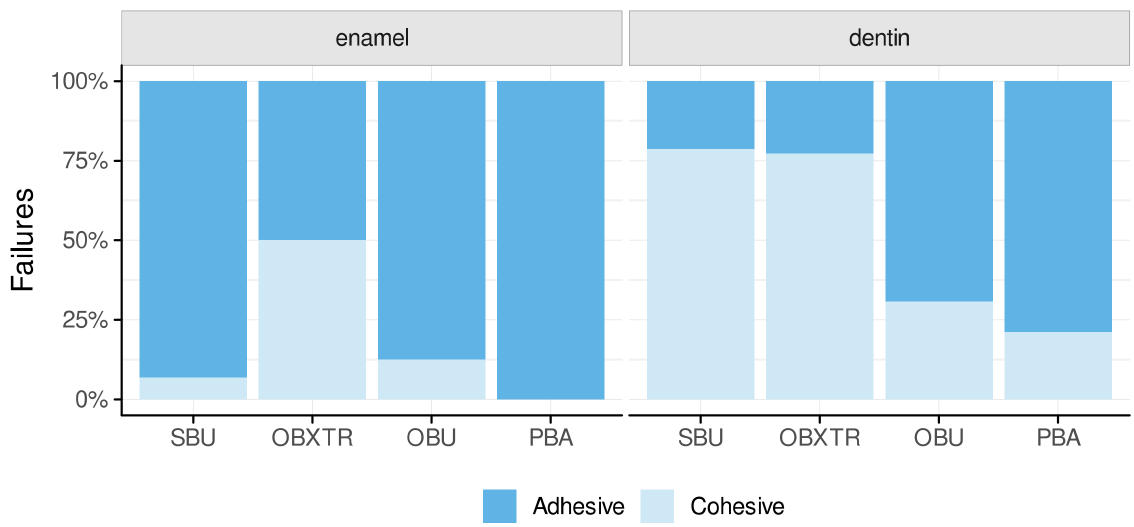

Fig. 1 – Percentage of adhesive and cohesive failures (enamel or dentin) for each adhesive material.

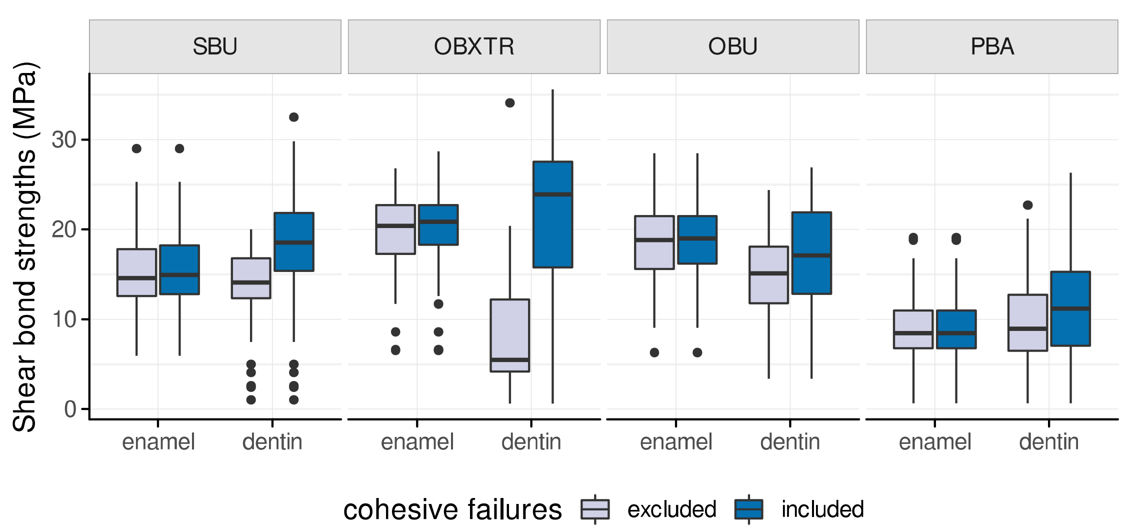

Fig. 2 – Shear bond strengths (MPa) for each adhesive material on enamel or dentin, with adhesive failures only (cohesive failures excluded) or all data (cohesive failures included).

Evaluation with excluded cohesive failures gave misleading results for some materials, such as OBXTR on dentin. Since, in this case, high shear bond strengths were measured and associated mostly with cohesive failures, the median dropped dramatically after exclusion of the cohesive failures. Hence, research-hypothesis [1] was accepted. Thus the adhesive fell behind other adhesives in the ranking of average shear bond strengths. This fails to correspond to the adhesive properties of this product, because higher shear strengths were excluded from the evaluation. Therefore, we do not recommend exclusion of data based on cohesive fracture behavior.

It has been recommended that the characterization and decision on the type of failure mode could be carried out exactly only via electron microscopy [4,13]. It is difficult to comply with this recommendation for experiments with a very high sample size. It is a limitation of our study that due to the very large total sample size (at least 84 samples for each material and dental hard tissue) we have restricted ourselves to the classification into cohesive and adhesive failures only.

5. Conclusion

Omitting shear bond strengths associated with cohesive failures can lead to biased conclusions, because for strong adhesives this is a frequent failure mode which is almost always related to high shear bond strengths. As a consequence only fewer results with lower adhesive strength associated with adhesive failures would remain and are taken into consideration for the evaluation.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.dental.2022.10.003.

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