กำลังแรงยึดแบบดึงระดับจุลภาคของวัสดุเรซินคอมโพสิตชนิดไซลอเรน เมื่อใช้สารยึดติดระบบเดียวกันในการบูรณะฟันคลาสวัน

นางสาว นันทวรรณ กระจ่างตา

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาทันตกรรมหัตถการ ภาควิชาทันตกรรมหัตถการ
คณะทันตแพทยศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย
ปีการศึกษา 2553

MICROTENSILE BOND STRENGTH OF SILORANE-BASED RESIN COMPOSITE AND ITS CORESPONDING ADHESIVE IN CLASS I RESTORATION

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Science Program in Operative Dentistry
Department of Operative Dentistry
Faculty of Dentistry
Chulalongkorn University
Academic Year 2010

Thesis Title	MICROTENSILE BOND STRENGTH OF SILORANE-BASED RESIN COMPOSITE AND ITS CORESPONDING ADHESIVE IN CLASS I RESTORATION
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นันทวรรณ กระจ่างตา : กำลังแรงยึดแบบดึงระดับจุลภาคของวัสดุเรซินคอมโพสิตชนิดไซลอเรนเมื่อใช้สารยึดติดระบบเดียวกันในการบูรณะฟันคลาสวัน. (MICROTENSILE BOND STRENGTH OF SILORANE-BASED RESIN COMPOSITE AND ITS CORESPONDING ADHESIVE IN CLASS I RESTORATION) อ. ที่ปรึกษาวิทยานิพนธ์หลัก: ผศ.ทพญ.ดร. ศิริวิมล ศรีสวัสดิ์, 96 หน้า

การศึกษานี้มีวัตถุประสงค์เพื่อศึกษาค่ากำลังแรงยึดแบบดึงระดับจุลภาคของวัสดุเรซินคอมโพสิตชนิดไซลอเรน (ฟิลเทค พี 90/ สารยึดติดระบบไซลอเรน) เปรียบเทียบกับวัสดุเรซินคอมโพสิตชนิดเมธาคริเลต (ฟิลเทค แซด 250/ สก็อตช์บอนด์มัลติเพอร์โพส) และศึกษาผลของซีแฟคเตอร์ และเวลาที่เก็บในน้ำต่อค่ากำลังแรงยึดแบบดึงระดับจุลภาคในการบูรณะฟันคลาสวัน โดยนำฟันกรามมนุษย์ซี่สุดท้ายมาเตรียมให้ได้พื้นผิวแบนของผิวเนื้อฟัน และแบ่งฟันออกเป็น 8 กลุ่มโดยการสุ่มตามชนิดของเรซินคอมโพสิต ซีแฟคเตอร์ และเวลาที่เก็บในน้ำ ดังนี้ (1) ฟิลเทค พี 90/ ซีแฟคเตอร์สูง/ 24 ชั่วโมง (2) ฟิลเทค พี 90/ ซีแฟคเตอร์ต่ำ/ 24 ชั่วโมง (3) ฟิลเทค แซด 250/ ซีแฟคเตอร์สูง/ 24 ชั่วโมง (4) ฟิลเทค แซด 250/ ซีแฟคเตอร์ต่ำ/ 24 ชั่วโมง (5) ฟิลเทค พี 90/ ซีแฟคเตอร์สูง/ 3 เดือน (6) ฟิลเทค พี 90/ ซีแฟคเตอร์ต่ำ/ 3 เดือน (7) ฟิลเทค แซด 250/ ซีแฟคเตอร์สูง/ 3 เดือน (8) ฟิลเทค แซด 250/ ซีแฟคเตอร์ต่ำ/ 3 เดือน หลังจากเก็บตัวอย่างในน้ำครบ 24 ชั่วโมง และ3 เดือน นำชิ้นทดสอบรูปนาฬิกาทรายมาทดสอบค่ากำลังแรงยึดระดับจุลภาคที่ความเร็วในการดึง 1 มิลลิเมตรต่อนาที และบันทึกประเภทการล้มเหลว นำข้อมูลมาวิเคราะห์ด้วยการวิเคราะห์ความแปรปรวน 3 ทาง และใช้การทดสอบแทมเฮนส์ทดสอบความแตกต่างค่าเฉลี่ยแบบพหุคูณ ที่ระดับนัยสำคัญ 0.05 พบว่าชนิดของ เรซินคอมโพสิต ซีแฟคเตอร์ และเวลาที่เก็บในน้ำมีผลต่อค่ากำลังแรงยึดระดับจุลภาคอย่างมีนัยสำคัญ โดยกลุ่ม ฟิลเทค แซด 250/ ซีแฟคเตอร์ต่ำ/ 24 ชั่วโมง มีค่ากำลังแรงยึดระดับจุลภาคสูงสุด (54.19 ± 9.05 เมกะปาสคาล) และฟิลเทค พี 90/ ซีแฟคเตอร์สูง/ 3 เดือน มีค่ากำลังแรงยึดระดับจุลภาคต่ำสุด (6.94 ± 2.07 เมกะปาสคาล) ทุกกลุ่มทดสอบมีค่ากำลังแรงยึดระดับจุลภาคลดลงในกรณีซีแฟคเตอร์สูง และเก็บในน้ำ 3 เดือนอย่างมีนัยสำคัญ ยกเว้นเมื่อเปรียบเทียบระหว่างกลุ่มฟิลเทค พี 90/ ซีแฟคเตอร์สูง/ 24 ชั่วโมง และฟิลเทค พี 90/ ซีแฟคเตอร์สูง/ 3 เดือน

ภาควิชา ทันตกรรมหัตถการ ลายมือชื่อนิสิต...
สาขาวิชา ทันตกรรมหัตถการ ลายมือชื่อ อ. ที่ปรึกษาวิทยานิพนธ์หลัก...........................
ปีการศึกษา 2553

5176113332 : MAJOR OPERATIVE DENTISTRY
KEYWORDS : MICROTENSILE BOND STRENGTH / SILORANE-BASED RESIN COMPOSITE / C-FACTOR

NANTAWAN KRAJANGTA: MICROTENSILE BOND STRENGTH OF SILORANE-BASED RESIN COMPOSITE AND ITS CORESPONDING ADHESIVE IN CLASS I RESTORATION. THESIS ADVISOR: ASST PROF SIRIVIMOL SRISAWASDI, PH. D., 96 pp.

The present study aimed to investigate microtensile bond strength (μTBS) of silorane-based resin composite (Filtek P90®/ Silorane system adhesive®) compared to methacrylate-based resin composite (Filtek Z250®/ Scotchbond Multipurpose®), and to determine the effect of c-factor and water storage time on μTBS in class I cavity. Extracted human third molars were ground flat to expose dentin, and randomly divided into 8 groups according to the type of resin composite, c-factor and water storage time, group 1 Filtek P90®/high c-factor/24 hours (PHC24h), group 2 Filtek P90®/low c-factor/24 hours (PLC24h), group 3 Filtek Z250®/high c-factor/24 hours (ZHC24h), group 4 Filtek Z250®/low c-factor/24 hours (ZLC24h), group 5 Filtek P90®/high c-factor/3 months (PHC3m), group 6 Filtek P90®/low c-factor/3 months (PLC3m), group 7 Filtek Z250®/high c-factor/3 months (ZHC3m), and group 8 Filtek Z250®/low c-factor/3 months (ZLC3m). After 24 hours and 3 months of water storage, bonded hourglass specimen were tested for μTBS using 1 mm/min cross-head speed and failure types were also determined. Data were analyzed using three-way ANOVA and Tamhane post-hoc test ($\alpha = 0.05$). Significant influence of the type of resin composite, c-factor and water storage time were observed. ZLC24h exhibited the highest μTBS (54.19 ± 9.05 MPa), and PHC3m the lowest (6.94 ± 2.07 MPa). All groups showed significant decrease in μTBS when used in high c-factor and 3 month water storage time, except when compared between PHC24h and PHC3m.

Department : Operative Dentistry Student's Signature ______

Field of Study : Operative Dentistry Advisor's Signature ______

Academic Year : 2010

Acknowledgement

I would like to express my appreciation to all those who have helped to make this thesis a success. I am always deeply grateful to my advisor, Assistant Professor Dr. Sirivimol Srisawasdi for her kindness, for helpful guidance and for spending many hours correcting this paper. Sincere appreciation is extended to all the members of my committee: Assistant Professor Dr. Suchit Poolthong, Associate Professor Chairat Vivatvorrapun and Dr. Pavinee Padipatvuthikul.

I wish to thank staffs of the Dental Material Science Research Center, the Faculty of Dentistry, Chulalongkorn University for their help in laboratory facilities.

I am grateful to all teachers in the Department of Operative Dentistry at Chulalongkorn University for their support and encouragement. Without them this project would not be possible.

I wish to thank my colleagues, graduate students in the division of Operative Dentistry of 2008-2010 for their great assistance and wonderful friendships.

Finally, I would like to express my deepest appreciation to my family for continuous encouragement that enables me to undertake this study.

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CHAPTER I

INTRODUCTION

Background and rationale

Dimethacrylate-based resin composites have been used in dentistry for over 50 years. Developments in filler technology and initiation systems have considerably improved the materials' physical properties and expanded their clinical applications.(1) However, Polymerization shrinkage, which ranged between 1.5 to 5% by volume, has still been regarded as the primary negative characteristic of rsin composites.(2) As resin composite shrunk, stresses developed at tooth-resin composite adhesive interface and within the tooth itself, and they have been found to be dependent upon the cavity shape, size, c-factor, modulus of elasticity of the tooth, characteristics of the resin composite, and rate of polymerization.(3, 4) These factors combine and interact simultaneously in complex ways, translating polymerization shrinkage into tooth stresses.(5)

Polymerization shrinkage stress of resin composite may have adverse clinical consequences, such as enamel fracture, cracked cusps, cuspal movement, post-operative pain, microleakage and recurrent caries.(6, 7) If a resin composite is bonded to cavity walls, shrinkage forces will start to build-up, resulting in stresses on the bond between resin composite and tooth structure.(8) Therefore, in areas where shrinkage forces are higher than the bond strength of resin composite to dental substrate, a gap will develop, particularly when restoration margin is placed in dentin or cementum.(9) Based on the knowledge accumulated over the years, different approaches have been proposed to reduce the magnitude and effects of contraction stress in resin composites. These methods included incremental placement technique(3, 10), development of light curing units with gradually increasing irradiance or pulsed emission (11), and use of low-modulus intermediate layers, known as the elastic wall concept.(12) Also, modifications of the current Bis-GMA resin matrix have been proposed as a means to reduce the stress values without compromising the mechanical properties of resin composites.(13, 14)

Modern resin composites demonstrate improved strength and wear resistance with reduced polymerization shrinkage and water sorption, as compared to earlier versions. Although polymerization shrinkage may be reduced in some systems, the induced contraction stress at the dentin-adhesive-resin composite bonded joint is still believed to lead to restorative failures.(15) Most studies, currently investigating the durability of adhesion, do not take into account polymerization contraction stress. These stresses put resin-tooth interfaces under severe tension, in particular when restoring cavities with high c-factor resulting in less chance to relaxation of shrinkage stress.(4, 16) New approaches in the development of low-shrinkage resin composite have been speculated to reduce the impact of shrinkage stress and produce restorations with greater clinical longevity.(17, 18) Silorane-based resin composite, which reduced shrinkage with a novel ring opening chemistry(18), has been introduced to the dental market within the last few years without a comprehensive set of independent test to validate the performance claimed by the respective manufacturer. Therefore, the aim of this *in vitro* study was to investigate µTBS of silorane-based resin composite compared to methacrylate-based resin composite in class I restoration, with high compared to low c-factor cavities after 24 hours and 3 months of water storage.

Research objective

To compare microtensile bond strengths of silorane-based resin composite and methacrylate-based resin composite, when used with their corresponding adhesive systems, in class I cavities with high compared to low c-factor cavities after 24 hours and 3 months of water storage.

Research question

The manufacturer claims that silorane-based resin composite provides low shrinkage and optimal bond strength. How would artificial aging and c-factor influence microtensile bond strength of silorane-based resin composite compared with methacrylate-based resin composite in class I restoration?

Expected benefit of the study

This study was to clarify the use of silorane-based resin composite by determining bond strength to class I cavities, *in vitro*. The results provide clinicians a consideration when using these low shrinkage resin composite systems in dental practice. Moreover, information obtained may assist researchers for further studies to help develop better resin composite system.

Limitation

The study was a laboratory experiment, the results of this *in vitro* study of microtensile bond test must be considered as comparisons, and not as absolute conclusions.

Hypothesis

The null hypotheses are

1. There is no difference in microtensile bond strengths between silorane-based resin composite and methacrylate-based resin composite, when used with their corresponding adhesive systems, in class I cavities.
2. C-factor does not affect the microtensile bond strengths of silorane-based resin composite and methacrylate-based resin composite, when used with their corresponding adhesive systems, in class I cavities.
3. Artificial aging time does not affect the microtensile bond strengths of silorane-based resin composite and methacrylate-based resin composite, when used with their corresponding adhesive systems, in class I cavities.

Research boundary

The study was performed in the laboratory setting using extracted human molars in order to investigate microtensile bond strengths of a silorane-based resin composite (Filtek P90®, P90) used with its corresponding adhesive (Silorane system adhesive, SIL), and a methacrylate-based resin composite (Filtek Z250®, Z250) used with Scotchbond Multipurpose®, in 2 different c-factor designs of class I restoration. The teeth were bonded according to manufacturers' instructions. Samples were also investigated the effect of 3-month storage in water on microtensile bond strength.

CHAPTER II

LITERATURE REVIEW

Polymerization shrinkage of a resin composite in a high configuration factor (c-factor) cavity led to high polymerization stress.(4, 15) In this study, the effect of c-factor and composite shrinkage (high- vs. low-shrinkage) on the microtensile bond strength (µTBS) to dentin was investigated, so the contents of this review literature are the followings:

1. Laboratory studies related to microtensile bond strength
    1.1. Bond strength testing methods
    1.2. Factors affecting microtensile bond test
        1.2.1 Storage methods
        1.2.2 Type of adhesive resin and substrate
        1.2.3 Surface preparation methods
        1.2.4 Configuration factor (c-factor) and polymerization shrinkage stress
        1.2.5 Artificial aging
        1.2.6 Crosshead speed
        1.2.7 Specimen designs
    1.3. Analysis of microtensile bond strengths using Weibull analysis
2. Polymerization of silorane-based resin composite and methacrylate-based resin composite

Laboratory studies related to microtensile bond strength

Bond strength tests are used to assess the ability of restorative material or dentin bonding system to establish bond between the restorative material and the biological substrate.(19) Bond strength tests are the most frequently used tests to screen adhesives. The rationale behind this testing method is that the stronger the adhesion between tooth and biomaterial, the better it will resist stress imposed by resin polymerization and oral function.

1.1 Bond strength testing methods

Dental manufacturers rely largely on *in vitro* testing to predict the clinical performance of a product.(20) They predominantly utilize tensile bond strength tests to evaluate and market their products. The measurement of bond strength is one of several laboratory parameters used to evaluate the efficacy of adhesives. Bond strength is the force per unit area required to break a bonded assembly with failure occurring in or near to adhesive interface.(21) Most commonly, bond strength is measured by subjecting resin composites bonded to enamel/dentin to tensile or shear stress. However, at bond strength values higher than 20 MPa in a shear test, cohesive failure of the substrate will more likely occur.(22) Therefore, a new test needed to be developed that differentiated between adhesives that produce higher bond strengths. A microtensile bond strength methodology was introduced by Sano et al. in 1994.(23)

Recently, the consensus (24) is that the microtensile bond strength studies are more desirable and have the following advantages: 1) more adhesive fractures, fewer cohesive fractures (cohesive fractures are supposedly not desirable because they do not provide the true interfacial bond strength.), 2) higher interfacial bond strengths can be measured, 3) ability to measure regional bond strengths, 4) means and variances can be calculated for single tooth, 5) permits testing of bonds to irregular surfaces, 6) permits testing of very small surface areas and 7) facilitates examination of the failed bonds by SEM.

