



# Enamel shear bond strength of different sealants: in vitro study

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## Abstract

**Objective** The aim of this study was to investigate the enamel shear bond strength of four sealants in vitro.

**Materials and methods** Sixty caries-free human premolars were randomly divided into four groups of 15. The materials used were a local-made sealant (LM), Super-Bond C&B (SB), Delton<sup>®</sup>(D) and Concise<sup>™</sup>(C). Each material was prepared according to the instruction of the manufacturer. The sealant was placed in a mold, cured, stored in distilled water for 24 hr and shear bond strength determined by the Instron Universal Testing Machine at a crosshead speed of 0.5 mm/min.

**Results** The results in MPa were: LM, 12.67 ± 2.16; SB, 20.91 ± 1.60; D, 11.98 ± 3.24 and C, 11.60 ± 3.44. Statistical analysis (One-way ANOVA) and Tukey test revealed that SB yielded significantly higher bond strength ( $p < 0.001$ ) while the other three materials did not exhibit significant difference.

**Conclusion** Based on in vitro enamel shear bond strength values, SB exhibited the highest strength. The clinical relevance of this has not been established.

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**Key words:** Pit and fissure sealant; Shear bond strength

## Introduction

Dental caries constitutes a major dental health problem in Thai children. According to the most recent national oral health survey conducted by the Ministry of Public Health in the year 2000, it was found that about 57.3% of children aged 12 experienced dental caries in the permanent dentition.<sup>1</sup> The majority of dental caries occurred in the pits and fissures of the first permanent molars, since these occlusal surfaces with the anatomic pits and fissures are

susceptible areas for initiation of dental caries. Although the use of fluoride has been shown to be highly effective in prevention of caries on smooth surfaces, pit and fissure areas receive minimal protection from either systemic or topical fluoride.<sup>2</sup> Thus there is a need to reduce or eliminate dental caries in the population, primarily targeting the occlusal surfaces of posterior teeth.

'Occlusal sealants', or 'pit and fissure sealants', is defined as the application and mechanical bonding of a resin material to an acid-etched enamel surface, thereby sealing existing pits and fissures from the oral environment. This mechanism prevents bacteria from colonizing in the pit and fissure areas, and the sealant acts as a physical barrier between the caries susceptible enamel surfaces and the rest of the oral environment.<sup>3</sup> Numerous studies have indicated that pit and fissure sealants effectively prevent or arrest dental caries.<sup>4-8</sup> However, the efficacy of sealants is based on the retention of the material. The caries preventive effect of sealants is achieved as long as the sealants remain intact and adhere firmly to the tooth surface.<sup>4-9</sup>

At present, the material of choice for pit and fissure sealant is a resin of Bis-GMA type, which is the reaction product of bis-phenol A and glycidyl methacrylate. Several factors may influence the efficiency of adhesive techniques. The main drawbacks of sealants are loss of material and/or microleakage. Adequate isolation is the most critical aspect of the sealant application process. Contamination of a surface by saliva during or after acid etching may have a deleterious effect on bonding. In addition, most of the materials available in Thailand are imported, thus expensive. Two possible alternatives to solve these problems are to find a hydrophilic sealant or to develop a local-made inexpensive material. Recently, a local-made sealant was developed by the National Metal and Materials Technology Center (MTEC). Its physical properties as well as cytotoxicity were tested and the laboratory results were comparable to commercial sealants.<sup>10,11</sup>

Another possible candidate for pit and fissure sealant is Super-Bond C&B, a self-curing adhesive resin cement. This cement is different from conventional cements in its monomer of 4-methacryloxyethyl trimellitate anhydride (4-META) and an additional polymerization initiator tri-n-butyl borane (TBB). No inorganic fillers of fine glass and silica were added so the cement is more flexible. It can bond to dentin, enamel, metal, porcelain and resin. Due to the good adhesive property of Super-Bond C&B, it is of our interest that the cement may be used as a pit and fissure sealant.

The objective of the present study is to evaluate and compare the enamel shear bond strength of four different sealants, i.e. two innovative ones (Local-made sealant and Super-Bond C&B) and two commercially available sealants (Delton and Concise), on human premolars *in vitro*.

