

## Research article

# Micro-tensile bond strength of two pit and fissure sealants to intact enamel

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## ABSTRACT

**Background:** Sealing dental pits and fissures with resin-based sealants effectively prevents occlusal dental caries. The effectiveness of resin-based pit and fissure sealants relies on maintaining a strong bond between the sealant and the enamel.

**Objective:** This *in-vitro* study compared the micro-tensile bond strength ( $\mu$ TBS) of a conventional resin-based sealant (Clinpro™) and a hydrophilic resin-based sealant (Embrace™ WetBond™) when applied to intact, aprismatic human enamel.

**Methods:** Forty extracted permanent premolar and molar teeth were divided into two groups and paired by tightly approximating two buccal surfaces to create an artificial enamel groove (fissure). Fissure sealants (Clinpro™ and Embrace™ WetBond™) were applied to the artificial enamel 'grooves' in each group. The specimens were then cut into beams with a cross-sectional area of 1 mm<sup>2</sup> and tested for the micro-tensile bond strength ( $\mu$ TBS). Fractured surfaces of samples were examined under a conventional microscope to identify the failure modes. Two specimens from each group were prepared and observed under a scanning electron microscope (SEM). Mann-Whitney U and Fischer-Freeman-Holton exact tests were used to test the statistical differences between the fissure sealants.

**Results:** The  $\mu$ TBS mean  $\pm$  SD for Clinpro™ was 16.43  $\pm$  7.08, and 10.57  $\pm$  6.64 for Embrace™ WetBond™. There was a statistically significant difference in  $\mu$ TBS between Embrace™ WetBond™ and Clinpro™ ( $p < 0.001$ ). There was no association between fissure sealant and failure modes ( $p = 0.922$ ).

**Conclusion:** Clinpro™ showed higher  $\mu$ TBS to enamel than Embrace™ WetBond™. Further studies are needed to conclude the clinical effectiveness of these sealants.

## 1. Introduction

The morphological complexity of teeth, such as occlusal pits and fissures, makes them highly susceptible to caries [1].

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**Table 1**  
List of materials.

Material	Manufacturer	Composition	Enamel conditioning
Clinpro™	3 M ESPE, St. Paul, MN, USA	<ul style="list-style-type: none"> <li>• Bisphenol A Diglycidyl Ether Dimethacrylate (BISGMA)</li> <li>• 2,2'-ethylenedioxydiethyl dimethacrylate</li> <li>• 2-Propenoic acid, 2-methyl-, 3- (trimetoxysilyl)propyl ester, hydrolysis products with silica</li> <li>• Tetrabutylammonium tetrafluoroborate</li> <li>• Diphenyl Hexafluorophosphate</li> <li>• Ethyl 4-Dimethylaminobenzoate (EDMAB)</li> <li>• Titanium dioxide</li> <li>• Triphenylantimony</li> <li>• Hydroquinone</li> </ul>	Absolutely dry
Embrace™ WetBond™	Pulpdent Corporation Watertown, MA, USA	<ul style="list-style-type: none"> <li>• Acrylate ester monomers in two-part, glass-filled, dual-cure paste</li> <li>• Uncured acrylate ester monomers</li> <li>• Silica, amorphous</li> <li>• Sodium fluoride</li> <li>• phosphoric acid (37%),</li> <li>• bio-compatible polymeric materials</li> </ul>	Relatively dry
Spident Fine Etch	Spident Co., Ltd, South Korea	<ul style="list-style-type: none"> <li>• phosphoric acid (37%),</li> <li>• bio-compatible polymeric materials</li> </ul>	

Epidemiological data show that pit-and-fissure surfaces carry a significant amount of the total caries burden in schoolchildren [2]. Preventive measures such as fluoride toothpaste and topical fluoride application have limited effectiveness against occlusal caries; therefore, physical preventive approaches such as pit and fissure sealants are currently considered the most effective [3].

Sealing pits and fissures of primary and permanent teeth can reduce the incidence of dental caries [4]. Applying pit and fissure sealants improves mechanical cleansing of teeth and reduces the surface area of the tooth exposed directly to demineralisation [5].

Resin-based pit and fissure sealants are the most used material for sealing pits and fissures [6–13]. Evidence suggests that placing resin sealants keeps the sealed occlusal surfaces caries-free and prevents the development of cavitated carious lesions in high-caries-risk individuals [14].