The major disadvantages of microtensile bond testing (24, 25) is 1) the rather labor-intensive, 2) technically demanding, 3) samples are so small that they dehydrate rapidly and 4) relatively fragile sample preparation technique (difficult to measure bond strength < 5 MPa). Special care should be taken to avoid/reduce the production of microfractures at the interface during specimen preparation.(24) They may weaken the bond and, thus, reduce the actual bond strength.(24)

The ISO Technical Specification on “Testing the adhesion to tooth structure” (No 11405, second edition 2003) is a useful approach to standardize some important variables for shear and tensile bond strength tests.(21) Table 1 lists some of the important variables which were also identified as most important variables by the meta-analysis of Leloup et.al.(26)

Table 1. Guidance on substrate selection, storage, handling and execution of bond strength test according to the ISO Technical Specification 11405 (21)

Test parameter	Characteristics description
Substrate	Superficial dentin of human permanent premolars or molars, preferably third molars from 16 to 40 year-old individual; Bovine mandibular incisor
Time after extraction	Not more than 6 months
Condition of teeth	Caries free, Unrestored
Storage of teeth	After cleaning storage in distilled water (Grade 3) and refrigerator (4°C) or after disinfection in 0.5% Chloramine T trihydrate for 1 week, storage in distilled water
Number of specimens	" Sufficient numbers of specimen"; for Weibull statistic at least 15 specimens are necessary
Tooth surface preparation	Flat surface prepared with polishing device and 600- grit SiC under running water
Application of adhesive	At room temperature (23± 2 °C) and 50± 5% humidity; limited bond area
Storage of test specimens	Type 1: 24 h in water at 37°C Type 2: Thermocycling (5°C /55°C x 500 cycle), starting after 20 h to 24 h storage in water at 37°C. The exposure to each bath should be at least 20s, and the transfer time between baths should be 5S to 10S. Type 3 : Six months in water at 37 °C
Strain rate	0.75 (±0.3) mm/min crosshead speed, load rate 50 (±2) N
Statistic analysis	Mean, standard division, coefficient of variation CV; CV has to be less than 50% If data are not normally distributed Weibull distribution and statistic (Probability of failure) must be calculated

1.2 Factors affecting microtensile bond test

It is important to note that a bond strength value cannot be considered as a material property.(27) The data recorded depend largely upon experimental factors such as, for example, storage media, type of adhesive resin and substrate, surface preparation methods, c-factor, polymerization shrinkage stress, sample size and geometry, the actual test method and more.(28, 29)

Therefore, the absolute test values can not be used to draw conclusions from, or be compared with, data gathered in other studies. Only relative study outcomes, in the sense of 'A is better than B', are a valid basis for further interpretation of the results. Nevertheless, bond-strength testing can reveal valuable clinical information, when gathered in a well-controlled design. For instance, by introducing an aging factor into the study design, one can assess the durability of adhesion.(27)

1.2.1 Storage methods

The samples used for *in vitro* bonding studies are principally obtained from human or bovine teeth.(30) Teeth from both these sources are contaminated with bacteria so that there is a potential for transmission of communicable diseases.(31) Therefore it is of importance that these teeth are decontaminated in a sterilizing medium before any bond strength tests are done in the laboratory.(31) A variety of medias that possess bactericidal and bacteriostatic properties have been used for storage purposes. Some of the solutions most commonly used as storage media for *in vitro* studies are chloramine, formalin, thymol, sodium hypochlorite and glutaraldehyde.(31, 32) The media, in which teeth are stored after harvesting and the duration of storage, may influence bond-strength results. Distilled water, normal saline (33) or freezing have also been used as methods of storage, although these are clearly unable to decontaminate teeth.(31, 34)

Chloramine T, an intermediate level disinfectant commonly used in distilled water, is recommended by a provisional technical report for guidance on testing of adhesion to tooth structure by the International Organization for Standardization (ISO).(21) ISO recommended that, before storage, the teeth should be thoroughly washed in running water, all blood and adherent tissues removed, and that the teeth should then be stored in distilled water, or in a 0.5% chloramine solution, for at least 1 week. Thereafter, they should be stored in distilled water in a refrigerator at 4°C. In order to reduce deterioration, the storage medium should be replaced periodically. Chloramine was also found to produce non-significantly different dentin shear bond strength values and failure modes as compared to freshly extracted or frozen teeth, unlike irradiation, thymol, methanol and glutaraldehyde (Figure 1).(31)

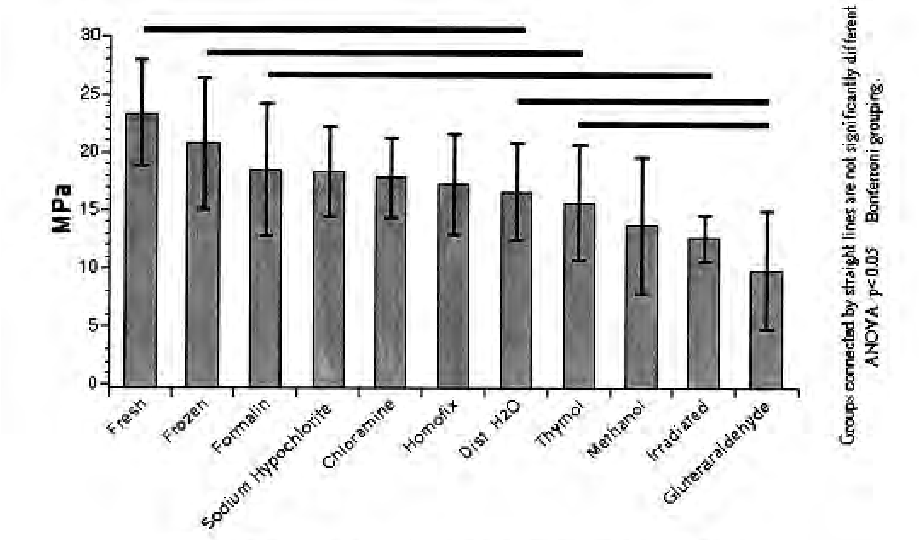


Figure 1. Comparison of shear-bond strength among various storage media. (31)

Extracted teeth undergo significant changes in surface chemistry and physical properties according to storage solution and time.(35) The effects of post extraction time on bond strength seem to be relatively minor for periods from 20 minutes to several years (36), but a six-month storage period has been recommended for dentin resin bond strength testing by ISO.(21) However, it is possible that post mortem changes could occur in dentin, which in turn could affect the outcome of microtensile bond tests, so that freezing the teeth immediately after extraction suspends these changes.(31) A significant influence of storage temperature on dentin bond strength, dentin bond strength is greater at lower temperatures, which probably allow for better preservation of tooth samples.(26) These facts corroborate the observations that the freezing of teeth would be the preferred storage method if the supply of fresh teeth is insufficient. (31)

1.2.2 Type of adhesive resin and substrate

1.2.2.1 Bond to enamel (Figure 2)

When bonding to enamel, an etch and rinse approach still results in the highest bonding effectiveness irrespective of a two- or three- step procedure and the product tested.(37-39) When pooling the microtensile bond strength of all etch and rinse adhesives tested, a μTBS of 39-40 MPa was achieved. A self-etch procedure, in general, has resulted in a lower bonding effectiveness of etch and rinse.(40) A pool μTBS of about 30 MPa was obtained for two-step self-etch adhesives. One-step self-etch adhesives produced significantly lower μTBS than etch and rinse and two-step self etch adhesives.(24, 40)

The pooled μTBS was about 16 MPa for the one-step adhesives, but the strong one step adhesives, present with μTBS in the same range as that recorded for the two-step self-etch adhesives. The glass-ionomer adhesive performed equally well as the two-step self etch adhesives. (24, 37)

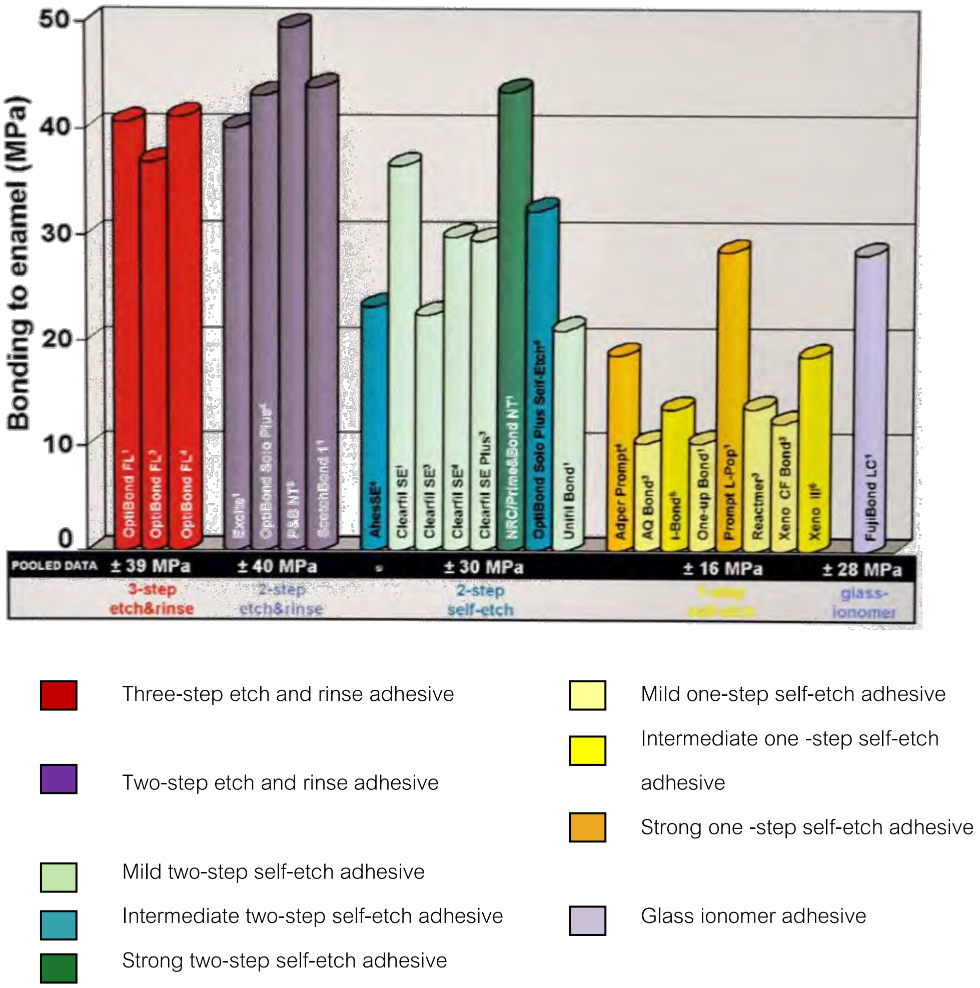


Figure 2. Microtensile bond strength (μTBS) to enamel of diverse commercial adhesive.(24)

1.2.2.2 Bond to dentin (Figure 3)

At dentin, three-step etch and rinse adhesives still surpassed all other adhesives that used simplified application procedures. No significant difference could be recorded between the bonding effectiveness to dentin of two-step etch and rinse and two-step self-etch adhesives. The lowest μTBS for the one-step self-etch adhesives that performed similarly to glass-ionomer based adhesive.(24, 37) The variation in the quality of the dentin substrate, such as presence of moisture, age, etc., also influenced bond strength.(41)

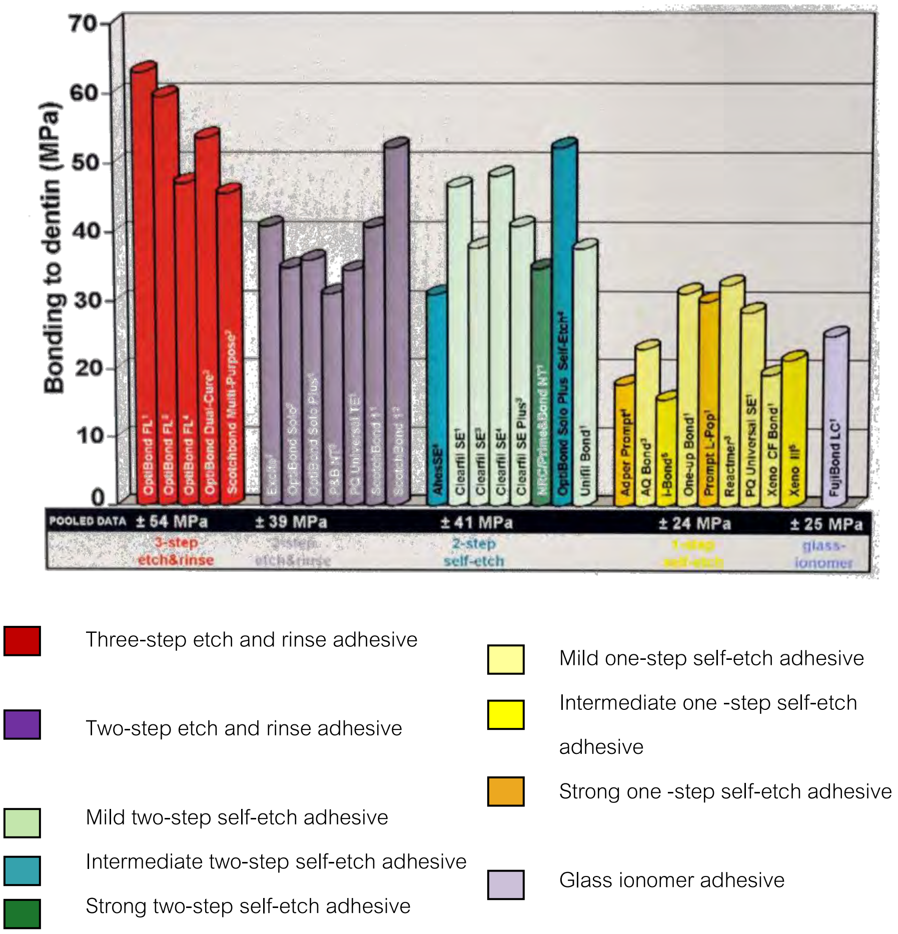


Figure 3. Microtensile bond strength (μTBS) to dentin of diverse commercial adhesive.(24)

1.2.3 Surface preparation methods

Several studies have evaluated the importance of surface preparation methods on bond strength, (42, 43) and have attempted to define the most clinically relevant smear layer preparation for use in *in vitro* tests.(43) The differences in surface preparation methods during laboratory testing can produce a variety of smear layer characteristics that have been reported to affect the bond strengths of resin to dentin.(43-47) The preparation of the sample's surface with a bur *in vitro* is complex and time-consuming, and may be difficult to standardize.(48-51) Most *in vitro* bond strength studies prepared dentin surfaces with a 600-grit abrasive paper.(52) Others treated tooth surfaces with 400-grit, (53) 320-grit, (54) or even 60-grit, (49) abrasive papers *in vitro*. Thickness and roughness of smear layers created by different grades of SiC papers may vary, thus influencing the resin adhesion, especially when self-etch adhesives are used (Figure 4 and 5).(43-45) Some studies (42, 43, 51, 55) showed no difference in bond strength of total-etch adhesive systems to different dentin smear layers, probably because these systems completely removed smeared debris from the surface.