## Materials and Methods

The following materials were evaluated:

1. Delton<sup>®</sup> light cured pit and fissure sealant, tinted (Johnson and Johnson, East Windsor, NJ, USA). Batch number 990225.
2. Concise<sup>™</sup> Light Cured White Sealant (3M Dental Products St. Paul, MN, USA). Batch number 19970528.
3. Local-made sealant, light curing, tinted (MTEC, Bangkok, Thailand). Batch number 301198.
4. Super-Bond C&B, self curing, ivory (Sun Medical CO., LTD., Moriyama, Shiga, Japan). Batch number 70601.

Sixty extracted caries-free human premolars, stored in distilled water, were used in this study. Each specimen was prepared by mounting the coronal portion of a tooth in the middle of a plastic ring (22 × 10 mm) with autopolymerizing acrylic resin. A flat bonding site was prepared on the buccal enamel surface of each specimen by wet grinding on a water-cooled, abrasive wheel with 800 grit silicon carbide paper, exposing at least 3 mm of enamel surface for bonding. Then all flat enamel surfaces were cleaned with aqueous slurry of pumice, washed and air-dried. The specimens were divided randomly into four groups of 15 each. After the ground surfaces were etched with phosphoric acid at the concentrations and times as recommended by the manufacturers, they were washed and air-dried. Custom-made silicone molds, 3.0 mm inside diameter by 2.0 mm height, were placed on the flat ground enamel surface and filled in with sealants. Polymerization of the light cured materials (Delton, Concise, and local-made sealants) were carried out by the application of curing light (XL3000, 3M Dental Products St. Paul, MN, USA) for 20 seconds, whereas Super-Bond C&B was left undisturbed for 30 minutes after mixing. After curing, the silicone molds were carefully removed, and all the bonded specimens were stored in distilled water at 37°C for 24 hours. The shear bond strength was determined using an Instron Universal Testing Machine (Instron<sup>®</sup> Corporation, Canton, MA, USA) with a cross head speed of 0.5 mm/min. The amount of force required to debond the specimen was measured and the shear bond strength was calculated in Megapascal (MPa) units.

### Statistical analysis

Shear bond strength values of the four sealants were expressed as mean ± standard deviations from the mean. Comparisons between groups were assessed by a one-way analysis of variance (ANOVA) and Tukey multiple com-

parison test. Differences were regarded as significant when  $p < 0.05$ .

**Results**

Figure 1 illustrates the enamel shear bond strength results and standard deviations for the four different sealant materials. It was clearly demonstrated the Super-Bond C&B, with the mean bond strength of  $20.91 \pm 1.60$  MPa, had the highest value whereas Concise had the lowest value.

An analysis of variance (ANOVA) revealed that there were significant differences among the four sealants ( $p < 0.001$ ) as shown in Table 1. Shear bond strength values in the Super-Bond C&B group were significantly greater than the other three materials. The mean differences of Super-Bond C&B were 8.93 (95% C.I. 6.31, 11.55  $p < 0.001$ ) compared to Delton, 9.31 (95% C.I. 6.69, 11.94  $p < 0.001$ ) compared to Concise and 8.25 (95% C.I. 5.63, 10.87,  $p < 0.001$ ) compared to the local-made sealant (Tukey test). However, no significant differences were found between Delton, Concise and the local-made sealant (Table 2).

