Strength of a 'No-Bottle' Adhesive System Bonded to Enamel and Dentine

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Abstract: Existing bonding systems permit effective bonding to enamel or dentine. Bonding to dentine is mainly achieved through the hybridization of dentine with resin. However, despite their success, the 'three-bottle' systems do have drawbacks – the large number of steps involved may be confusing and prone to errors of application, as well as being time consuming. Recently developed systems have significantly reduced the number of steps and the total treatment time, but deliver a reliable outcome.

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Clinical Relevance: Effective bonding systems that incorporate a simpler delivery system and reduce the number of application steps can minimize errors during placement and lead to more successful restorations. They also reduce the treatment time for both the patient and the clinician.

Modern restorative dentistry places a definite emphasis on adhesion. Adhesive agents for dentine bonding are widely employed, although the multiple application steps involved make the process time consuming and thus unpopular. The currently accepted mechanism of bonding to dentine is understood to consist of an interpenetrating interfacial network of collagen and polymerized monomer, typically 4 microns in thickness, known as the hybrid layer.[1–3] Dentine bonding systems consisting of separate conditioners, primers and bonding liquids have been well established as effective agents for bonding to dentine and enamel. The conditioners have an inorganic acidic component, commonly aqueous orthophosphoric acid, to demineralize the surface of the dentine. Their use is followed by a primer containing a hydrophilic solvent and a hydrophilic/hydrophobic bifunctional molecule that penetrates the exposed collagen fibrils. Finally, dimethacrylate resin monomers are able to interact with the primer solute and contribute to the hybrid layer.

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TRENDS IN DEVELOPMENT OF ADHESIVES

Simpler bonding systems have been favoured for polyacid modified composites (compomers),[4] principally for non- or low-stress-bearing regions of primary and secondary teeth. These may avoid the use of a conditioning or etching step. However, the primer solutions applied may incorporate some self-etching capability. This trend with compomer adhesives has also been seen in relation to bonding with more conventional resin composites, leading to so-called 'single-bottle' adhesives.[5,6]

In all of these developments an important consideration has been the ease of use and convenience of the delivery systems. Although the three-component (bottle) systems are successful, they present several drawbacks: significant time is required to apply each step (up to 2 minutes), and error in performing one of the steps (e.g. over-etching, over-wetting) will result in failure of the restoration. Therefore, the trend has been towards developing simpler and less time-consuming adhesives. However, a major dilemma in simplification has been whether combining conditioning, priming and bonding into a single bottle will compromise the specificity and shelf life.[7] An alternative approach is a 'twin-barrelled' delivery device (Clicker System, 3M, Co., USA), which advances paired droplets of adhesive components from separate reservoirs with a single press.[7]

This study was designed to evaluate the strength performance of a new adhesive system which is presented in a novel 'no-bottle' delivery system. This incorporates a pair of discrete reservoirs in a single sheath device combined with a disposable applicator

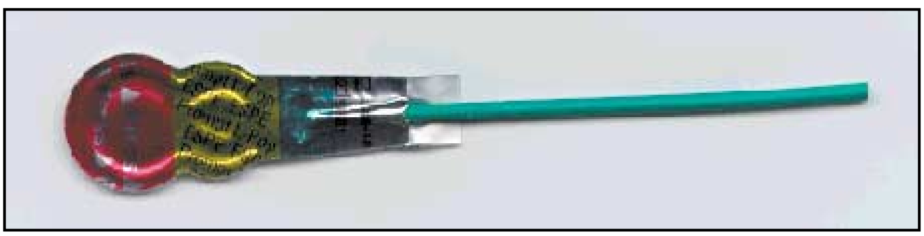

Figure 1. The L-POP delivery device for Prompt adhesive, showing the colour-coded reservoirs.

Group	Code	Adhesive system	Restorative	Manufacturer
1	PL/HY	Prompt L-POP	Hytac Aplitip	ESPE AG, Germany
2	P&B/DY	Prime & Bond 2.1	Dyract AP	Dentsply, DeTrey, England
3	SY-SC/CG	Syntac Single-Component	Compoglass	Ivoclar AG, Liechtenstein
4	F2 P-A/F2	F2000 Primer/Adhesive in Clicker	F2000 Compomer	3M Dental Products, St. Paul, USA

Table 1. *Materials used in the study.*

tip and compartment. The objectives were to compare resultant shear bond strengths with a number of established competitor systems and to image the etching pattern produced by the new self-etching adhesive molecules on dentine surfaces.