The effectiveness of sealants in preventing caries relies on their clinical retention over time [6,15–17], which in turn requires stable and strong adhesion (bonding) between a sealant and the enamel [8]. A dry tooth surface is a necessary condition for the application of a resin-based sealant to ensure effective adhesion of the sealant to the enamel [8,13,18–20].

Traditional pit and fissure sealants, such as Clinpro (Clinpro™), require a completely dehydrated enamel surface for effective bonding, whereas newly developed moisture-tolerant sealants, such as Embrace (Embrace™ WetBond™), require a lightly dried but not completely desiccated enamel surface [15,21–23].

The newly developed fissure sealants thus offer an advantage over the traditional sealants since achieving and maintaining a completely desiccated enamel surface is more time-consuming and challenging than achieving a relatively dry surface [19,21–23]. This difference becomes particularly significant when treating paediatric patients, children of special needs and other potentially uncooperative patients [24].

In-vivo studies have evaluated the retention rates of different fissure sealants. These studies found no statistically significant difference between the retention rates of the conventional sealant Clinpro and the moisture-tolerant sealant Embrace [15,25,26]. However, limited studies have been conducted to test the effectiveness of different sealant types based on their retention and comparative bonding strengths [27–29].

The micro-tensile bond strength ( $\mu$ TBS) test is the best *in-vitro* measure of dental resin-based adhesive restorative retention [27,30]. The  $\mu$ TBS test is a reliable *in-vitro* testing method for evaluating experimental variables in adhesive bonding materials to the tooth structure. The  $\mu$ TBS test method is relatively easy to perform, could be repeatable within and between laboratories, and is ultimately helpful in predicting clinical outcomes [28]. Ideally, adhesion to tooth structure should provide retentive strength and marginal seal, be relatively simple to achieve and have clinical durability. The  $\mu$ TBS test, especially after subjecting the specimens to a durability challenge, is currently recommended as the best surrogate measure of dental composite restoration retention [29].

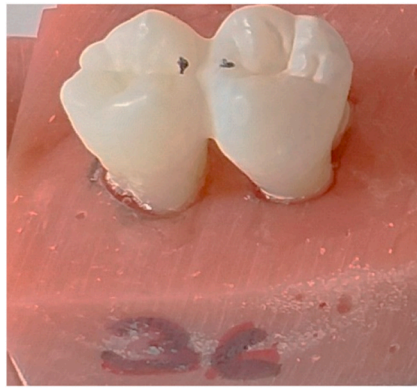
Although the bond strengths of some fissure sealants have been compared [31], to date, no study has compared the micro-tensile bond strengths to the enamel of conventional (Clinpro) and moisture-tolerant (Embrace) sealants.

Therefore, this *in-vitro* study aimed to measure and compare bond strengths of conventional (Clinpro) and hydrophilic (Embrace) pit and fissure sealants. Both Clinpro and Embrace are currently available on the market. Clinpro, like all conventional sealants, requires a perfectly dry enamel surface. On the other hand, Embrace, a hydrophilic sealant, requires a moist enamel surface for bonding.

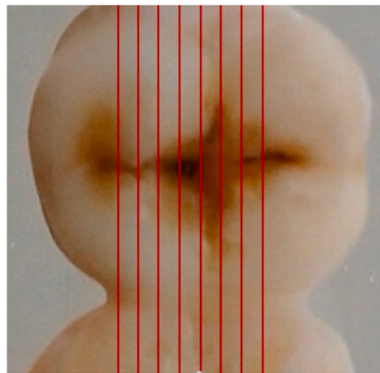
The study hypothesis proposed no difference between the micro-tensile bonding strength ( $\mu$ TBS) of Clinpro and Embrace bonded to intact enamel when tested soon after the bonding.

## 2. Materials and methods

Forty sound permanent premolar and molar teeth extracted for orthodontic reasons from patients aged 20 to 40 were obtained from dental clinics across Auckland. The teeth were cleaned and separately stored in distilled water at 4 °C with the details of the extraction date to be used within one month following extraction. Teeth were then removed and polished with pumice to remove any surface



**Fig. 1.** Sealed teeth embedded in a resin block.



**Fig. 2.** Tooth sectioning (parallel lines).

debris or contaminants. Then, they were stored in 0.1% thymol solution at room temperature before the experiment.

The teeth were not subjected to thermocycling or ageing because this study intended to compare the bond strengths immediately after the bonding, as the technique of using the two sealant materials was different.

This study adopted and modified the simplified enamel fissure model for a more realistic representation of a fissure [12] because occlusal morphology is an essential factor in the depth of penetration of the sealant into the enamel [32].