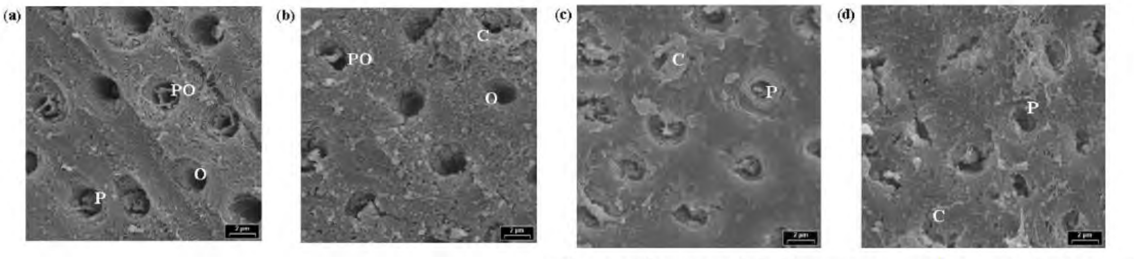


Figure 4. SEM micrographs of the reaction of the smear layer to the Clearfil SE® primer (Kuraray America, Inc.) treatment. (a) 600-grit smear layer; (b) 320-grit smear layer; (c) carbide bur smear layer; (d) coarse diamond bur smear layer. Open tubules (O), partially opened tubules (PO), plugged tubules (P) and closed tubules (C), are indicated in the images. (43)

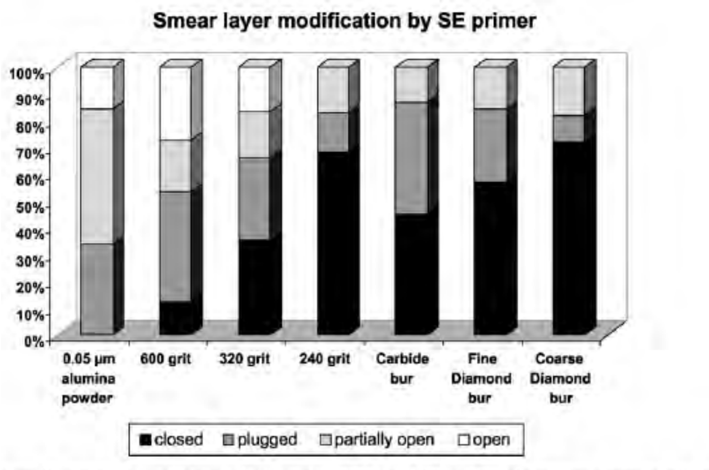


Figure 5. Reaction of the smear layers, by surface preparation method, to the primer of Clearfil SE® Bond(Kuraray America, Inc.). Tubule openness decreased with increased coarseness level and was lower among burs than paper abrasives (both, P < 0.001). The tubule openness was similar for specimens abraded with carbide bur, 240-grit or 320-grit paper. (43)

While several studies have reported low resin–dentin bond strengths over thick smear layer (43-45), there has been a number of published works showing no influence of smear layer thickness on bond strength.(43, 56-58) Regardless of the coarseness of SiC paper, this surface preparation technique may not be clinically relevant.(52) The use of SiC paper produced a looser smear layer and tended to have more open dentinal tubules than that produced by dental burs. Dental burs are available in a variety of types and coarseness, which could produce qualitative and quantitative different smear layers.(43, 46) Oliveira et al.(43) demonstrated that dental burs produced a thinner but more compact smear layer compared to that obtained from SiC papers. An accumulation of smear layer, therefore, might reflect underlying dentin surface topography.(59) Carbide bur, on the other hand, generated the smoothest surface with apparently thin smear layers and smear plugs.(43, 59) This might be due to the fact that carbide bur uses bladed cutting rather than the abrasive cutting of diamond burs or SiC papers. The blades scrape dentin and produce new surface, whereas the abrasive particles wear the surface down, while debris is displaced laterally by passage of the abrasive particles.(60) However, even though carbide bur created the thinnest smear layer of all burs, a study reported that it tended to produce the weakest bond strength.(52) The results were contrary to the studies that were previously reported (43, 61), thus they yet need to be clarified. Under TEM and micro-RAMAN spectroscopy observations, Spencer et al.(62) reported that carbide bur created a fibrous smear layer, composed of well arranged and undisrupted collagen fibrils. This smear layer might not be as easily dissolved by phosphoric acid or acidic monomer conditioning steps and, subsequently, might interfere with the permeation of bonding resin.(63) As resin infiltration of demineralized intertubular dentin has been reported to account for a substantial proportion of adhesion to dentin(20), relatively less resin infiltration in this region with carbide bur preparation might explain the lower bond strengths observed in the current investigation.(52)

1.2.4 Configuration factor (c-factor) and polymerization shrinkage stress

Bond strengths of current adhesive systems may surpass polymerization shrinkage stresses, and possibly affect the magnitude and direction of resin composite polymerization shrinkage vectors.(64) Thus, when bonding to a cavity, one must investigate the effect of cavity configuration factor on bond strength to expedite interpretation of the nature of adhesion within the prepared cavity. (65)

Microtensile bond strength test, which measures the interfacial bond strength more precisely in a significantly smaller sample, helps obtain true ultimate stress and delivers a more uniform stress. Clinically, restorations have a very large surface area, and are placed into a three-dimensional cavity preparation, therefore, greater stresses occur within the material in a three-dimensional cavity preparation. (4, 66) Resin composites shrink as they polymerize, and contraction stresses of up to 7 MPa develop within the resin.(66) However, when resin is bonded to a single surface, as they are for most bond strength studies, flow relaxation occurs relieving some of the contraction forces, and, therefore, these values are not realistic.

Davidson et al.(66) studied the effect of polymerization contraction stress in two-dimensional and three dimensional cavity models using both chemical and light cured resin composite restorative materials. The observed difference in bond durability was attributed to the relatively limited stress relieving flow possibility in the three-dimensional cavity (Figure 6). The authors also hypothesized that a contraction stress of 20 MPa could be generated at the cavity margin in the class V cavity design used in the study. The information above is often used in a simplistic argument for a desired minimum bond strength of 17- 20 MPa for current dental adhesive systems. The more highly constrained the resin composite material is upon setting, the greater the contraction stress will be.

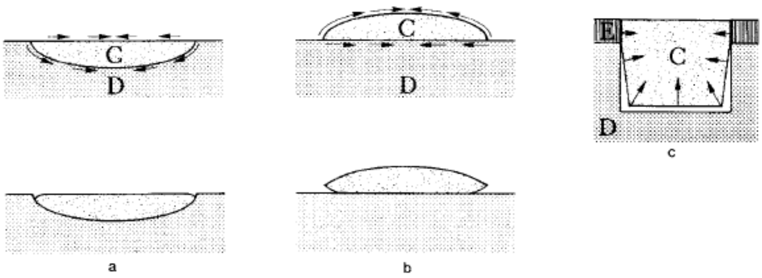


Figure 6. The various contraction patterns in differently shaped cavities and the respective forms of disruption of the adhesion. C = composite, D = dentin, and E = enamel. (66)

According to the study (65) that investigated microtensile bond strength to the dentin of class I cavity floor in high and low c-factor cavities, bond strengths were measured only on the pulpal floors, using three adhesive systems. When the c-factor was increased from 1 to 3 by creation of three-dimensional cavities, the bond strengths of all adhesive systems decreased. The concept of the cavity configuration factor appeared to offer an appropriate explanation for the decrease in bond strengths observed, when deep dentin was bonded as part of a three dimensional cavity. Polymerization stresses would tend to weaken the bond with the lowest strength in the restored cavity.(65) Armstrong et al.(15) and Nikaido et al.(67, 68) previously investigated microtensile bond strength in high versus low c-factor design, and reported that the presence of a high c-factor resulted in lower dentin bond strength to the cavity floor.(15)

1.2.5 Artificial aging

In an oral cavity, the interface between restoration and tooth is exposed to diverse forces that act simultaneously. During setting of a resin composite, resin shrinkage puts stress on the bond, pulling it away from the cavity wall.(64) During function, mechanical stresses by chewing forces, thermal and chemical stresses with changes in temperature and pH have an effect on the bond integrity. Since long term clinical trials are costly and time consuming, laboratory test data are usually used to demonstrate the quality of a dentin adhesive relative to its competition.

Most current adhesives perform well in bond strength test, at least when tested shortly after application, and under controlled *in vitro* conditions.(38, 39, 69) However, the oral cavity with temperature changes, chewing loads and chemical attacks by acids and enzymes, forms a severe challenge for tooth-resin composite bonds. Clinically, marginal deterioration of resin composite restorations remains problematic, and is the major reason that dramatically shortens lifetime of adhesive restorations.(70) A factor known to degrade tooth resin composite bonds is exposure to water.(15, 39, 71, 72) Nanoleakage, or the ingress of oral fluids through nanometer-sized channels along collagen fibrils within the hybrid layer, is considered detrimental to bond integrity.(73-75) The most commonly used artificial aging technique is long-term water storage. Most studies reported significant decrease in bond strengths, even after relatively short storage periods.(76-80) Damage or degradation of the adhesive bond may be elucidated after water storage due to hydrolytic attack of organic tissues or alterations in mechanical properties of resinous components.(15) Most degradation processes are diffusion-rate-dependent. Consequently, the length of the diffusion path is as important as the diffusion time itself. A way to exclude diffusion-dependent effects is to age micro-specimens so as to render the diffusion path as short as possible. For example, when small µTBS sticks were stored, a significant decrease in bond strength to dentin was detected within as few as 90 days.(15, 78) Storing tiny µTBS sticks may thus be considered as a form of accelerated aging.

Enamel-resin bonds, when produced by etch and rinse adhesives, are more stable over time.(81) They could seal off path of water diffusion to the more vulnerable dentin-resin bond, and so retarded bond degradation(39, 74) DeMunck et al. (39) studied long-term degradation of resin-dentin bonds using a microtensile bond strength testing through exposure to water for four years, either directly or indirectly, when resin-dentin interface was surrounded by resin bonded to enamel. Direct exposure to water resulted in a significant decrease in microtensile bond strength of the two-step but not of the three-step etch and rinse adhesives, Indirect exposure to water did not significantly reduce the microtensile bond strength of any adhesive, indicating that resin bonded to enamel protected the resin-dentin bond against degradation, implying that in the clinical situation, one can rely on durable dentin bonding using three- or two- step etch and rinse adhesives if all cavity margins are located in enamel. For cavities with margins ending in dentin, three step total etch adhesives are preferred.

Another widely used aging technique is thermocycling. Thermocycling was introduced in order to simulate the impact of different temperatures on stability of dental materials. Intraoral temperature changes may be induced by routine eating, drinking and breathing, and concluded that 10,000 cycles corresponded approximately to 1 year of *in vivo* functioning (82), rendering 500 cycles, as proposed by the ISO standard, as being very minimal in mimicking long-term bonding effectiveness. The artificial aging effect induced by thermocycling can occur in two ways: (1) hot water may accelerate hydrolysis of interface components, and subsequent uptake of water and extraction of breakdown products or poorly polymerized resin oligomers occur(75), (2) due to the higher thermal contraction/expansion coefficient of the restorative material (as compared with that of tooth tissue) repetitive contraction and expansion stresses are generated at the tooth biomaterial interface.(82)

A recent meta-analysis(26), concerning data published between 1992 and 1996, concluded that thermocycling had no significant effect on bond strength. Most studies included in the meta-analysis were carried out following the ISO standard of 500 cycles (mean number of cycles in the studies analyzed was 630) in water between 5 and 55°C. This number of cycles was probably too low to obtain an aging effect.(26, 67, 82) In most studies of this review, relatively large resin composite cylinders bonded to flat surfaces were thermocycled, prior to being pulled apart following a shear or tensile bond strength test protocol. (26) As a result, a large part of the interface must have been thermally protected by surrounding dentin and resin composite (which are known to be good thermal insulators). Because of the low c-factor of a flat restored surface, little repetitive expansion/ contraction stress might have been generated at the interface.(4) Both reasons might explain why thermocycling did not affect bonding effectiveness in those studies.(83)

1.2.6 Crosshead speed

A lower rate of load application is often employed with brittle materials compared to elastic materials. Although dentin and resin composites are brittle materials, the rate of load application (crosshead speed) used to evaluate dentin bond strengths can vary significantly.(84) Bond-strength test also incorporates the modulus of adhesive area consisting of a bonding agent, a resin–dentin interdiffusion zone and dentin.(84, 85) Decreasing modulus of elasticity results in a more even stress distribution over the adhesive area, so as to become less concentrated at the point of load application. The rate of load application might be another influential factor that affects the results of bond-strength tests.(84) It has been reported that different crosshead speeds could influence the dentin bond strength.(86, 87) Moreover, relatively high crosshead speeds might cause abnormal stress distributions during the bond strength test, which would influence the bond strength value.(88) According to a review by Oshida and Miyazaki(86), crosshead speeds varied widely ranging from 0.1 to 10.0 mm/min, and crosshead speeds of 0.5, 1.0, 2.0 and 5.0 mm/min were commonly employed to evaluate dentin bond strengths for both tensile and shear modes, although none of the reports mentioned their rationale for selecting these crosshead speeds. Although the ISO Standard has recommended that in bond strength tests to tooth structure, the load should be applied with a crosshead speed of 0.75 ± 0.3 mm/min. Reis et al.(89), and Poitevin et al. (90) found no significant difference in microtensile bond strength if nontrimmed, square specimen were tested with a cross head speed of 0.01 mm/min, 0.1 mm/min and 1 mm/min. Similarly, Yamaguchi et al.(84) found no significant difference when same adhesive system with the different crosshead speeds of 0.5, 1.0, 5.0 and 10.0 mm/min were tested. However, the lower the speed, the more differences were recorded between “stress at maximum load” and “stress at break”. A more uniform stress-time pattern was seen for 1mm/min. Therefore, a crosshead speed of 1mm/min was suggested.(90)

1.2.7 Specimen designs

Microtensile bond strength studies utilize various shapes, including rectangular stick, dumbbell, and hourglass (Figure 7). The cylinder-shaped specimen has been shown to have a better stress distribution than a rectangular hourglass specimen.(28) SEM analysis revealed that trimmed specimens, especially from enamel, often exhibited lines of fracture in the area of action of the bur.(91) Trimming the micro-specimen was very technique sensitive (25, 92), and it induced additional stress, especially in weaker bonds, thus facilitating pre-test failures.(41)

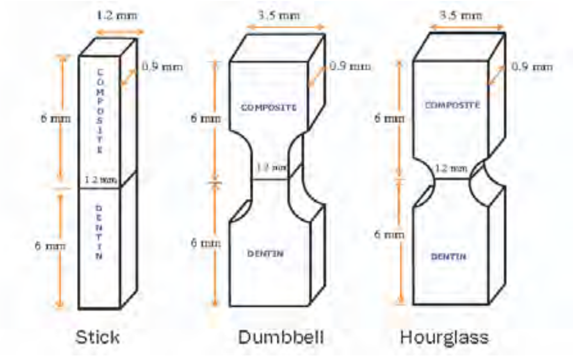


Figure 7. Various shapes of specimen for microtensile test (93)

Phrukkanon et al. (28) concluded that the cross-sectional shape had little effect on μTBS , but in their study both circular and rectangular specimens were trimmed (Figure 8). Direct comparison has been conducted between the bond strengths yielded by trimmed and untrimmed microtensile specimens, there were, in the literature, indications that a higher percentage of premature failures, and accordingly lower values of yielded bond strength, were associated with the trimming modalities.(41) Additionally, as regards to specimen thickness, suggestions have been given that for adequate testing the cross-sectional area should not exceed 1.5 mm^2 (92) and not be lower than 0.5 mm^2 (94), but these guidelines only applied to hourglass shaped specimens. No similar indications have so far been provided for untrimmed specimens.

Figure 8. Circular and rectangular specimens(28)

Naeima et al., 2007 (93) found that the three different specimen designs shown in Figure 7 had no influence on bond strength to dentin within one adhesive system (Figure 9).