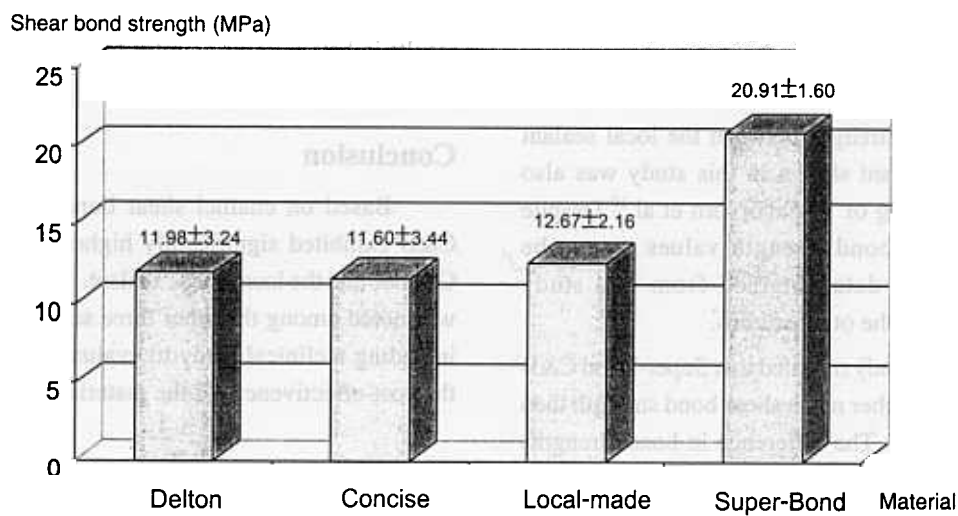


Fig. 1 Shear bond strength to enamel of four different sealants (MPa)

**Table 1** Comparisons of the mean enamel shear bond strengths of four sealants using One-Way ANOVA

Source of variation	SS	df	MS	F	p
Between groups	886.39	3	295.46	40.17	0.00*
Within groups	411.90	56	7.36		
Total	1298.30	59			

\* $p < 0.001$

**Table 2** Multiple comparisons of the mean enamel shear bond strengths of four sealants using Tukey test.

Material	Mean difference	95% C.I	p-value
Delton - Concise	0.38	-2.23 - 3.01	0.98
Delton - Local-made	-0.68	-3.30 - 1.94	0.90
Concise - Local-made	-1.07	-3.67 - 1.56	0.71
Super-Bond - Delton	8.93	6.31 - 11.55	0.00*
Super-Bond - Concise	9.31	6.69 - 11.94	0.00*
Super-Bond - Local-made	8.25	5.63 - 10.87	0.00*

\* $p < 0.001$

## Discussion

Laboratory adhesion tests are performed to evaluate the efficacy of experimental restorative systems or to predict the clinical performance of commercial bonding systems. Although laboratory studies cannot be extrapolated directly to clinical situations, they are useful to serve as screening tests.<sup>12</sup> The measurement of bond strength is one of the parameters in the laboratory adhesion test. Since there are variations in test methodologies, the results obtained from different laboratories cannot be directly compared.<sup>12,13</sup> In this study, standardization of bond strength testing was performed by following the procedure outlined by the ISO (International Organization for Standardization) in ISO CD TR 11405 Dental Materials-Guidance on testing of adhesion to tooth structure.

The results of this study, indicating the average enamel shear bond strength of different materials are in agreement with previous works using Delton<sup>14,15</sup> and Concise.<sup>10,16</sup> Comparable shear bond strength between the local sealant and the commercial sealant shown in this study was also consistent with the finding of Tossaborvorn et al.<sup>10</sup> Despite the fact that the shear bond strength values cannot be directly compared, the data obtained from this study showed similar trends as the other studies.

The results of this study revealed that Super-Bond C&B exhibited significantly higher mean shear bond strength than the other three materials. The difference in bond strengths may be related to the chemical compositions of the materials. Delton, Concise and the local-made sealant are composed of a Bis-GMA resin, while Super-Bond C&B consists of a small amount of 4-META, a derivative of methyl methacrylate (MMA), in its monomer and an additional TBB as a catalyst. 4-META, a monomer contained both hydrophobic groups (like the phenyl group) and hydrophilic groups (like the carboxyl groups) within the molecule, acts as a diffusion promoting monomer.

According to the generally accepted tag theory of enamel bonding, pretreatment using phosphoric acid creates mechanical undercuts on the enamel surface. The resin adhesive then penetrates and locks into these undercuts forming resin tags, which result in mechanical retention.