### 2.1. Teeth preparation

The roots of all teeth were trimmed below the cementoenamel junction (CEJ), leaving about one-third of the roots. The crowns were randomly assigned to one of the two groups ('Clinpro' or 'Embrace'), differing in the procedure employed for enamel preparation and the type of sealant used (Table 1). Each tooth was secured with sticky wax on a stable surface. The selected surfaces of two teeth were tightly approximated to create an artificial occlusal 'groove' using a spacer to a mean distance of  $0.6 \pm 0.1$  mm (Fig. 1). Next, all teeth were conditioned with 37% phosphoric acid and rinsed with distilled water. The teeth were then air-dried according to the manufacturer's instructions for the type of sealant used. Fissure sealants were applied to the groove and light-cured (with SDI Radium Plus LED curing light) for 20 s. After applying the sealant, each tooth was removed from sticky wax and embedded in acrylic resin (Unifast Trad II, GC, Tokyo, Japan), making a block of roughly 1.5 cm x 1.5 cm in dimension. The roots of the teeth were embedded in the resin block, with the crowns bonded to sealant visible from the CEJ (Fig. 1).

### 2.2. Specimen preparation

The teeth were stored in distilled water at room temperature for less than 24 h before sectioning. Then, they were sectioned with the slow-speed diamond saw featuring a diamond-coated blade (Isomet, Buehler, Lake Bluff, IL, USA). Each tooth was sectioned perpendicular to the bonded surface under copious water cooling to obtain 1.00 mm thick slices (Fig. 2). Each slice was further sectioned into beams with a sealant interfacing surface area of 1.00 mm<sup>2</sup> (Fig. 3).

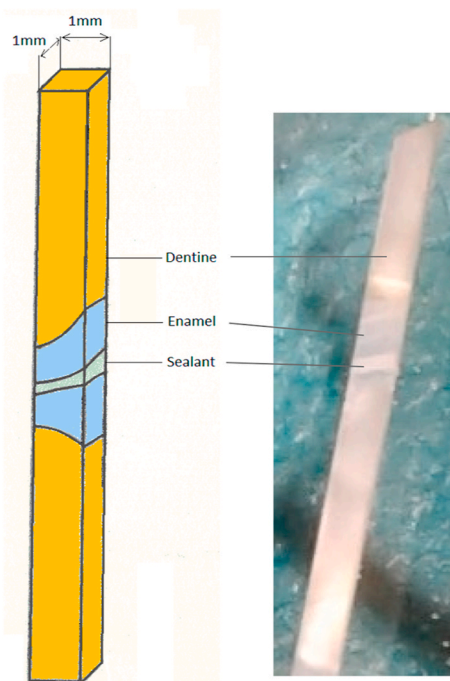


Fig. 3. A specimen with a sealant surface area of 1.00 mm<sup>2</sup>.

### 2.3. Testing the micro tensile bond strength

A single operator performed individual testing of each specimen. Before the testing, all specimens were examined with an optical microscope (Leica ICC50 HD) (magnification  $\times 30$ ) for defects at the bonding interface. Specimens showing defects such as apparent interfacial spaces, formations of air bubbles, or any other imperfections were removed and replaced with sound specimens. The specimens were fixed to opposing arms of the testing device (TA.XT.plus Texture Analyser by Stable Micro Systems) using Cyanoacrylate adhesive (Selleys Quickfix Liquid Supa Glue). Each specimen was fractured under tension at a crosshead speed of 1 mm/min. The maximum load (peak force) at the moment of fracture was measured in Newtons and recorded.

### 2.4. Failure modes

After  $\mu$ TBS testing, the fractured samples were examined under a conventional microscope with a camera attachment (Leica ICC50 HD) at an original magnification of  $\times 40$  to explore and categorise the failure modes. The failure modes were sorted into one of the following four types: (1) adhesive failure between the fissure sealant and the enamel, (2) cohesive failure in the enamel, (3) cohesive failure in the fissure sealant, (4) mixed failure.

### 2.5. SEM observation

Two specimens from each group were selected for SEM observation. Four bonded enamel/sealant specimens were cross-sectioned at the interfaces after storing for 24 h at 37 °C to observe the interfaces between enamel and the adhesive resin. The specimens were polished with polishing sheets in a descending sequence up to 3- $\mu$ m roughness. They were then etched with 0.1 N HCl for 30 s, washed with distilled water, and dried in an incubator for 24 h. Finally, the surfaces were sputter-coated with platinum using a Hitachi E-1045 sputter coater and observed under a scanning electron microscope (Hitachi SU-70).