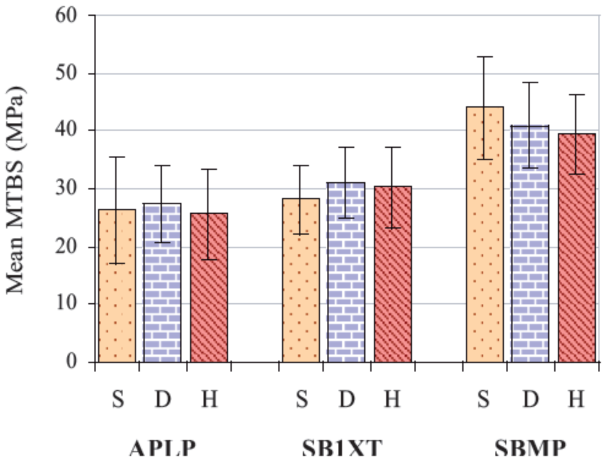


Figure 9. Mean μTBS of Adper Promp L-Pop® (APLP), Adper Scotchbond 1XT® (SB1XT) and Adper Scotchbond Multipurpose Plus ® (SBMP) (S=stick, D = dumbbell, H = hourglass) (93)

Published studies showed a large variation in the hourglass designs, with different degrees of the curvature of the notch (as shown in Figure 10). The specimens were prepared with different outlines, which led to different bond strength values.(95) A study conducted to determine whether differences in the μTBS existed between a range of hourglass designs - circular, parabolic and spline(95) (Figure 11). Result of this study revealed that the μTBS was highly dependent on specimen design at the adhesive joint. The mean μTBS for circular hourglass was significantly higher than those of the other two designs (Figure 11). The circular hourglass design was a more predictable design compared with the others.

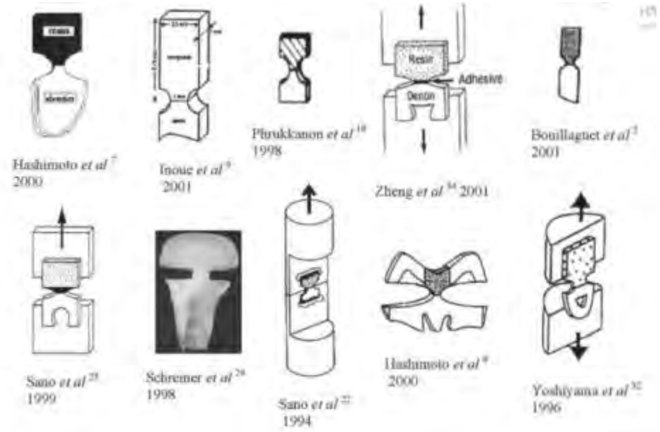


Figure 10. Variation of hourglass specimen designs. (95)

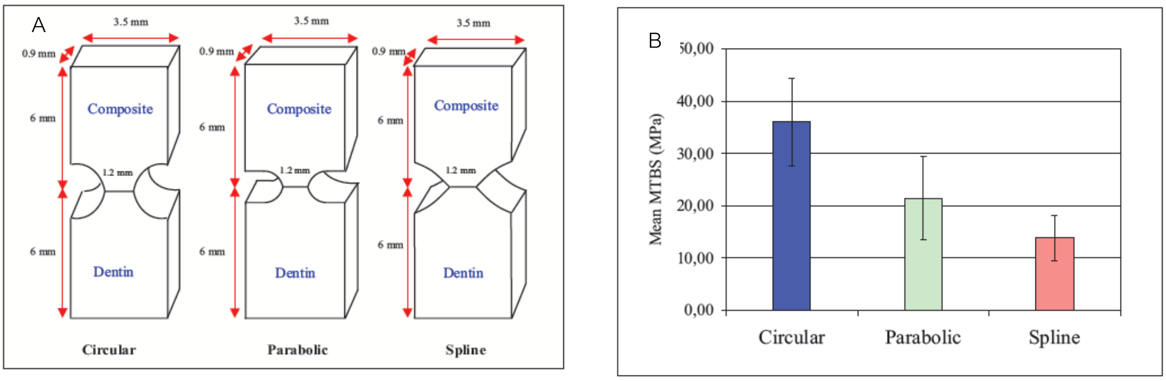


Figure 11. Shapes and mean µTBS of the circular, parabolic and spline hourglass specimen design

A. Shapes of the circular, parabolic and spline hourglass specimen design.

B. Mean µTBS for circular, parabolic and spline hourglass specimen design.(95)

1.3. Analysis of microtensile bond strengths using Weibull analysis.

Currently, many data of presenting microtensile bond strength are reported in term of mean values with standard deviations calculated by assuming normal data distribution, however bond strength outcomes show wide variations.(79) Therefore, the evaluation of good or poor bond strength requires a method that might be a better predictor for clinical performance of restorative materials.(96) Dental resin composite exhibited susceptible to brittle fracture and thus have poorly defined ultimate tensile bond strengths. For brittle materials, the maximum strength, that a sample can withstand, varies unpredictably from specimen to specimen. Hence, the Weibull distribution well suited for brittle materials evaluation. Weibull analysis is a sound method to evaluate bond strength of resin composite to enamel or dentin(96), as recommended by ISO/TS 11405.(21)

Two Weibull parameters are generally shown for analyze bond strength of resin composite to enamel or dentin.(97) 1) Weibull modulus (m) is a measure of variability of the microtensile bond strength. A low Weibull modulus reflects a high variation in measured strengths and greater inconsistency of strength. 2) The characteristic strength is a measure of stress at 63% of the specimens have failed. The advantage of Weibull analysis is the ability to predict survival propability (Ps) and failure propability (Pf) at any microtensile bond strength. A more relevant clinical approximation for the risk level of bond failure can be made. The reliability of adhesives can be evaluated clinically using criteria from the American Dental Association, which stated that failure rate of class V restorations should be no more than 5% after 6 months for “provisional acceptance”, and 10% after 18 months for “full acceptance”.(98) However, minimum bond strength value for *in vitro* conditions has never been defined. Therefore, the stress levels which cause the first 5% and 10% of the specimens to fail were considered more clinically relevant to evaluate the reliability of the bond, rather than using the average strength values.(99)

Polymerization of silorane-based resin composite and methacrylate-based resin composite

Most resin composite restorations placed today involve the use of dentin/enamel adhesive and light cured restorative material.(100, 101) The main problem faced by clinicians when restoring cavities with resin composites is how to deal with the marginal quality of the restoration. The most relevant factors related to this are polymerization shrinkage, adhesion to the cavity walls, viscosity and stiffness of resin composite, and flexibility of the cavity walls.(4, 102) Despite many improvements in resin composite materials, polymerization shrinkage remains a major problem. Polymerization shrinkage stress values vary according to the ratio of bonded to unbonded (free) surface area of the resin composite in a cavity, the configuration or c-factor(4) An increased c-factor leads to a decreased flow capacity which causes a higher rate of shrinkage stress development.(2, 4) The less the restoration is bonded to opposing walls, the less shrinkage interference there will be. If two class I cavities have the same volume but a different design, the deeper and narrower cavity has a higher c-factor than the shallower and larger one.(2) Polymerization shrinkage of resin composite creates contraction stress that can disrupt bond to preparation walls and margins.(103) Competition between stress within polymerization resin composites and adhesion to preparation walls is one of the main causes of marginal failure and subsequent microleakage.(66)

Polymerization shrinkage is a complex process depending on several factors. These can be separated into material formulation factors (filler content, monomer chemistry and structure, filler/ matrix interactions, additives, etc.) and material polymerization factors (polymerization rate, i.e. catalyst and inhibitor concentration, external constraint conditions, cavity geometry, curing method, placement technique, etc.).(104) Recently, several attempts have been made to reduce shrinkage by changing nature of resin. New monomers with low volumetric shrinkage have been prepared recently. Silorane-containing resins are being developed. The term “silorane” was introduced to represent hybrid monomer systems that contained both siloxane and oxirane structural moieties.(105) Weinmann et al. (106) described the synthesis of a new monomer system, silorane, obtained from the reaction of oxirane and siloxane molecules. The novel resin is considered to have combined the two key advantages of the individual components: low polymerization shrinkage due to the ring-opening oxirane monomer and increased hydrophobicity due to the presence of the siloxane species.

Siloranes (Figure 12) are silicon-based monomers with oxirane (epoxide) functionality.(107) Compounds containing oxirane groups are known to be reactive with water.(107) However, like some other silicon-containing monomers, siloranes could be extremely hydrophobic, perhaps making the oxirane groups inaccessible to be attacked by water or water-soluble species.

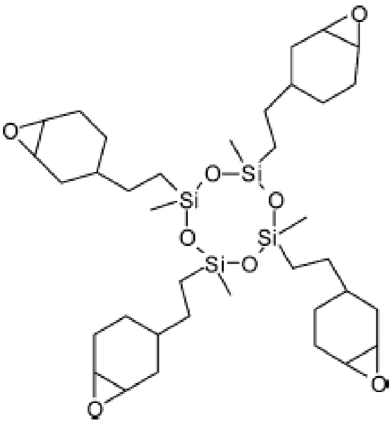


Figure 12. Silorane monomer (106)

Filler of silorane-based resin composite is fine particular quartz of below 0.5 μm (Figure 13). As it is known for methacrylates, silane layer acts as interface between filler and resin facilitating reinforcement of resin with the hard filler particles. At the same time, silane layer increases the hydrophobic character of the surface of the filler. A very important function of silane layer (Figure 14) in silorane technology is to prevent an attack of the acidic Si–OH groups of the quartz, potentially resulting in undesired initiation of the cationic polymerization process.(106)

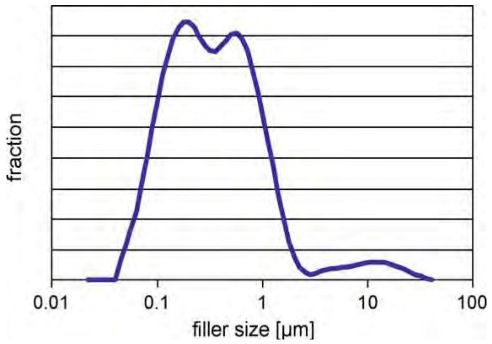


Figure 13. Filler size distribution of silorane-based resincomposite(106)

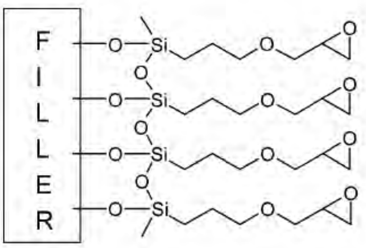


Figure 14. Silane layer for silorane-based resin composite (106)

The network of siloranes is generated by the cationic ring opening polymerization of the cycloaliphatic oxirane moieties, which stand for their low shrinkage and low polymerization stress.(106, 108) The most important difference is that methacrylates are cured by radical intermediates, and oxiranes polymerize via cationic intermediates (Figure 15).

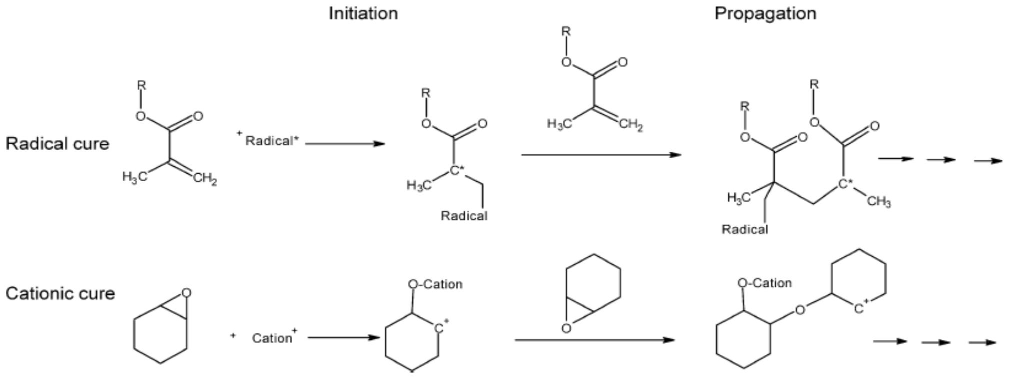


Figure 15. Polymerization of radical curing methacrylates and cationic curing ring opening epoxies. (106)

Generation of radical species for methacrylate cure is realized by a two component system consisting of camphorquinone, which is the actual photoinitiator, and a tertiary amine (Figure 16 A), responsible for hydrogen transfer reaction. This system decomposes immediately by exposure to light with a wavelength between 430 and 490 nm, and generates radical species to start polymerization process. Development of a photoactivated silorane-based resincomposite was realized with a three component initiating system comprising camphorquinone, an iodonium salt, and an electron donor.(Figure 16 B) In this reaction path, the electron donor acts in a redox process and decomposes the iodonium salt to an acidic cation, which starts the ring opening polymerization process. (106) However, like the methacrylate-based resin composites, silorane-based resin composite also contains camphorquinone, so that currently available dental curing units can be used for polymerization initiation.(17)

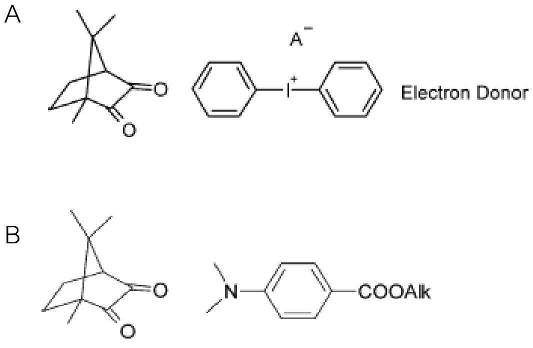


Figure 16. Photoinitiating system

A Photoinitiating system for radical cure (106)

B Photoinitiating system for cationic cure (106)

Ilie and Hickel(18) showed that in terms of degree of cure and mechanical properties at 2-mm depth, no statistically significant differences were recorded between curing units MiniLED® and Bluephase® for the same curing time. However, it was observed in a study that a higher irradiance also caused higher shrinkage stress rate. In the case of the methacrylate-based resin composite, the shrinkage stress curve gradually increased after photoinitiation.(17) However, this was not for the silorane-based resin composite, whereby shrinkage stress development soon plateaued to a constant after photoinitiation (Figure 17), indicating that no additional shrinkage occurred after photoinitiation. Investigations on development of degree of conversion in silorane material with time revealed that degree of cure continued to increase even at 20 minutes after photoinitiation.(17) This meant that silorane rings continued to be opened with time, and were able to subtend shrinkage caused by formation of the three-dimensional polymer network.

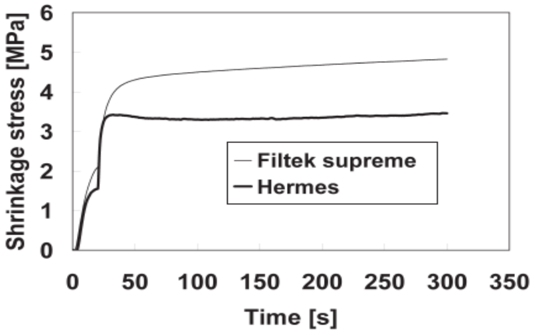


Figure 17. Comparison of shrinkage stress development as a function of time for silorane-based material (Hermes®, 3M ESPE) and a regular methacrylate-based material (Filtek supreme®, 3M ESPE) cure for 20 seconds with the LED curing unit, Bluephase®.(17)

The ring-opening polymerization of the silorane molecule is cationic polymerization reaction where no oxygen inhibition layer exists on the surface of resin composite after polymerization in air.(109) Oxygen inhibition layers of free radical polymerized dimethacrylate resins are known to allow good interfacial adhesion of the resin layers.(107) Silorane resin adhesive is based on methacrylate chemistry with phosphate groups. Possible reaction of the phosphate group with oxirane and the acrylate group with dimethacrylate might be the reason for increased bond strength obtained. Suggested chemical reaction is shown in Figure 18

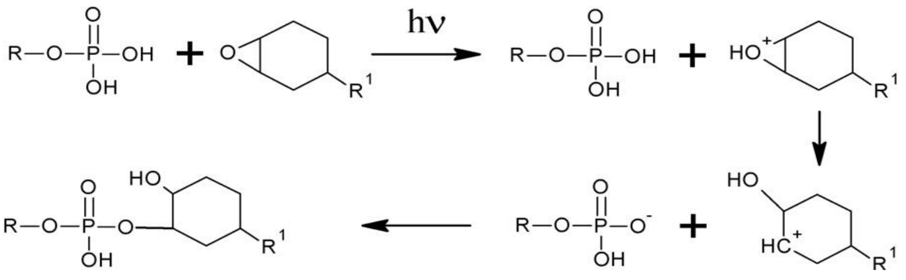


Figure 18. Suggested formation of chemical bond between Silorane System Adhesive Bond organophosphate group) and Filtek Silorane (oxirane group). (109)

Previous studies showed almost 50% lower shrinkage stress for the silorane-based resin composite compared to dimethacrylate-based resin composites.(17) This was attributed to the differences in free radical or cationic-based polymerization reaction kinetics. With most *in vitro* test set up, the c-factor of flat substrate surface is low, resulting possibly in a low shrinkage stress on the adhesive bonds. However, at the clinical settings, with a high c-factor, the low shrinkage stress of silorane-based resin composite could be an advantage(5, 110), and the difference found in bond strengths might be partly compensated due to the low shrinkage stress implemented by silorane-based resin composite.