THE PROMPT L-POP SYSTEM

The Prompt L-POP system (ESPE Dental AG, Germany) is a two-component adhesive consisting of two reservoirs and a disposable applicator compartment. These are colour-coded red, yellow and green (Figure 1). The red part contains methacrylated phosphoric acid derivatives (esters), photosensitizers and stabilizers, while the yellow part contains water and soluble fluoride components. The green part houses the applicator tip. The advantages of this delivery system are that the separation of the reservoir pockets ensures that there is no possible mixing of the components before application, thus extending the shelf life. In addition, this system is simple to use and minimizes the number of steps involved. It is a 'non-rinse' system and thus does not require intermediate washing and drying steps, which greatly reduces application times.

The L-POP system is activated by successive operations of squeezing (on the red reservoir), folding (red on yellow), and squeezing (red plus yellow). This ejects the mixed components around the applicator brush head in the green chamber. The adhesive is applied via the brush to the entire surface of the cavity: enamel *and* dentine. This may entail rubbing into the demarcated area with rotating movements for 15 seconds. A gentle stream of air is then applied, facilitating solvent evaporation and ensuring that a glossy surface appearance is produced. The applicator tip may be re-wetted and the mixed components remain active for 1 hour. This enables re-application, presuming that contamination has not occurred. The applicator system is, however, intended as a single-use device, for the avoidance of cross-contamination.

INVESTIGATIONS OF BOND STRENGTH

Sample Preparation

Recently extracted human molar and premolar teeth with no restoration or decay were used in this study, and were stored according to international recommendations.[8] The teeth were washed vigorously under running tap water to remove plaque and gingival tissues and then left to dry at room temperature. Roots were removed by sectioning at the enamel–cementum junction.

The teeth were sectioned with a low-speed diamond saw under water cooling to obtain dentine slices parallel to the occlusal surfaces, in the upper third of the tooth crowns. Specimens of enamel were also prepared from buccal surfaces by sectioning to 1.0–1.5 mm thickness.

A total of 80 specimens were prepared for shear bond strength measurements (eight groups of ten teeth: four groups for enamel bonding and four for dentine bonding). Specimens were randomly allocated to the groups.

Specimens were initially mounted in a low-exotherm resin composite using moulds and then in dental stone, within brass rings (internal diameter 14 mm). Care was taken during the embedding process to ensure the test surface of specimens was at the same level of the edge of the mould. Specimens were kept wet and stored in tap water before surface preparation and resin application.

Preparing the Bonds

Enamel and dentine surfaces were polished with 600 grit wet SiC abrasive paper. Circular bonding areas of 2 mm diameter were prepared and the adhesive systems applied (these are listed in Table 1). In the case of the L-POP system the liquid on the micro-brush was rubbed into the demarcated area of the specimens with rotating movements for 15 seconds. The treated surface was then gently dried with oil-free compressed air for 10 seconds, so that a shining damp layer was obtained. Hytac Aplitip restorative (Elipar® Highlight light-curing unit, ESPE Dental AG, Germany) was injected into a Teflon mould and polymerized for 40 seconds. Control groups were prepared and bonded to the enamel and dentine with the following adhesive systems:

- Prime & Bond 2.1/Dyract AP;
- Syntac Single-Component/ Compoglass; and

Material shear bond strength (MPa)	Group 1	Group 2	Group 3	Group 4
Enamel	31.12 (3.96)^{A}	14.67 (1.48)^{C}	21.19 (2.21)^{B}	23.51 (3.21)^{B}
Dentine	22.46 (3.43)^{B}	15.52 (1.28)^{C}	15.40 (1.28)^{C}	15.62 (1.09)^{C}

Table 2. *Mean shear bond strength values. ANOVA gave F = 44.2; P<0.001. Same letters denote groups that are not statistically different (P>0.05, SNK). Standard deviations are shown in parentheses.*

Material	Group 1	Group 2	Group 3	Group 4
Enamel	6C, 4A	10A	4C, 6A	4C, 5A, 1M
Dentine	6C, 4A	10A	10A	10A

Table 3. *Modes of failure. C =cohesive; A = adhesive; M = mixed failure.*

- F2000 Primer-Adhesive/ F2000 Compomer.