### 2.6. Statistical analysis

Ultimate stresses in MPa were calculated as a ratio of the peak force (N) to the surface area of 1.00 mm<sup>2</sup>. The mean  $\pm$  SD for  $\mu$ TBS (in MPa) was calculated. A Mann-Whitney *U* test was run to determine if differences existed between the  $\mu$ TBS measured for each sealant. Frequencies and percentages were determined for modes of failure. A Fisher-Freeman-Halton exact test was conducted between sealant type and failure modes. A *p*-value  $< 0.001$  was considered statistically significant. Statistical analyses were performed with the SPSS software (IBM Corp. Released 2020. IBM SPSS Statistics for Windows, Version 27.0. Armonk, NY: IBM Corp).

**Table 2**  
 $\mu$ TBS to enamel (mean  $\pm$  SD) in MPa.

Specimens for $\mu$ TBS test	$\mu$ TBS (mean $\pm$ SD)	p-value
Clinpro™ specimens N = 53	16.43 $\pm$ 7.08	0.001
Embrace™ WetBond™ specimens N = 59	10.57 $\pm$ 6.64	

Note. \*p-value calculated using Mann-Whitney U test for continuous variables.

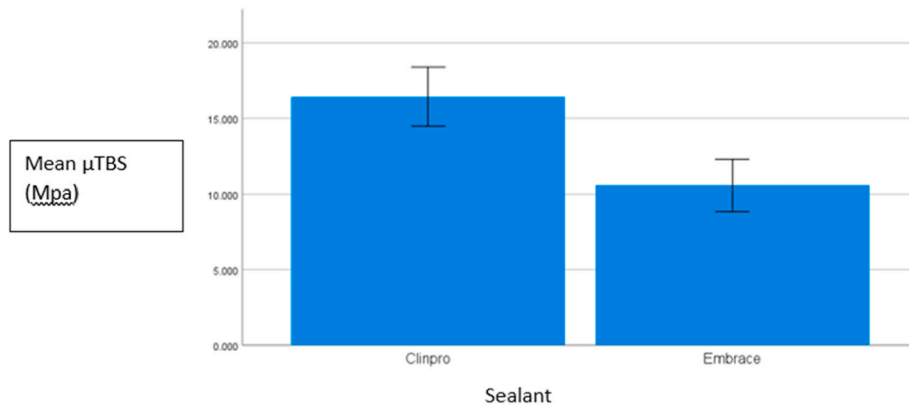


Fig. 4.  $\mu$ TBS (MPa) for Clinpro™ and Embrace™ WetBond™.

**Table 3**  
 Modes of failure following  $\mu$ TBS.

Specimens identifying modes of failure	Adhesive n (%)	Cohesive in the enamel n (%)	Cohesive in the sealant n (%)	Mixed n (%)	p-value
Clinpro™ specimens N = 59	10 (16.9)	2 (3.4)	32 (54.2)	15 (25.4)	0.922
Embrace™ WetBond™ specimens N = 59	12 (20.3)	2 (3.4)	28 (47.5)	17 (28.8)	

Note. n = failure modes frequencies; % = n/NX100.

\*p-value calculated using Fisher-Freeman-Halton exact test for categorical variables.

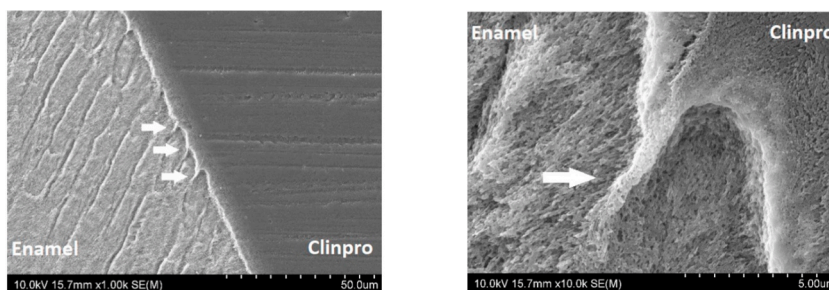
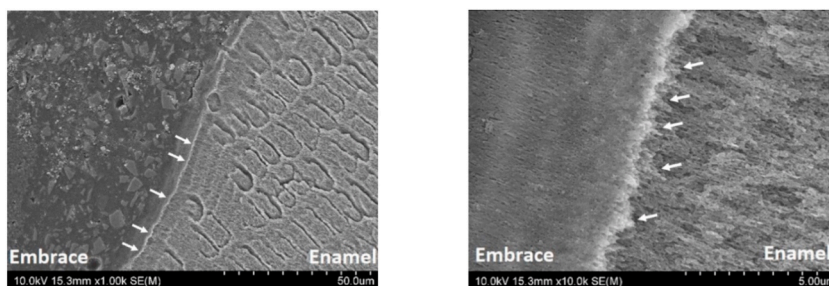