Another clinically interesting point to consider with the use of cationic polymerization systems is how to achieve a successful of cationic polymerization reaction in a moist environment such as oral cavity. The cationic initiators are generally prone to inactivation in the presence of water, which might affect the bonding and mechanical properties in the oral cavity. The presence of water can disrupt polymerization since its nucleophilicity allows it to compete with monomer for the oxonium ions. However, this issue was not addressed in the current study(109) and was suggested for the further investigation.

CHAPTER III

MATERIALS AND METHODS

Materials and methods

Materials

1. Extracted human third molars, free of caries, cracks or other defects
2. Resin composites
    2.1. Filtek P90® (Shade A2) (3M ESPE, St. Paul, MN, USA)
    2.2. Filtek Z250® (Shade A2) (3M ESPE, St. Paul, MN, USA)
3. Adhesives
    3.1. Silorane system® adhesive(3M ESPE, St. Paul, MN, USA)
    3.2. Adper Scotchbond Multipurpose® adhesive (3M ESPE, St. Paul, MN, USA)
4. 0.5% Chloramine solution (Merck, Darmstadt, Germany)
5. Distilled water (Dental Material Science Research Center, Chulalongkorn University)
6. Cylindrical diamond bur (Crosstech Diamond bur, BKK, Thailand, ISO 021)
7. Pear shape diamond bur (Crosstech Diamond bur, BKK ,Thailand, ISO 032)
8. Taper diamond bur (Crosstech Diamond bur, BKK, Thailand, ISO 021)
9. Small round diamond bur (Crosstech Diamond bur, BKK, Thailand, ISO 007)
10. Microbrush (3M ESPE, St. Paul, MN, USA)
11. Cyanoacrylate (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan)
12. Velmix® stone (Kerr, Orange, CA, USA)

Equipments

1. Mobile unit (Kavo, Heidelberg, Germany)
2. Light curing unit (Demi™ LED light curing system, Kerr, Orange, CA, USA)
3. Digital caliper (Mitutoyo, Tokyo, Japan)
4. Incubator (Contherm 160M, Contherm Scientific Ltd., Petone, New Zealand)
5. Slow speed model trimmer (MT-7, J Morita Tokyo Mfg. Corp.,Tokyo, Japan)
6. Slow speed cutting machine (Accutom-5, Struers, Copenhagen, Denmark)
7. Universal testing machine (Instron, Canton, MA, USA)
8. Stereomicroscope (ML9300,Meiji Techno Co. Ltd., Tokyo, Japan)
9. Scanning electron microscope (JEOL JSM-5410LV, Tokyo,Japan)
10. Radiometer (Kerr, Orange, CA, USA)
11. Dental loupes with 2.5X magnification (Orascoptic, Kerr, Orange, CA, USA)

Sample size determination (n)

For this study, sample size determination based on a pilot study has been performed for a test of difference in 2 independent means with specified absolute precision as below

$$n = \frac{2\sigma^2 (Z_{1-\alpha/2} + Z_{1-\beta})^2}{(\mu_1 - \mu_2)^2} \qquad \sigma^2 = S_p^2 = \frac{(S_1^2 + S_2^2)}{2}$$

n = sample size per group

σ^2 = population variance

S = sample standard deviation

α = probability of type I error

β = probability of type II error

$\mu_1 - \mu_2$ = difference in mean between 2 groups

Where $\alpha = 0.05$ (2-sided), $Z_{1-\alpha/2} = 1.96$

$\beta = 0.05$, $Z_{1-\beta} = 1.96$

This study expected the mean difference between groups at 95% statistic power, and *p* value at 0.05. Sample sizes were calculated to detect the mean difference between two groups by performing estimation in the sample size from results of pilot study (5 samples per each group in pilot study), as Table 2.

Table 2. Means and Standard Deviations (SD) of microtensile bond strength to dentin from pilot study at 24 hour storage time.

Between group	μ_1	S_1	μ_2	S_2	σ^2	**n**
PHC&PLC	8.334	2.85809	19.32	3.769217	11.18784	2.848847
PHC&ZHC	8.334	2.85809	24.72	4.819959	15.70034	1.797071
PHC&ZLC	8.334	2.85809	51.02	10.43226	58.50034	0.98671
PLC&ZHC	***19.32***	***3.769217***	***24.72***	***4.819959***	***18.7195***	***19.72917****
PLC&ZLC	19.32	3.769217	51.02	10.43226	61.5195	1.881466
ZHC&ZLC	24.72	4.819959	51.02	10.43226	66.032	2.933898

Abbreviations
PHC = Filtek P90® + high c-factor,
PLC = Filtek P90® + low c-factor,
ZHC = Filtek Z250® + high c-factor,
ZLC = Filtek Z250® + low c-factor
μ_1 = Mean of first group sample
S_1 = Standard deviation of first group sample
μ_2 = Mean of second group sample
S_2 = Standard deviation of second group sample
σ^2 = population variance
n = sample size per group

Pilot study to determine sample size for microtensile testing showed that at least 20 samples would be required to satisfy the constraints of α=0.05, power, 0.95.

Tooth selection

One hundred and sixty non-carious, non restored human third molars (collected following informed consent approved by the Ethical Review Board of the Faculty of Dentistry, Chulalongkorn university, Bangkok, Thailand, with approval number 7/2009), stored in 0.5% chloramine for 1 week, before storage in distilled water at 4 °C for no longer than six months.

Sample preparation

One hundred and sixty non-carious, non restored human third molars were cleaned and mounted in dental Velmix® stone, using a mold, to the level of approximately 2.0 mm. below the cementoenamel junction. Long axes of the tooth and the mold were adjusted to be paralleled. After Velmix® stone set, the mounted tooth was removed from the mold and then was trimmed with slow-speed diamond model trimmer (MT-7, J Morita Tokyo Mfg. Corp.,Tokyo, Japan) to prepare flat occlusal dentin surfaces. Mounted teeth were kept in distilled water at 4°C until used for testing. All specimens were randomly divided into eight groups. (2 resin composites x 2 c-factors x 2 aging times) (Figure 19).

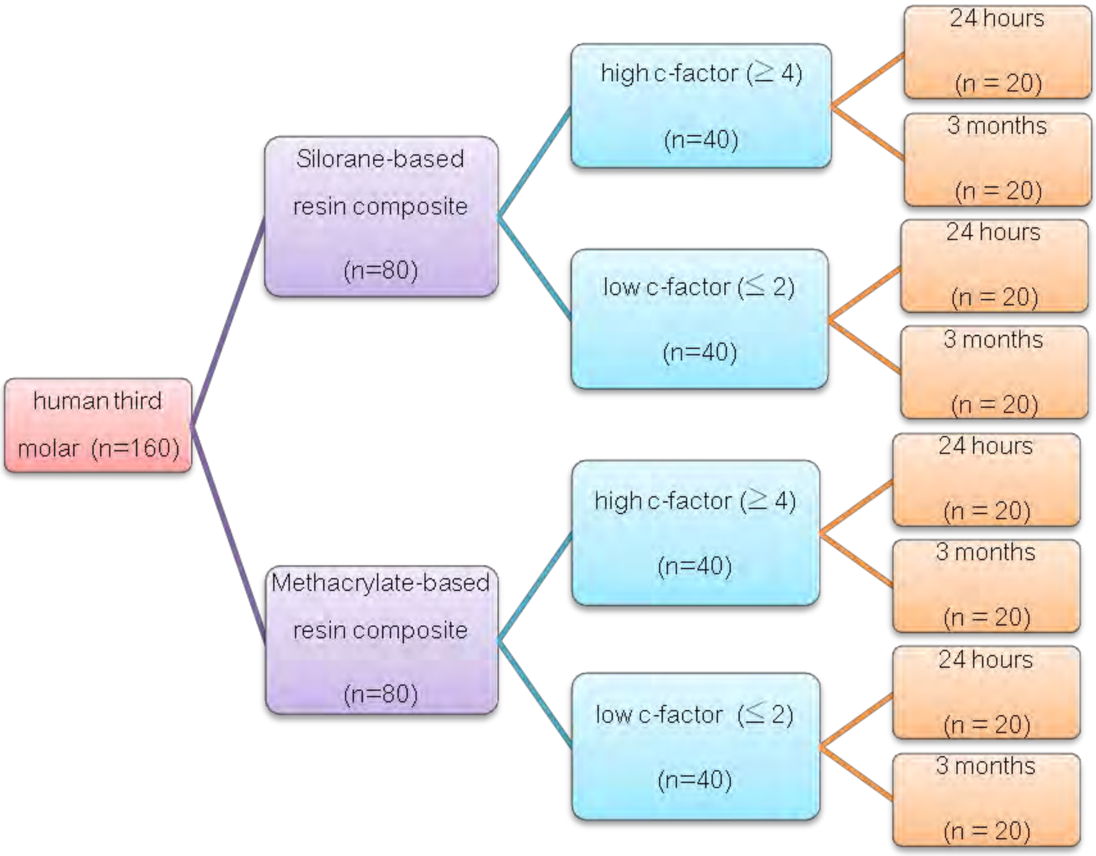


Figure19. Experimental groups in the present study

Cavity preparation (Figures 20A to C)

All specimens were randomly divided into 2 groups in order to prepare class I cavities with 2 c-factor designs. Cavity preparation was performed by one operator using diamond burs. Each bur was replaced after two preparations to maintain sharpness.

Group I: High c-factor group (c-factor $\geq$ 4.0)

The extent of the preparation area was determined using a plastic mold with a square hole (6.0 mm. x 3.0 mm.) to mark an occlusal outline (Figure 20A). The depth guide of preparation was determined using a small round bur, as shown in Figure 20B. Box-shape class I cavity was prepared on occlusal surface using a cylindrical diamond bur (Crosstech, BKK, Thailand) at high speed with water coolant. Cavity dimensions were 6.0 mm long, 3.0 mm. wide and 3.0 mm. deep. The depth of cavity was determined using a periodontal probe.

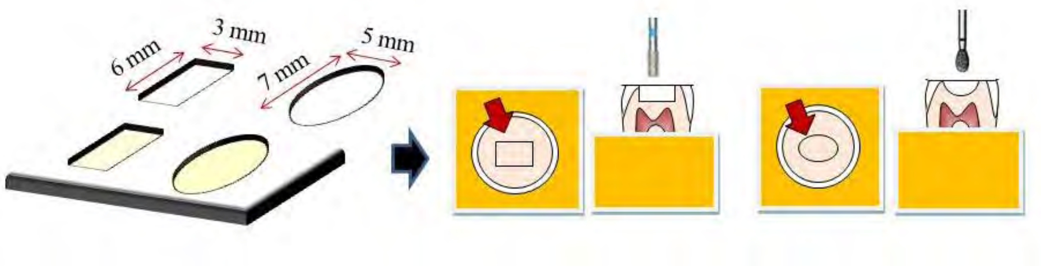
Group II: Low c-factor group (c-factor $\leq$ 2)

The extent of the preparation area was determined, using a plastic mold with ellipsoidal hole (7.0 mm. x 5.0 mm.) to mark an occlusal outline (Figure 20A). The depth guide of preparation was determined, using a small round bur, as shown in Figure 20B. Ellipsoid-shape class I cavity was prepared on occlusal surface using a large-sized pear shape diamond bur (Crosstech, BKK, Thailand) at high speed with water coolant. Cavity dimensions were 7.0 mm. long, 5.0 mm. wide and 3 mm deep. The depth of cavity was determined using a periodontal probe.

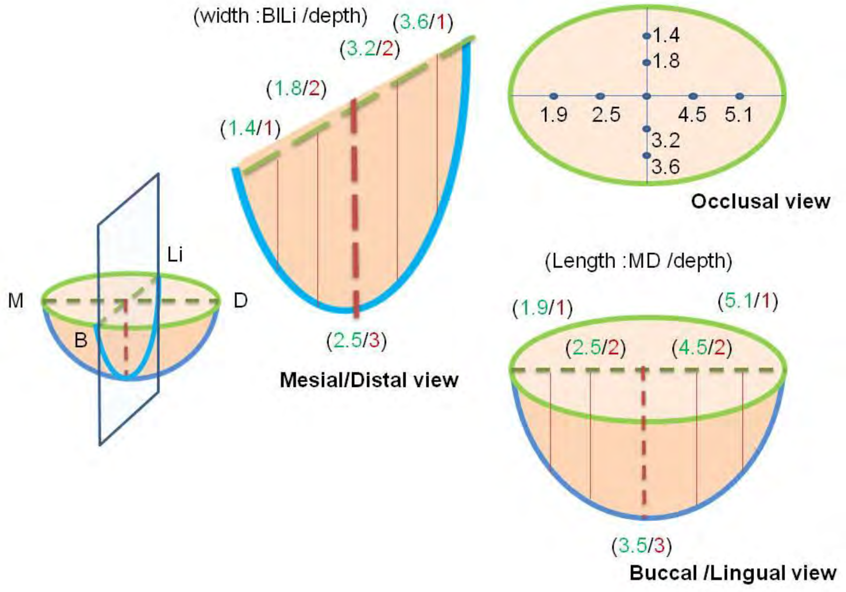
The two cavity preparation designs provided, high and low c-factor cavities, however, the two cavities of different designs contained approximately equal volume of resin composite material. Ellipsoidal shaped class I cavities, prepared with a length of 7.0 mm., a width of 5.0 mm. and a depth of 3.0 mm. was computed to have a volume of 55 mm^3 and a c-factor (bonded area/unbounded area) of 1.4. Box shaped class I cavities, prepared with a length of 6.0 mm. a width of 3.0 mm. and a depth of 3.0 mm., was computed to have a volume of 54 mm^3 and a c-factor (bonded area/unbounded area) of 4.0 (Figure 21).

Figure 20. Cavity preparation method

A) A Plastic mold used as guide for cavity preparation.