Nakabayashi et al. found that additional of 4-META to the monomer promotes the diffusion of monomers deeper into the tooth structure.<sup>17</sup> The resin impregnated the interprismatic material and formed a new zone of resin-reinforced tissue, i.e. hybrid layer. The authors hypothesized that this hybrid layer was the reason for the dramatic difference

in bonding stability.<sup>17,18</sup> Combination of the ability of 4-META to promote diffusion of MMA and TBB as a polymerization initiator may contribute to the higher bond strength of the material. However, the shortcomings of Super-Bond C&B noted in this study are that the mixing and application requires some operator's experience and the setting time of 30 minutes is rather lengthy. Therefore it may be impractical in clinical use since it is difficult for a child patient to keep his mouth open until the material sets.

The increase in bond strength is expected to result in better retention of sealants in clinical use. Future areas of research suggested would be investigating whether Super-Bond C&B, shortly after initial set before moisture contamination in simulating with the oral cavity, would result in the same high strength. In addition, it is necessary to determine if the observed increase in bond strength measured in vitro results in better retention rates for sealants in children.

## Conclusion

Based on enamel shear bond strength, Super-Bond C&B exhibited significantly higher retention than Delton, Concise and the local-made sealant. No significant difference was noted among the other three sealants. Further research including a clinical study to evaluate the retention rate and the cost-effectiveness of the material is needed.

## Acknowledgement

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# แรงยึดเหนี่ยวของวัสดุเคลือบหลุมร่องฟันต่างชนิด: การศึกษาในห้องปฏิบัติการ

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## บทคัดย่อ

**วัตถุประสงค์** การศึกษานี้มีวัตถุประสงค์เพื่อเปรียบเทียบแรงยึดเหนี่ยวของวัสดุเคลือบหลุมร่องฟัน 4 ชนิด โดยเป็นการศึกษาเชิงทดลองในห้องปฏิบัติการ

**วัสดุและวิธีการ** ใช้ฟันกรามน้อยจำนวน 60 ซี่ แบ่งออกเป็น 4 กลุ่มเท่าๆ กันโดยการสุ่ม วัสดุเคลือบหลุมร่องฟันที่ใช้ทดสอบได้แก่ วัสดุเคลือบหลุมร่องฟันที่ผลิตในประเทศไทย (LM) Super-Bond C&B (SB) Delton (D) และ Concise (C) วิธีทดสอบ นำวัสดุเคลือบหลุมร่องฟันยึดกับผิวเคลือบฟันตามคำแนะนำของผู้ผลิต โดยใส่ลงในแบบหล่อที่ทำขึ้น เมื่อขึ้นตัวอย่างแข็งตัวนำไปแช่ในน้ำกลั่นเป็นเวลา 24 ชั่วโมง หลังจากนั้นนำไปทดสอบแรงยึดเหนี่ยวด้วยเครื่องทดสอบสากลอินสตรอนที่ความเร็ว 0.5 มิลลิเมตร ต่อนาที

**ผลการศึกษา** ผลการทดสอบพบว่าค่าแรงยึดเหนี่ยวดังนี้: LM,  $12.67 \pm 2.16$ ; SB,  $20.91 \pm 1.60$ ; D,  $1.98 \pm 3.24$  และ C,  $11.60 \pm 3.44$  MPa ผลการทดสอบด้วยสถิติการวิเคราะห์ความแปรปรวนแบบทางเดียวและการทดสอบตุ๊ก ที่ระดับความเชื่อมั่นร้อยละ 95 พบว่า SB มีค่าแรงยึดเหนี่ยวมากที่สุด ( $p < 0.001$ ) ในขณะที่วัสดุอีก 3 ชนิดที่ทดสอบมีค่าเฉลี่ยแรงยึดเหนี่ยวไม่แตกต่างกันอย่างมีนัยสำคัญทางสถิติ

**สรุป** จากการศึกษาแรงยึดเหนี่ยวในห้องปฏิบัติการ พบว่า SB มีค่าแรงยึดเหนี่ยวมากที่สุด อย่างไรก็ตามควรมีการทดสอบทางคลินิกต่อไป

(ว.ทันต. จุฬาฯ 2546;26:23-8)