Fig. 5. 5.1 and 5.2: SEM images of the adhesive interface of Clinpro™ bonded to enamel showing resin tag-like penetrations of about 10  $\mu$ m into the enamel (arrows).

### 3. Results

#### 3.1. Micro tensile bond strengths and failure modes

The  $\mu$ TBS (in MPa) mean  $\pm$  SD for Clinpro and Embrace are shown in Table 2 and Fig. 4. The mean  $\mu$ TBS and  $\pm$ SD of Clinpro was 16.43  $\pm$  7.08 and 10.57  $\pm$  6.64 for Embrace. There was a statistically significant difference in  $\mu$ TBS between Embrace and Clinpro at  $p < 0.001$ .



**Fig. 6.** 6.1 and 6.2: SEM images of the adhesive interface of Embrace™ WetBond™ bonded to enamel showing irregular resin tag-like penetrations of about 5  $\mu\text{m}$  into the enamel (arrows).

Of 224 samples, 118 were analysed to detect the failure modes. The frequencies and percentages of failure modes following the  $\mu\text{TBS}$  test are summarised in Table 3. The most common failure mode was cohesive in the sealant (54.2% for Clinpro and 47.5% for Embrace). There was no association between fissure sealant and failure modes as assessed by the Fisher-Freeman-Halton exact test,  $p = 0.922$ .

### 3.2. SEM observations

SEM photomicrographs of the adhesive interface between enamel and the sealants are shown in Figs. 5.1 and 5.2 (Clinpro), and in Figs. 6.1 and 6.2 (Embrace). There is no discontinuity in the enamel-resin interface in either sealant system, although the Embrace–resin bonding interface exhibited an interrupted interface in some areas. Images of 10k magnification of the interface showed about 10  $\mu\text{m}$  resin tags in the hybrid layer with Clinpro and about 5  $\mu\text{m}$  with Embrace. Pictures of the bonding interface of Embrace showed an irregular pattern of resin tags at the interface, unlike demarcated tags in Clinpro.

## 4. Discussion

Most of the studies on  $\mu\text{TBS}$  examined the effect of ageing on the retention or bond strength, but this study compared the bond strength ( $\mu\text{TBS}$ ) of newly placed sealants that were not subjected to ageing. One study concluded that the results of the micro tensile or shear bond strength of different fissure sealants bonded to enamel were not influenced by the thermal cycling of the specimens [33].

The present *in-vitro* study showed a significant difference in the  $\mu\text{TBS}$  between the two commercially available resin-based sealants – Clinpro, a conventional (Bis-GMA-based) sealer and Embrace, a hydrophilic (acrylate monomer-based) sealant ( $p < 0.001$ ). Weaker values of  $\mu\text{TBS}$  were observed for Embrace, using a wet bonding technique, compared to Clinpro, using a conventional bonding technique, when measured soon after bonding to enamel.

Similar studies have been conducted on different sealant products, comparing their  $\mu\text{TBS}$  [31]. An *in-vivo* study [15] found that the conventional resin-based and hydrophilic resin-based sealants showed similar retention rates after a follow-up period of 6 months, and after 12 months, the rate of completely retained sealants was 72.7% for the Embrace; in contrast to the studies by Bhatia [25] and Subramanian [26], which showed the 12-months complete retention rates for Embrace sealants as 23% and 10%, respectively. However, none of the three studies showed a statistically significant difference between the retention rates of the conventional sealant Clinpro and the moisture-tolerant sealant Embrace.

The success of sealants is technique-sensitive, as most products require maintaining a dry field. A study showed that Embrace had demonstrated reliability over time after being wet bonded [15]. Another study that compared the marginal adaptation under SEM of the sealants Clinpro and Embrace found that the marginal adaptation of Embrace was statistically superior to Clinpro [24]. This finding is in sharp contrast to the present study, where it was found that under high magnification (10K), SEM samples bonded with Embrace sealant had multiple areas where the sealant was stripped away from the enamel surface. The tag-like penetrations into the enamel were also irregular and inconsistent in samples bonded with Embrace under the SEM.

