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Does tin pre-treatment enhance the bond strength of adhesive systems to enamel?

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ABSTRACT

Objectives: The study investigated the modification of composite-to-enamel bond strength by pre-treatment of enamel with a concentrated, acidic $SnCl_2$-solution.

Methods: Six groups of flat human enamel specimens ($n = 44$ per group) were treated as follows: OB-H: H_3PO_4 etching, Optibond FL application (primer + adhesive; manufacturer's instructions); OB-S: $SnCl_2$ pre-treatment, Optibond FL application (primer + adhesive); OB-HS: H_3PO_4 etching + $SnCl_2$ pre-treatment, Optibond FL application (primer + adhesive); CF-N: Clearfil SE application (primer + bond; manufacturer's instructions); CF-H: H_3PO_4 etching, Clearfil SE application (primer + bond); CF-S: $SnCl_2$ pre-treatment, Clearfil SE application (primer + bond). Enamel specimens were then built up with resin composite (Clearfil Majesty Esthetic) and stored (100% humidity, 37 °C, 1 week). μTBS-measurement and failure mode analysis of one-half of the specimens were performed immediately after storage, while the other half was analysed after a thermocycling procedure (8500 cycles; 5 °C and 55 °C; dwell time 30 s). Additional specimens were prepared for SEM- and EDX-analysis.

Results: Highest values were measured for OB-H before and after thermocycling, lowest values for CF-N. Compared to OB-H treatment, OB-S treatment reduced μTBS before/after thermocycling by 23%/28% and OB-HS treatment by 8%/24% (except for OB-SH before (n.s.), all $p \leq 0.001$ compared to OB-H). In the Clearfil SE treated groups pre-treatment increased μTBS significantly compared to CF-N (before/after: CF-H: +46%/+70%; CF-S: +51%/42%; all $p \leq 0.001$).

Conclusion: Pre-treatment with H_3PO_4 or $SnCl_2$ markedly increased the μTBS of Clearfil SE to enamel. However, thermocycling partly reduced the gain in μTBS obtained by $SnCl_2$ pre-treatment.

Clinical significance: The application of an acidic and highly concentrated $SnCl_2$ solution is a good option to increase the μTBS between enamel and a resin composite mediated by an adhesive system containing the multifunctional monomer MDP.

1. Introduction

Stannous ions are widely used in dentistry and numerous studies have investigated the effect of these ions on enamel solubility, showing that tin is a very effective anti-cariogenic agent.[1,2] Tin has also notable anti-erosive properties, acting by the formation of acid resistant precipitates on sound, but also on eroded enamel surfaces.[3,4] After erosive acid impacts, tin can also be incorporated in the upper layer of the eroded

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dental hard tissue,[3] indicating that this ion can intensively interact with the dental hard tissue in particular at low pH. An issue still under debate in this context is, whether the use of oral hygiene products containing stannous and/or fluoride ions prior to the application of an adhesive and a restorative material has a negative impact on resin-to-enamel bond strength. Various studies have investigated the impact of the application of fluoride solutions after acid etching on bond strength and found a negative effect for e.g. sodium or basic phosphate fluoride solutions.[5] However, only few studies exist investigating the effect of tin pre-treatments on this parameter. One study showed that the application of an 8% SnF_2-solution had no negative impact on bond strength of a fissure sealant.[5] However, the study included no control group. Two other studies found that bond strength was increased by a 4-minute application of an 8% stannous fluoride solution in comparison to a 2% sodium fluoride[6] or acidulated phosphate fluoride solution.[7] A recent study has also shown that the pre-treatment of eroded dentine twice a day for 2 min each with a stannous (800 ppm Sn^{2+}) and fluoride ion (500 ppm F^-) containing mouth rinse led to higher microtensile bond strength (μTBS) of the adhesive system Clearfil SE than did no pre-treatment or pre-treatment with a sodium fluoride solution (500 ppm F^-).[8] These studies indicate that tin could be a promising agent to increase the bond strength between the dental hard tissue and resin composite.