All were applied according to the manufacturer's recommendations.

After finishing the preparation, the Teflon moulds were dissected away with a scalpel, resulting in smooth and cylindrical restorative specimens bonded to the enamel or dentine surfaces. These were stored in tap water at 37° C for 24 hours before testing.

Measuring Bond Strength

Shear bond strengths of the specimens were measured with a Universal Testing Machine at a standard crosshead speed of 0.5 mm/min and using a knife-edge blade placed parallel to the bonded surfaces, to induce fracture. The values obtained are shown in Table 2.

Fractured specimens were placed under an optical microscope to view the complete failure area at X20. The mode of failures were categorized as follows:

A: adhesive failure (between the dentine bonding agent and the tooth structure, either enamel or dentine);
C: cohesive failure (within tooth structure);
M: mixed failure (adhesive and cohesive fracture of the material with part of it remaining on the tooth structure).

See also Table 3.

Data were statistically analysed by one-way ANOVA, and the SNK test was used to detect differences in shear bond strength between the adhesive and substrate groups.

Shear bond strengths recorded at 24 hours are shown in Figure 2.

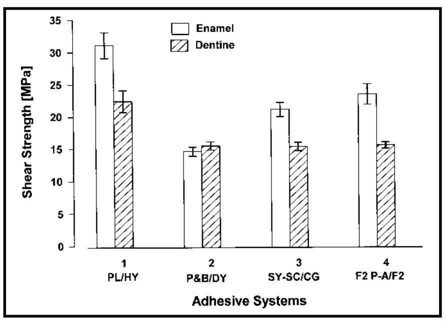

Figure 2. *Bond strength data for the adhesive agents tested. See Table 1 for codes used.*

The shear bond strength has some limitations: the elastic modulus of the substrate does influence the magnitude of the failure stresses encountered.[9] However, in the present work, substrates involved with each of the different test and adhesion groups were all human enamel or dentine.

Atomic Force Microscope Study

The effect of the Prompt L-POP self-etching adhesive on dentine surfaces was examined using Atomic Force Microscopy (AFM). Dentine discs were produced in a manner similar to that described above and the surfaces were polished with 600 grit wet SiC paper. The adhesive was then applied for 15 seconds, rinsed off and air dried. A multi-mode scanning probe microscope (Nanoscope IIIa, Digital Instruments, CA, USA) operating in Tapping mode was used, and the tubule dimensions were analysed. An AFM micrograph of a dentine surface etched with Prompt L-POP is shown in Figure 3. Exposed tubules were clearly visible. These had a mean diameter of 4.05 (±0.23) microns, and a mean depth of 1.64 (±0.17) microns. The roughness parameter (Ra) of a 50 x 50 microns scan size was 395 nm.

DISCUSSION

The aqueous phosphoric ester solution of Prompt L-POP acts as the etchant of the system. It has a low pH (1.8) and can etch the enamel or dentine in similar manner to phosphoric acid itself. This was demonstrated in the AFM results, which revealed a type of etching comparable to that of ScotchBond One and ScotchBond MP.[10] Dentine tubules were shown to be adequately exposed after application, showing that the smear layer was substantially removed. A necessary, if not sufficient, condition was thus met for hybrid layer formation.

The maleic acid in the formulation of Syntac Single-Component acts as the etchant of the system. It partially dissolves the smear layer, while the monomers can penetrate the network of collagen and form the hybrid layer.[11] Prime and Bond 2.1 and F2000 adhesive are both considered to form conventional hybrid layers by a similar etch and penetration mechanism.

CONCLUSION

All adhesive groups gave relatively high bond strengths. However, the Prompt L-POP system exhibited significantly higher shear bond strength values to both enamel and dentine than the controls. This novel delivery system produced excellent *in vitro* results and reduced the number of work steps – which may also reduce the risk of incorrect clinical application.