B) Cavity parameter of an ellipsoidal cavity



C) Cavity preparation

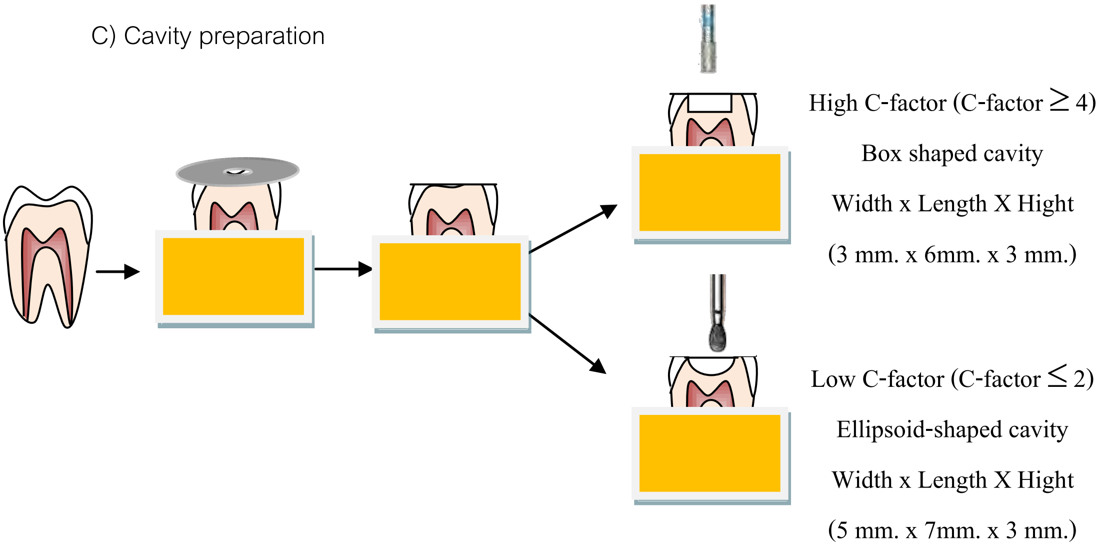
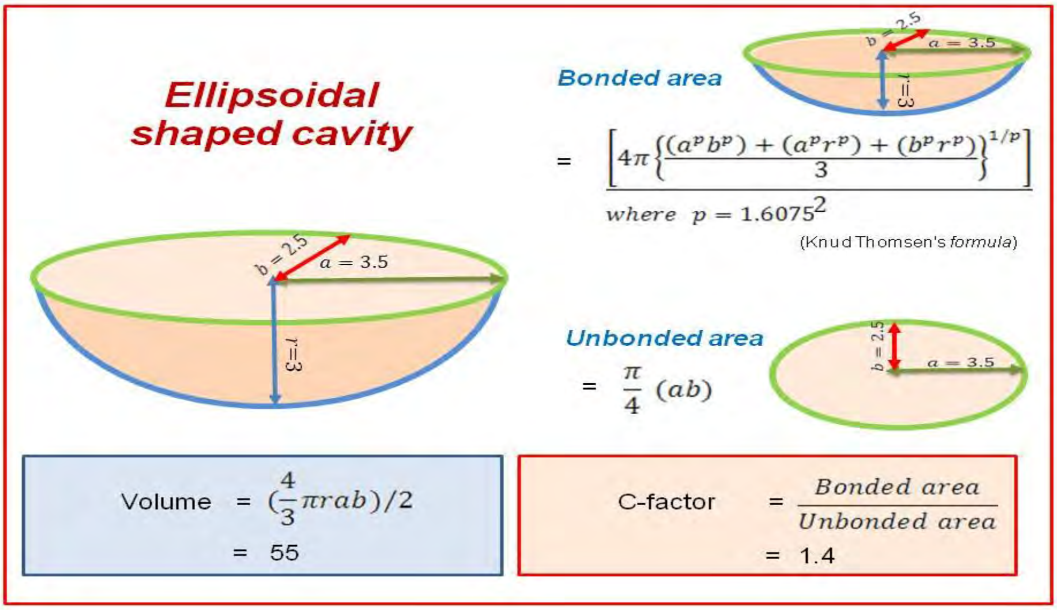


Figure 21. C-factor and volume of cavity

A) Ellipsoidal shaped cavity



Bonded area

$$= \frac{\left[4\pi\left\{\frac{(a^pb^p)+(a^pr^p)+(b^pr^p)}{3}\right\}^{1/p}\right]}{}$$

where $p = 1.6075^{2}$

(Knud Thomsen's *formula*)

Unbonded area

$$= \frac{\pi}{4}(ab)$$

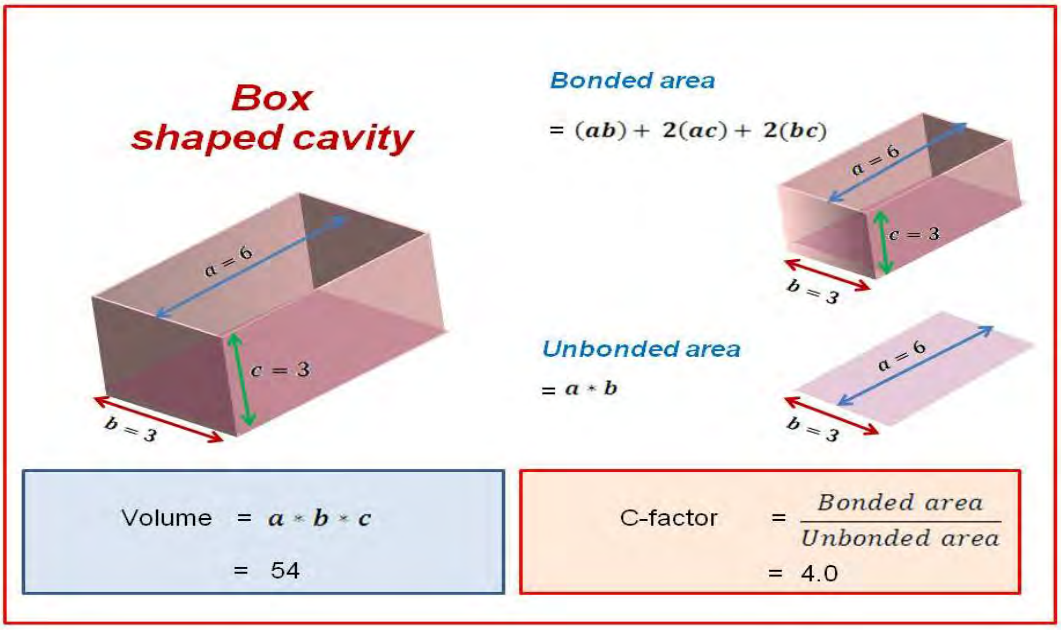
Volume $= (\frac{4}{3}\pi rab)/2$

= 55

C-factor $= \frac{Bonded\ area}{Unbonded\ area}$

= 1.4

B) Box shaped cavity



Bonded area

$= (ab) + 2(ac) + 2(bc)$

Unbonded area

$= a * b$

Volume $= a * b * c$

= 54

C-factor $= \frac{Bonded\ area}{Unbonded\ area}$

= 4.0

Adhesive and restorative procedures

One commercially available methacrylate-based resin composites (Filtek Z250®) and one novel silorane-based resin composite (Filtek P90®) were assessed in this study. Filtek P90® with its Silorane system® adhesive (3M ESPE, St Paul, MN, USA) were used in half of each c-factor group, and the other half was restored using Filtek Z250® and Scotchbond Multipurpose® (3M ESPE, St Paul, MN, USA) (Figure 19). Light-curing was performed using a LED curing light (Demi® LED light curing system, Kerr, Orange, CA, USA). The irradiance at the tip of the curing light was 1,100–1,300 mW/cm^2 as measured by a radiometer (Kerr, Orange, CA, USA). Placement of the restorative resin composite was performed incrementally. Resin composite was placed in two horizontal consecutive increments. The first increment of resin composite was inserted 2 mm. thick horizontally, and the second increment on the upper part of cavity (Figure 22). Each increment was light activated for 40 seconds. The specimens were stored at 37°C in distilled water either for 24 hours or for 3 months prior to bond tests. Resin composites and adhesives composition and application modes are listed in Tables 3 and 4.

Table 3. Adhesives investigated in this study

Adhesive	Manufacture	Component	Ingredients	Application
Scotchbond Multipurpose® (3-step total etch adhesive)	3M ESPE, St Paul, MN, USA	Etchant (Batch:3007)	- 35% Phosphoric acid - Water - Silica thickener	1. Apply on tooth surface for 15s. 2. Rinse for 15 s. 3. Gently air blow for 2 s.
		Primer (Batch:3008)	- HEMA (30-40%) - Polyalkenoic acid copolymer - Water	1. Apply on tooth surface and brush for 15 s 2. Gently air blow (Evaporate the volatile ingredients with a mild air stream) for 5 s.
		Adhesive (Batch:3009)	- Bis-GMA (60-70%) , HEMA (30-40%) and CQ	1. Apply on tooth surface 2. Light cure for 20 s.
Silorane system® adhesive (2-step self etch adhesive)	3M ESPE, St Paul, MN, USA	Self etch primer (Batch: 4763P)	- Bis-GMA (15-25%) - HEMA (15-25%) - Water (10-15%) - Ethanol (10-15%) - Silane treated silica (8-12%) - Phosphoric acid-methacryloxy-hexylesters (5-15%) - 1, 6-Hexanediol dimethacrylate (5-10%) - Copolymer of acrylic and itaconic Acid (<5%) - (Dimethylamino) ethyl methacrylate (<5%) - CQ (<3%) - Phosphine oxide (<3%)	1. Apply on tooth surface and brush for 15 s 2. Gently air blow (Evaporate the volatile ingredients with a mild air stream) for 5 s. 3. Light cure for 10 s.
		Adhesive (Batch 4763B)	- Substituted dimethacrylate (70-80%) - Silane treated silica (5-10%) - TEGDMA (5-10%) - Phosphoric acid methacryloxy-hexylesters (<3%) - CQ (<3%) - 1, 6-Hexanediol dimethacrylate (<3%)	1. Apply on tooth surface 2. Light cure for 20 s.

Table 4. Resin composites investigated in this study (111)

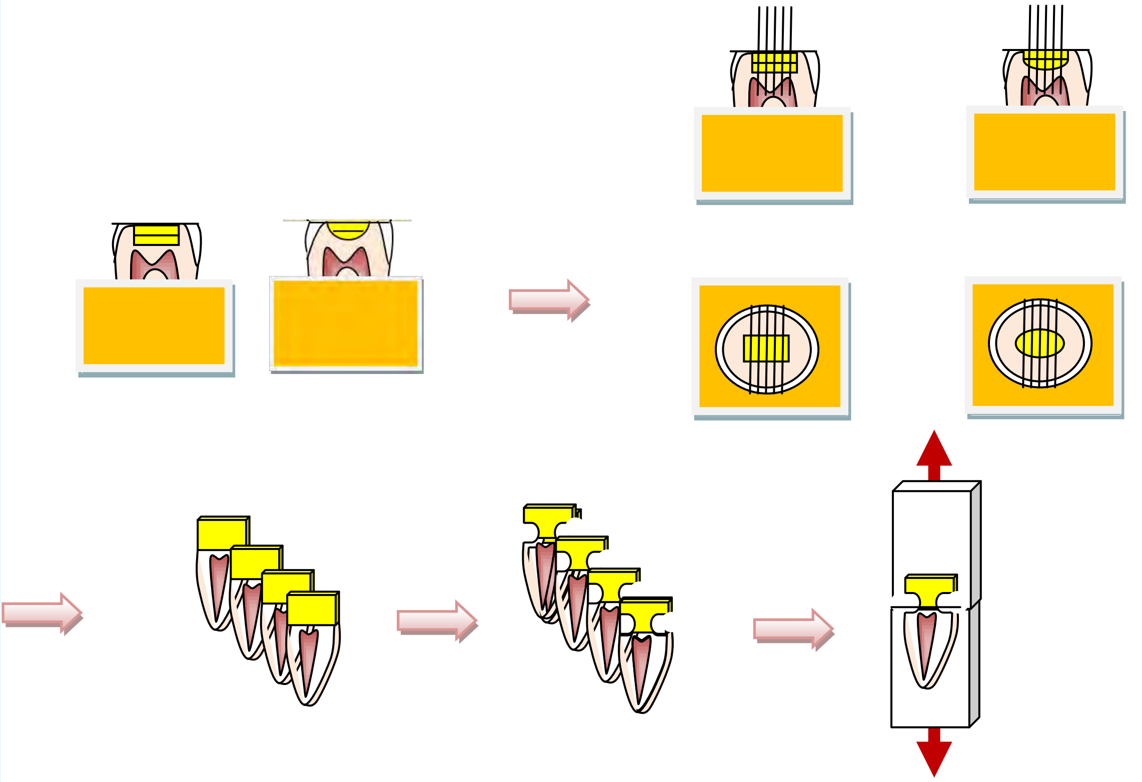
Resin Composite	Manufacture	Type	Filler	Resin matrix
Filtek P90®	3M ESPE, St Paul, MN, USA	Microhybrid (0.04-1.7µm)	Silicon dioxide, ytterbium trifluoride (76% by weight / 55% by volume)	Silorane
Filtek Z250®	3M ESPE, St Paul, MN, USA	Microhybrid (0.01-3.5 µm)	Zirconium glass and colloidal silica (78% by weight / 60% by volume)	Bis-GMA, Bis-EMA, UDMA and TEGDMA

Abbreviations Bis-GMA = bisphenol-A-glycidyl methacrylate
Bis-EMA = bisphenol-A-ethoxylate glycidyl methacrylate
TEGDMA = Triethyleneglycol methacrylate
HEMA = 2-Hydroxyethyl methacrylate
CQ = camphorquinone

Microtensile bond strength test (Figure 22)

The restored cavities were sectioned serially perpendicular to the adhesive–tooth interface using an Isomet diamond saw (Accutom-5, Struers, Copenhagen, Denmark) to create 3 or 5 slabs of 0.8 mm. thick. The specimens were trimmed into an hourglass shape with a narrowest portion of 1 ± 0.25 mm. wide located at the adhesive dentin interface, using a taper diamond bur (Crosstech Diamond bur, BKK, Thailand) with copious air-water spray. Each sample was examined for flaws using dental loupes with a 2.5X magnification (Orascoptic, Kerr, Orange, CA, USA). The exclusion criteria included the presence of any obvious flaw, or specimen debonding before testing. The procedure created a total of 20 hourglass specimens per group. 20 specimens for each group were attached to the test apparatus with a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan), and were stressed to failure in tension using a universal testing machine (Instron, Canton, MA, USA) at a cross-head speed of 1 mm/min. The maximum load to debond the specimens was recorded in newtons. Microtensile bond strength was calculated by the ratio of maximum load to the cross-sectional area of the bonded interface. The μTBS values (MPa) at breaking point were recorded automatically by the testing machine. After testing, adhesive area of each sample was measured using a digital caliper (Mitutoyo, Tokyo, Japan). The μTBS values were determined from the specimens that survived specimen processing, with an explicit note of the number of pre-testing failures.

Figure 22. Sample preparation for microtensile bond strength test



Evaluation of fracture mode

The fractured surfaces of both resin and dentin were examined under a stereomicroscope (ML9300 MEIJI, Japan) at a 40x magnification to determine the mode of failure. Some of specimens were then selected to confirm the mode of failure under a SEM. The actual mode of failure was recorded according to the following criteria (112, 113)

Adhesive failure: No signs of dentin fracture or remnants of resin on the tooth, failure in adhesion.

Mixed failure: A mixture of adhesive and cohesive failure.

Cohesive failure in dentin: Complete fracture of dentin, failure of the tooth substrate.

Cohesive failure in resin: Complete fracture of resin, failure of the resin composite.

Two fractured dentin sides and two fractured resin composite sides from each group were selected for evaluation under a SEM. Both the dentin and resin sides were air-dried and sputter-coated with gold before being examined with a SEM at 2000x magnification.

Data analysis

After the data distribution was tested. The microtensile bond strength data were statistically analyzed using SPSS (version 17) for windows (Chicago, IL, USA). The data were subsequently by analyzed using parametric statistics (three-way ANOVA and Tamhane post hoc test), at 95% confident interval.

1. Microtensile bond strength data between different types of resin composite (silorane-based resin composites and methacrylate-based resin composite) were analyzed using Tamhane post hoc test.
2. Microtensile bond strength data between different c-factor cavities (high c-factor cavities and low c-factor cavities) were analyzed using Tamhane post hoc test.
3. Microtensile bond strength data between different water storage times (24 hours water storage time or 3 months water storage time) were analyzed using Tamhane post hoc test.

Means of the microtensile bond strength values were determined from the specimens that survived specimen processing with an explicit note of the number of pretest failures.

The results reported were obtained by applying linear regression analysis to the appropriate natural logarithmic transforms of the raw data to give Weibull moduli and 95% confidence limits for the values using Excel 2007, in order to test reliability of the method of microtensile bond test and adhesive performance.