In case of the total-etch technique, the most commonly used method for conditioning teeth prior to direct restorative treatment is the use of phosphoric acid with a concentration of approximately 35% and a pH below 1. Highly concentrated stannous chloride solutions have a comparably low pH, holding a high demineralising potential. It has been shown for dentine that the application of and the etching with such a highly concentrated stannous chloride solution prior the application of two different adhesive systems (Optibond FL and Clearfil SE) can lead to notable demineralisation and can modify the μTBS of both adhesive systems.[9] However, it is not clear whether the acidic tin chloride solution can also sufficiently demineralise enamel and can produce an etching pattern comparable to that created by phosphoric acid, and whether the retention of tin on and in the dental hard tissue has an impact on the bond strength to enamel.

The aim of the present study was, therefore, to investigate whether the application of a highly concentrated $SnCl_2$-solution (35%, pH < 1) changes the μTBS of two different adhesive systems (total etch adhesive system, Optibond FL and self-etching adhesive system, Clearfil SE) to enamel in comparison to the standard procedure recommended by the manufacturers. The μTBS was measured before and after ageing by thermocycling. The null hypothesis was that there would be no effect of the $SnCl_2$-solution. Additionally, the enamel surfaces of all groups were qualitatively assessed by scanning electron microscopy (SEM) and by energy dispersive X-ray spectroscopy (EDX).

2. Material and methods

2.1. *Preparation of specimens*

Human, caries-free molars without cracks as inspected under a stereomicroscope were used. Teeth were cleaned with scalpel and scaler to remove debris and soft tissue and stored in Chloramin (1%; 4 °C). Afterwards, each tooth was cleaned with pumice and a prophylaxis cup at low speed for 10 s. Molars were sectioned in mesio-distal direction into halves in order to obtain two specimens per tooth. The buccal and lingual parts, respectively, were randomly divided into 6 groups (*n* = 44 per group). The teeth were individually embedded in resin material (Paladur; Heraeus Kulzer GmbH, Hanau, Germany) for better handling. The smooth surfaces were ground flat with grit #320 and grit #500 SiC abrasive paper (Struers LaboPol-21/SiC #320, Struers, Ballerup, Denmark) under water flow until an experimental area of approximately 4 mm × 4 mm was produced with no dentine exposed. Specimens with exposed dentine within the enamel area were discarded. The border between the specimens and the embedding material was marked with a carbide drill and a contra-angle hand piece. A total of 264 specimens were prepared. For SEM and EDX analysis, 18 additional specimens (*n* = 3 per group) were produced.

2.2. *Adhesive treatments*

Six different adhesive treatments were tested (Table 1). Etching was performed for 15 s with either a H_3PO_4 solution (35%, ortho-phosphoric acid 85%; 79623; Sigma Aldrich; Seelze, Germany) or a $SnCl_2$ solution (35%, provided by GABA International AG, Therwil, Switzerland). The primer of Optibond FL (Kerr, Bioggio, Switzerland) or Clearfil SE (Kuraray, Okayama, Japan; for details see Table 2) was applied for 15 s

Table 1 – Treatment of enamel specimens prior resin composite built up.

Group	Etching + rinsing	Primer
H_3PO_4 – Optibond FL (**OB-H**)	15 s H_3PO_4; 15 s rinsing with tap water	15 s application of Optibond FL primer; gentle blow drying; application of Optibond FL adhesive
$SnCl_2$ – Optibond FL (**OB-S**)	15 s $SnCl_2$; 15 s rinsing with tap water	
H_3PO_4/$SnCl_2$ – Optibond FL (**OB-HS**)	7 s H_3PO_4; 7 s rinsing with tap water; 7 s $SnCl_2$; 7 s rinsing with tap water	
Clearfil SE (**CF-N**)	—	15 s application of Clearfil SE primer; gentle blow drying; application of Clearfil SE bond
H_3PO_4 – Clearfil SE (**CF-H**)	15 s; H_3PO_4; 15 s rinsing with tap water	
$SnCl_2$ – Clearfil SE (**CF-S**)	15 s $SnCl_2$; 15 s rinsing with tap water	