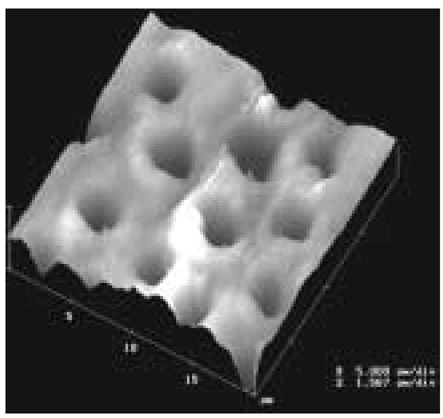

Figure 3. *The self-etching pattern produced on human dentine by Prompt L-POP, imaged by an atomic force microscope. This shows the clearly patent tubule openings, confirming the etching efficiency.*

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BOOK REVIEW

Local Anaesthesia in Dentistry. By P. D. Robinson, T. R. Pitt Ford and F. McDonald. Heinemann, Oxford, 2000 (104pp., £13.99). ISBN 0-72361063-0.

This is the second pocket-sized paperback from Paul Robinson this year and really serves to update two previous older texts on a subject central to the practice of dentistry, local anaesthesia. Its 11 chapters cover all you would expect on such a subject, from the fundamental physiology and pharmacology, through techniques of administration, to complications and problem areas. The format of the text lends itself to easy reading and is complemented by numerous line drawings, black and white photographs and tables.

Current practices are emphasized, particularly the use of aspirating syringes which, if used more widely, might prevent a large number of the ‘adverse reactions’ to adrenaline. On that subject, it was pleasing to see an explanation of direct challenge testing for allergy to local anaesthetics. The use of lignocaine and adrenaline is recommended for most patients and the rarely contemplated risks and side-effects of using alternative formulations, such as prilocaine, are also highlighted. Surprisingly, re-sheathing of needles was advocated, not a procedure we endorse in our establishment.

A sign of the times perhaps, but the final chapter deals with the medico-legal considerations of local anaesthetic use and, although not common, is food for thought for all of us.

The text is obviously aimed at undergraduate dental students and is the sort of text unlikely to be referred to often. However, practising dentists would learn from reading this book, particularly for the few ‘unconventional’ patients that come their way. One suggested improvement would be a table outlining the specific injections required for particular teeth and procedures, something dental students and some dentists have difficulty grasping. Despite this, the book is to be highly recommended.

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ABSTRACTS

HOW BONDED IS A BONDED CROWN?

In Vitro Leakage of Resin-bonded All-porcelain Crowns. M. Ferrari, F. Mannocci, P. N. Mason, G. Kugel. *Journal of Adhesive Dentistry* 1999; **1:** 233-242.

The demand for all-ceramic restorations has resulted in the development of high-translucency and low viscosity cements to permit proper seating of the restorations. It is suggested that these cements have a low solubility in the oral environment, and may allow seating of the restoration to within 20 microns.

This study evaluated the sealing ability of single all-porcelain crowns with margins placed on cementum-dentine, comparing two different luting systems. Twenty extracted molars were prepared in a standardized manner, and crowns fabricated. Following cementation, the specimens were subjected to thermal cycling, and then stained for marginal leakage at the cervical margin. Finally, an evaluation was made of the cement thickness, and the formation of a hybrid layer between the cement and dental substrate.

The results showed that up to 80% of the specimens exhibited leakage which extended up to the axial wall of the preparation. The average cement thickness was 82 microns. There was no significant difference between the two cement systems.

DO YOU TRUST QUESTIONNAIRE SURVEY REPORTS?

Validity of a Questionnaire Survey: The Role of Non-response and Incorrect Answers. O. Sjöström, D. Holst, S. O. Lind. *Acta Odontol Scand* 1999; **57:** 242-246.

Questionnaire surveys are often used in health research. However, this interesting paper must cause the reader to have doubts about the validity of many of the reports in the dental literature based upon such postal questionnaire type surveys. The authors sent a questionnaire to 9,283 Swedish citizens, for whom the answers to the questions could be checked and verified. They received a 43% response, which creates a bias in its own right. Whilst it would be almost impossible to achieve a 100% response rate, it is known that the further removed from this figure the greater the potential bias, and the less credible the results.

What the authors found, however, was that of the 3,949 responders, 14% gave an untrue answer to one or more of the questions. Whilst this may not be deliberate, the reader may be more cautious in interpreting the results of such surveys in the future!

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