CHAPTER IV

RESULTS

This study measured microtensile bond strength (µTBS) values of a methacrylate-based resin composite (Filtek Z250® with Scotchbond Multipurpose®) and a silorane-based resin composite (Filtek P90® with Silorane system® adhesive) in class I cavities of high and low c-factor at 24 hours or 3 months of water storage. The Shapiro-Wilk test indicated a normal distribution of µTBS values in all groups ($p > 0.05$). Three-way ANOVA (Table 5) revealed that factors including types of material, c-factors and water storage times had significant effects to µTBS values, with $p < 0.01$. There was a significant interaction between type of material and c-factor ($p < 0.01$), interaction between type of material and water storage time ($p < 0.01$) and interaction between c-factor and water storage time ($p < 0.01$). There was a significant three-factor interaction between type of material, c-factor and water storage time ($p < 0.05$).

Table 5. Summary of results of the three-way ANOVA

Factors	*p*-value
Type of material	0.000
C-factor	0.000
Water storage time	0.000
Type of material * C-factor	0.000
Type of material * Water storage time	0.000
C-factor * Water storage time	0.000
Type of material * C-factor* Water storage time	0.047

The means of microtensile bond strength to dentin, standard deviations and the percentage of pretest failures are shown in Table 6. At both 24 hours and 3 months of water storage, µTBS values of Filtek Z250® were significant higher than

Filtek P90® for both high and low c-factor cavity. The µTBS values of the Filtek Z250® in high c-factor cavities (ZHC) were 32.63 ± 7.33 MPa and 19.97 ± 4.48 MPa for 24 hours and 3 months of water storage, respectively, while µTBS values of Filtek P90® in the same cavity design (PHC) were 8.55 ± 1.76 MPa and 6.94 ± 2.07 MPa. The µTBS values of the Filtek Z250® in low c-factor cavity (ZLC) were 54.19 ± 9.05 MPa and 29.95 ± 7.88 MPa for 24 hours and 3 months of water storage, respectively, while µTBS values of Filtek P90® in the same cavity design (PLC) were 19.89 ± 2.82 MPa and 13.71 ± 2.60 MPa. The ranking of µTBS values was divided into five levels, as shown by the bar graphs in Figure 23: 1) ZLC24h (54.19 ± 9.05 MPa) 2) ZHC24h (32.63 ± 7.33 MPa) and ZLC3m (29.95 ± 7.88 MPa) 3) ZHC3m (19.97 ± 4.48 MPa) and PLC24h (19.89 ± 2.82 MPa) 4) PLC3m (13.71 ± 2.60 MPa) 5) PHC24h (8.55 ± 1.76 MPa) and PHC3m. (6.94 ± 2.07 MPa). It is noteworthy that the lowest bond strengths for experimental groups that used Filtek Z250® (ZHC3m) were comparable to that of the highest bond strengths of the experimental groups that used Filtek P90® (PLC24h).

Table 6. Microtensile bond strength to dentin, standard deviations and statistical significance after 24 hours and 3 months of water storage

Materials	c-factor	n	Mean ± SD (MPa)		Ptf (% of sum)	
			24 hours	3 months	24 hours	3 months
P90	High	20	8.55 ± 1.76 ^{A}	6.94 ± 2.07 ^{A}	18 (47.37%)	61 (75.31%)
P90	Low	20	19.89 ± 2.82 ^{B}	13.71 ± 2.60 ^{C}	15(42.86%)	17 (45.95%)
Z250	High	20	32.63 ± 7.33 ^{D}	19.97 ± 4.48 ^{B}	12(37.5%)	15(42.86%)
Z250	Low	20	54.19 ± 9.05 ^{E}	29.95 ± 7.88 ^{D}	3(28.57%)	15 (42.86%)

Abbreviations SD = standard deviation, ptf = pre-testing failure, n = total number of specimens.

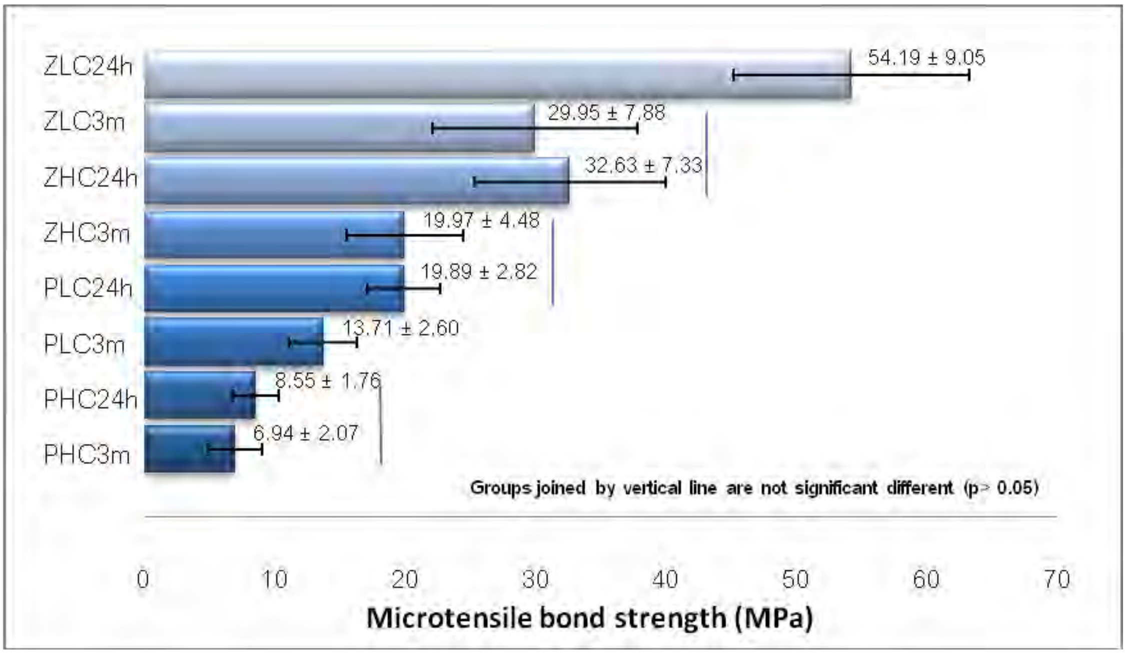
Different letters mean statistically difference (Three – way ANOVA and Tamhane post hoc-tests)

(Pre-testing failures, were not included in the calculation of the mean µTBS)

Regarding the influence of c-factor, µTBS values significantly decreased in high c-factor condition, compared to low c-factor condition in all groups of both at 24 hours and 3 months of water storage. In terms of influence of water storage time, the type of resin composite and c-factor were affected differently. After 3 months of water storage, μTBS values of PLC, ZHC and ZLC groups were significantly reduced ($p < 0.05$) compared to their bond strength at 24 hours of water storage, except μTBS values of PHC group that were reduced by water storage but not statistically significant.

A high number of pretesting failures during sample preparation for microtensile bond strength test was recorded for PHC3m (75.31%), PHC24h (47.37%), PLC3m (45.95%), PLC24h (42.86%), ZHC3m (42.86%), ZHC24h (37.5%), ZLC3m (42.86%) and ZLC24h (28.57%) (Table 6). Highest number of pretest failure in the group that presented the lowest bond strength values (PHC3m) and lowest number of pretest failure in the group that presented the highest bond strength value (ZLC 24h).

Figure 23. Bar graphs showing means and standard deviations of microtensile bond strength (MPa)



Abbreviations.

ZLC24h = Filtek Z250® / low c-factor/ 24 hours

ZLC3m = Filtek Z250® / low c-factor/ 3 months

ZHC24h = Filtek Z250® / high c-factor/ 24 hours

ZHC3m = Filtek Z250® /high c-factor/ 3 months

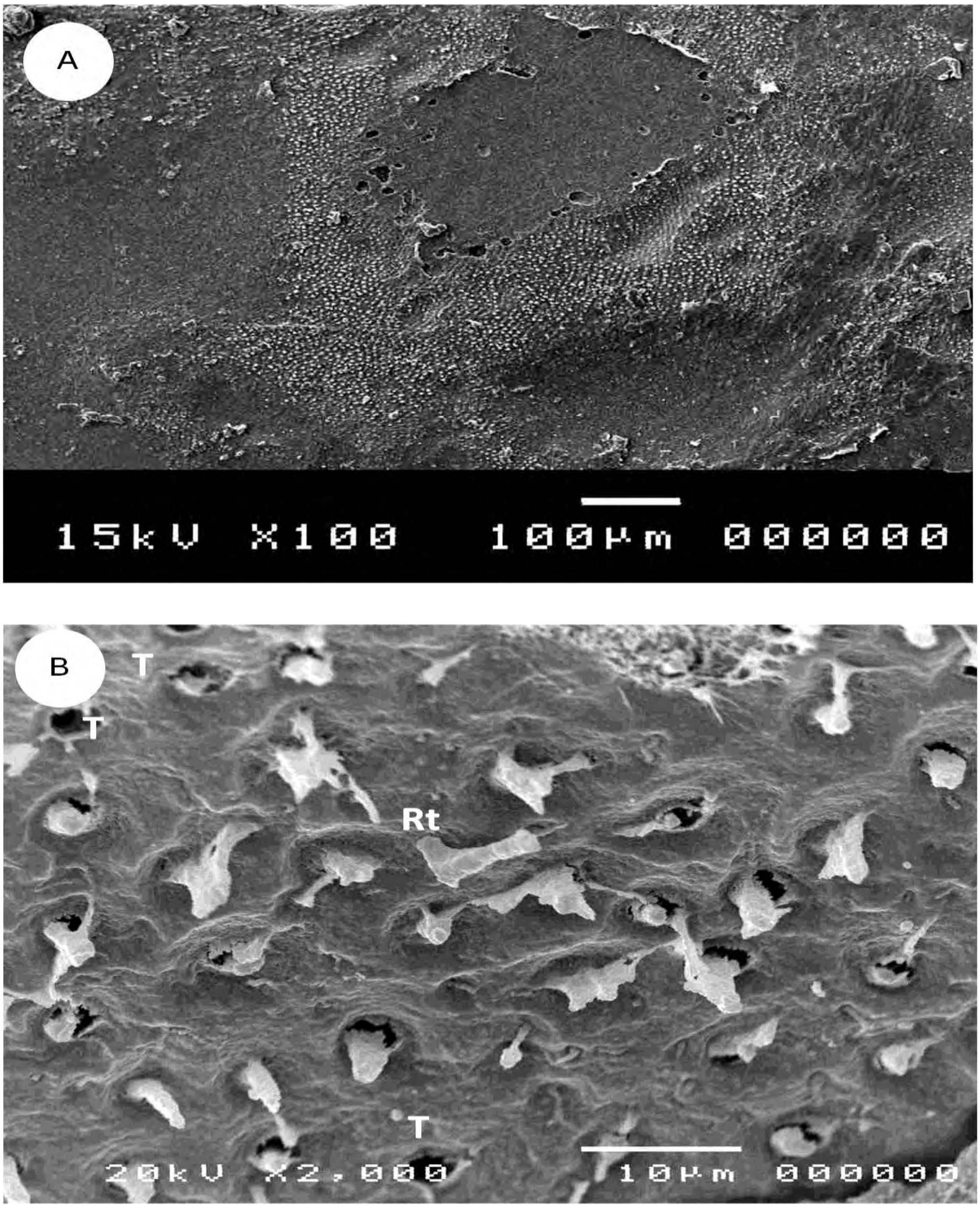
PLC24h = Filtek P90® / low c-factor/ 24 hours

PLC3m = Filtek P90® / low c-factor/ 3 months

PHC24h = Filtek P90® / high c-factor/ 24 hours

PHC3m = Filtek P90® /high c-factor/ 3 months

Figure 24. Scaning electron micrographs of adhesive failed specimens.



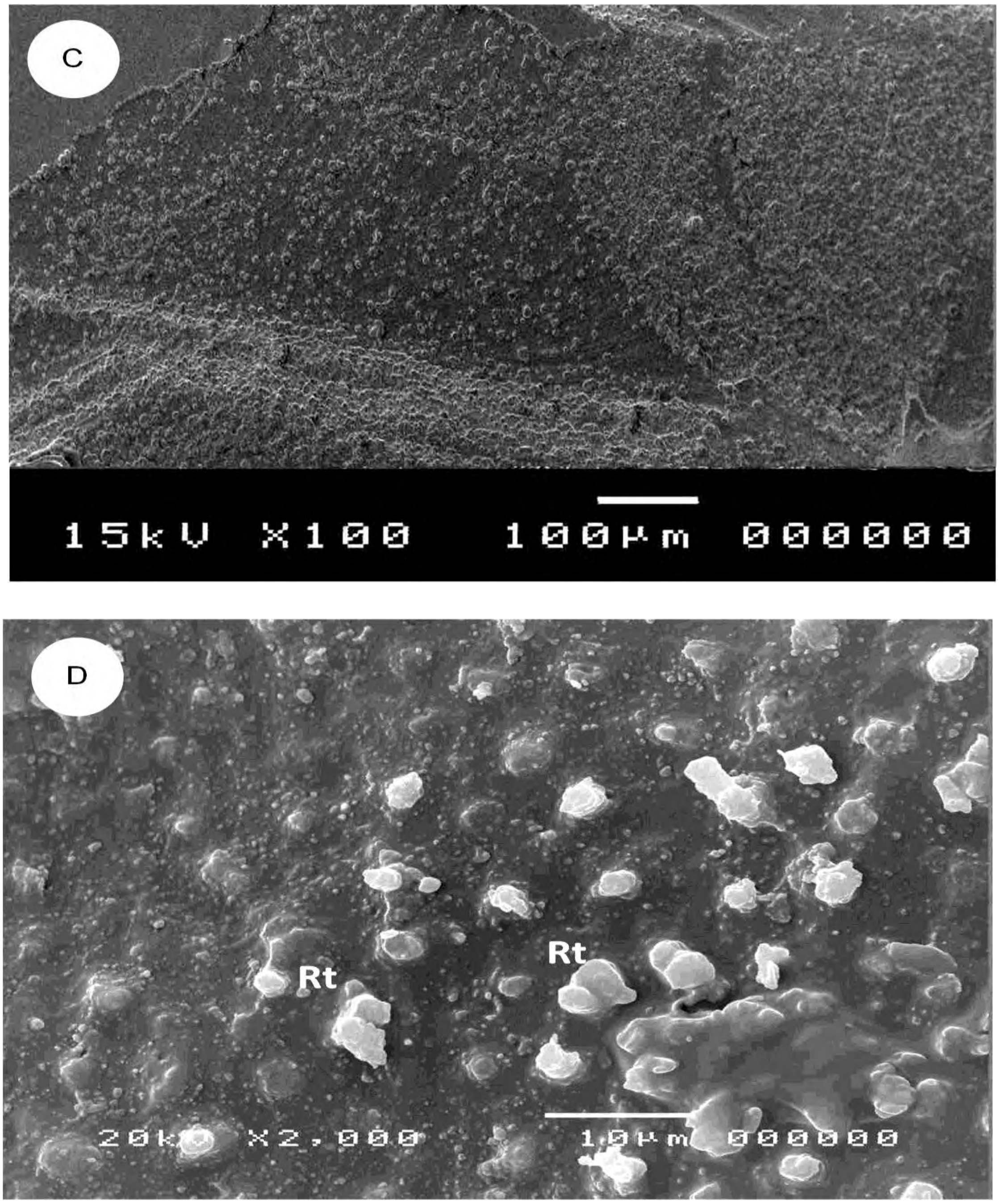
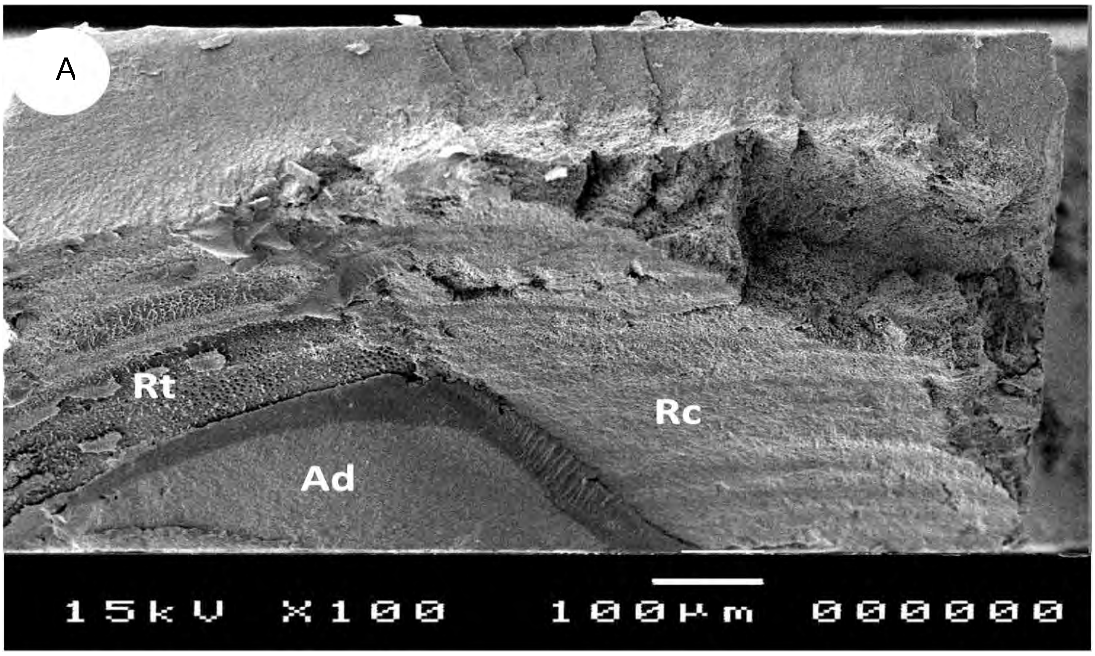
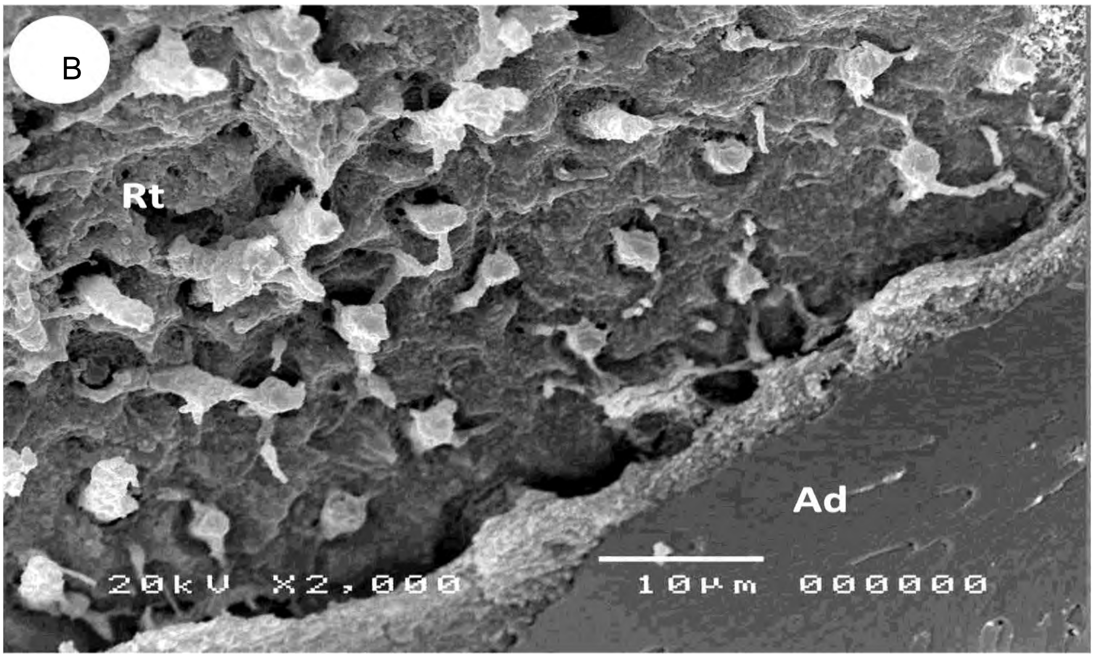