Al-Quahtani et al., 2022 compared the  $\mu\text{TBS}$  of bioactive resin-based sealants (Bio-RBS) and resin-based sealants (RBS) applied to primary and permanent teeth with and without bonding agents. They concluded that when applied to permanent teeth, Bio-RBS exhibited superior  $\mu\text{TBS}$  compared to RBS [34].

The null hypothesis was rejected in the present study. Rejecting the null hypothesis may mean that the wet bonding technique using Embrace sealant does not provide the anticipated bonding strength soon after the bonding procedure is completed.

In conclusion, this study provided baseline information for the  $\mu\text{TBS}$  of Clinpro, a traditional sealant requiring a completely dry tooth surface and Embrace, a hydrophilic sealant requiring a moist tooth surface for bonding. Moreover, it studied the bonding surfaces under SEM for two sealants. It is appropriate to say that there is a need for further studies to compare the Embrace, a hydrophilic sealant, to other available pit and fissure sealant materials, not only in the  $\mu\text{TBS}$  but also in the micro-shear bond strength, micro-leakage, and longevity. The bonding of the sealant application techniques on different uncontaminated, saliva-contaminated, and water-contaminated enamel conditions is another factor that could be investigated. Moreover, there is a need for clinical studies to evaluate the long-term success and retention of Clinpro and Embrace. Although inferior to Clinpro, the bonding strength achieved with

Descriptives					
Batch			Statistic	Std. Error	
Peak Force (Cycle: 1)	Clinpro	Mean	16.43054	.972705	
		95% Confidence Interval for Mean	Lower Bound	14.47866	
			Upper Bound	18.38241	
		5% Trimmed Mean	16.28382		
		Median	16.65016		
		Variance	50.146		
		Std. Deviation	7.081396		
		Minimum	3.039		
		Maximum	34.804		
		Range	31.765		
		Interquartile Range	8.938		
		Skewness	.214	.327	
		Kurtosis	.014	.644	
			Embrace	Mean	10.57118
95% Confidence Interval for Mean	Lower Bound			8.84112	
	Upper Bound			12.30124	
5% Trimmed Mean	10.37892				
Median	10.99881				
Variance	44.073				
Std. Deviation	6.638725				
Minimum	.592				
Maximum	24.700				
Range	24.109				
Interquartile Range	11.407				
Skewness	.138			.311	
Kurtosis	-.936			.613	

Fig. 7. Descriptive statistics.

Embrace may still be adequate for the long-term retention of the sealant when used in patients. Therefore, further clinical trials would be recommended to evaluate the long-term retention of the two types of sealants.

#### 4.1. Limitations of the study

As this was an *in-vitro* laboratory study, only the  $\mu$ TBS of the bonding of two sealants was studied soon after the sealant placement. An *in-vivo* (clinical) study will need to investigate the clinical application and retention of the sealants over time.

#### 4.2. Clinical relevance

Fissure sealants are routinely applied as a preventive measure globally. Clinpro and Embrace are two commercially available sealants used in oral health clinics. Research on the bond strengths would provide more information about the two sealants and their retention ability over time.

#### Ethics statement

- This study was reviewed and approved by the **Health and Disability Ethics Committee** and the **Auckland University of Technology Ethics Committee**, with approval numbers HDEC-17/STH/226 and AUTEK-18/250.

## Clinical significance

Fissure sealants are routinely applied as a preventive measure globally. Clinpro™ and Embrace™ WetBond™ are commercially available sealants used in oral health clinics. Research on the bond strengths would provide more information about the two sealants and their retention ability over time.

## Data availability statement

Data associated with the study has not been deposited into a publicly available repository. Data has been included in the appendix section of the article (Fig. 7).

## CRedit authorship contribution statement

**Erekle Sesiashvilli:** Writing – review & editing, Writing – original draft, Visualization, Resources, Investigation, Data curation. **Priyadarshane M. Ratnaweera:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Formal analysis, Conceptualization. **Claudia Zagreanu:** Writing – review & editing, Validation, Resources, Formal analysis.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Adrian Owens: Senior Technician. Department of Applied Science, AUT.

tored in distilled water at room temperature for less Meie Zou: Senior Technician - Teaching. Department of Applied Science, AUT.

Tim Layt: Laboratory and Health & Safety Manager, School of Science, AUT.

Yuan Tao: Research Officer - SEM. Department of Design & Creative Technologies, AUT.

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