Figure 24 (A-D) Scanning electron micrographs identifying failure mode of fractured specimens from P90/ Low c-factor/ 24h group. Failure occurred along the adhesive – dentin interface.

A. Fractured surface on dentin side was magnified to a 100x to show adhesive failure.
B. Higher magnification of Figure 24A at 2,000X. Many of the dentinal tubules were occluded by resin tags (Rt) with few opened dentinal tubules (T).
C. Fractured surface of resin composite was magnified to a 100x to show adhesive failure.
D. Higher magnification of Figure 24C at 2,000X. Some area on resin composite side of fractured surface which remained attached to adhesive layer. Short resin tags could be observed.

Figure 25. Scaning electron micrographs of mix failed specimens.





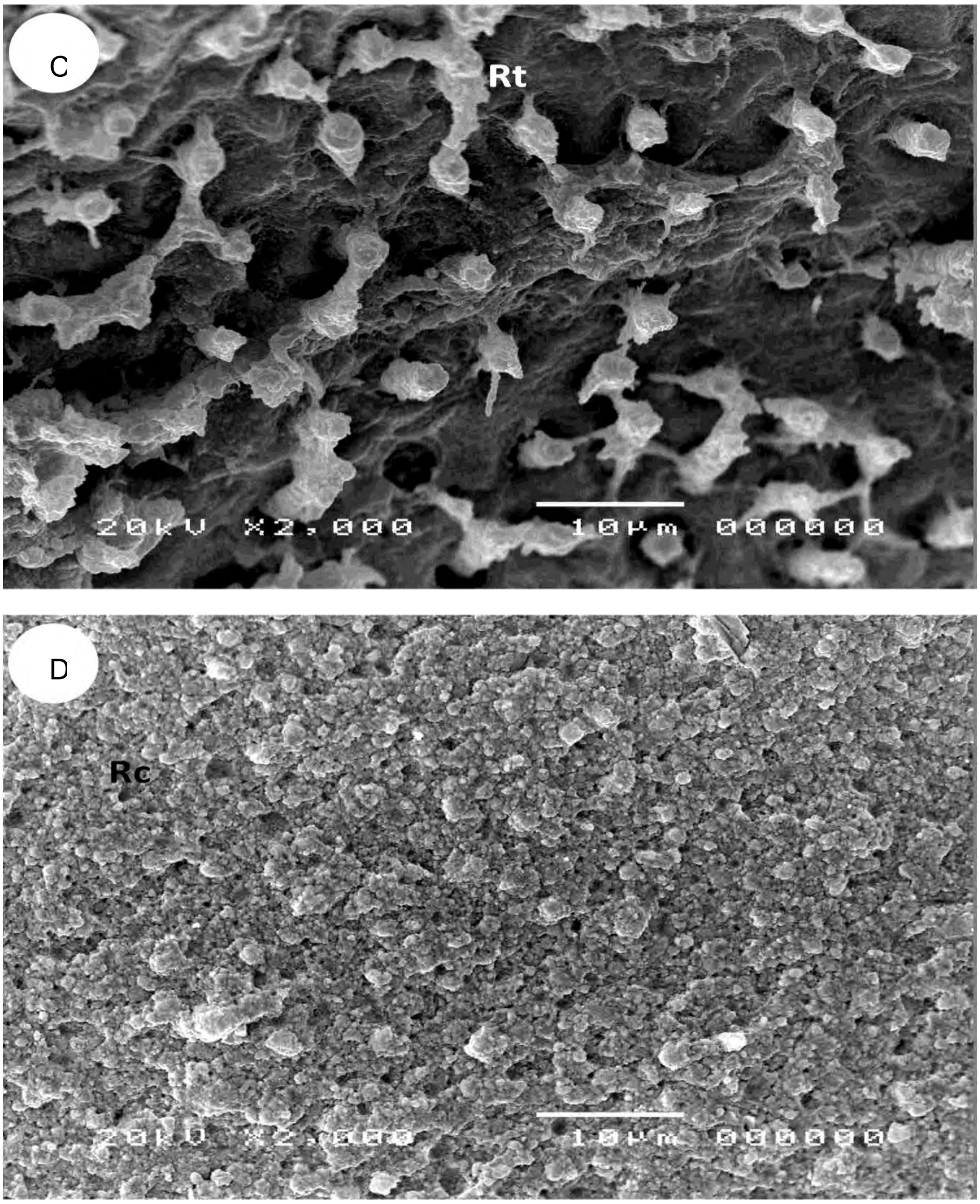


Figure 25 (A-D) Scanning electron micrographs identifying failure mode of fractured specimens from P90/Low c-factor/24h group. The specimen exhibited a mixed failure pattern involving adhesive failure along adhesive-dentin /adhesive composite interface as well as cohesive failure of resin composite.

A. Fractured surface of dentin side was magnified to a 100x to show the mixed failure exhibiting resin composite (Rc), resin tag (Rt) and resin adhesive (Ad).

B. Higher magnification of Figure 25A (2,000x) at the transition area between adhesive failure of adhesive-dentin interface and adhesive-resin composite interface.

C. Higher magnification of Figure 25A (2,000X) at the area that the failure was located at the bottom of hybrid layer leaving small remnants of resin tag (Rt) in dentinal tubules.

D. Higher magnification of Figure 25A (2,000X) at the area that failed within the resin composite (Cohesive failure in resin composite). Irregular shape of filler particles could be observed.

Failure modes were determined by stereomicroscope evaluation and some of the specimens were selected to confirm the mode of failure under SEM (Figures 24, 25 and Table 7). Percentage of failure modes of all groups demonstrated primarily adhesive failure between the resin composite and dentin (ZHC24h 85%; ZHC3m 95%; ZLC24h 90%; ZLC3m 85%; PHC24h 95%; PHC3m 100%; PLC24h 90%; PLC3m 100%), few mixed failure (ZHC24h 15%; ZHC3m 5%; ZLC24h 10%; ZLC3m 15%; PHC24h 5%; PHC3m 0%; PLC24h 10%; PLC 3m 0%) and no cohesive failures of dentin or resin composite were identified.

Figures 24A, 24B, 24C and 24D show the example of scaning electron micrograph of adhesive failure from P90/Low c-factor/24h group. Figures 24A and 24C show the total adhesive fractured surface on dentin side and resin composite side, respectively. A section in Figure 24A was shown as scaning electron micrograph at higher magnification in Figure 24B. Impregnation of dentinal tubules with resin tags can be seen in Figure 24B. A section in Figure 24C was shown as scaning electron micrograph at higher magnification in Figure 24D. Short resin tags that remained on some area of resin composite side can be seen in Figure 24D,

Example of mixed failure scaning electron micrographs from P90/Low c-factor/24h group shown in Figures 25A, 25B, 25C and 25D. Figure 25A shows the total fractured surface on dentin side. In Figure 25A, both adhesive failure between dentin and resin composite and cohesive failure in resin composite can be seen. The area that partial cohesive failure in resin composite and partial adhesive failure was shown as scaning electron micrograph at higher magnification in Figure 25B. Resin tags into the tubules of dentin failed adhesive can be seen in the magnified section in Figure 25C and irregular shape of filler particle of resin composite failed cohesive can be seen in the magnified section in Figure 25D.

.

Table 7. Number of specimens according to the failure mode for each experimental condition.

Groups			Cohesive in resin	Cohesive in dentin	Adhesive	Mixed
Material	c-factor	Water storage time				
Z250	High	24 hours	-	-	17/20 (85%)	3/20 (15%)
		3 months	-	-	19/20 (95%)	1/20 (5%)
	Low	24 hours	-	-	18/20 (90%)	2/20 (10%)
		3 months	-	-	17/20 (85%)	3/20 (15%)
P90	High	24 hours	-	-	19/20 (95%)	1/20 (5%)
		3 months	-	-	20/20 (100%)	-
	Low	24 hours	-	-	18/20(90%)	2/20(10%)
		3 months	-	-	20/20 (100%)	-

A Weibull analysis was performed to determine bond reliability at specific loads (Figure 26), the modulus and characteristic strength values at 95% and 90% probability of survival are shown in Table 8. The Weibull distribution parameters varied for different groups in this experiment. Overall, for all eight groups, the varied Weibull moduli in microtensile bond strength ranged between 3.81 and 8.02. The 95% probability of survival ($Ps_{0.95}$) represented the 5% failure probability. The 90% probability of survival ($Ps_{0.90}$) represented the 10% failure probability. For example, it could be interpreted that when ZHC3m group was subjected to the stress value of 11.78 MPa, 5% bond failure would be predicted. The maximum stress levels with $Ps_{0.95}$ and $Ps_{0.90}$ were ranked from the highest to the lowest as (1) ZLC24h, (2) ZHC24h, (3) ZLC3m, (4) PLC24h, (5) ZHC3m, (6) PLC3m, (7) PHC24h and (8) PHC3m as shown in Table 8 and Figure 26. The Weibull modulus ranking from the most variation to the least variation were (1) PHC3m (3.81) (2) ZLC3m (4.26) (3) ZHC24h (4.69) (4) ZHC3m (4.83) (5) PHC24h (5.64) (6) PLC3m (6.01) (7) ZLC24h (6.92) and (8) PLC24h (8.02), respectively. The higher Weibull modulus indicated better reliability. The correlation coefficient (*R* Square) was generally above 0.85 which could be interpreted that more than 85% of the data fit very well with the Weibull distribution.

Table 8. Critical bond strength, $Ps_{0.95}$ (corresponding to 95% probability of survival), $Ps_{0.90}$ (corresponding to 90% probability of survival) and Weibull modulus for microtensile bond strength.

Material	c-factor	Water storage time	Probability of survival; Ps (MPa)		R^2	Weibull modulus
			$Ps_{0.95}$	$Ps_{0.90}$		
Z250®	High	24 h	18.99	22.15	0.98	4.69
		3 m	11.78	13.68	0.97	4.83
	Low	24 h	37.83	41.98	0.85	6.92
		3 m	16.39	19.40	0.96	4.26
P90®	High	24 h	5.39	6.12	0.90	5.64
		3 m	3.53	4.26	0.90	3.81
	Low	24 h	14.56	15.92	0.96	8.02
		3 m	9.00	10.15	0.97	6.01

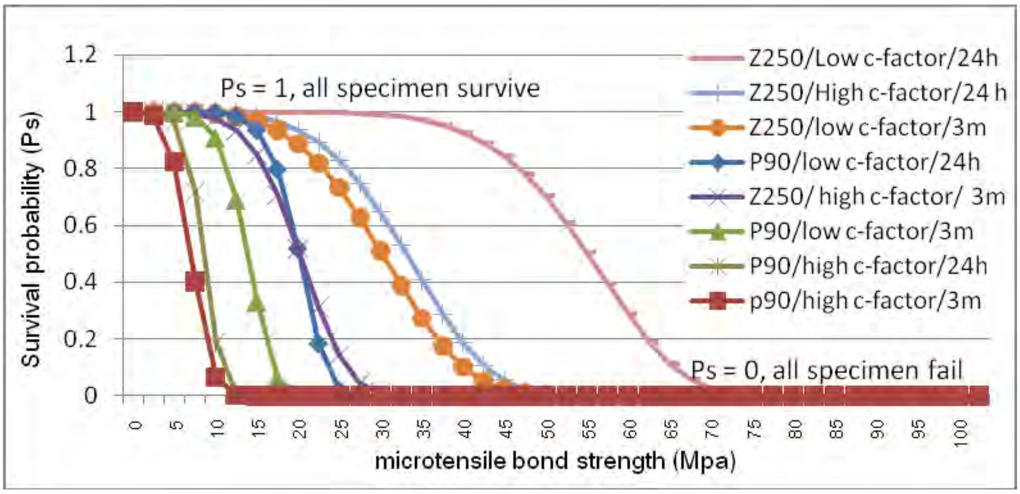
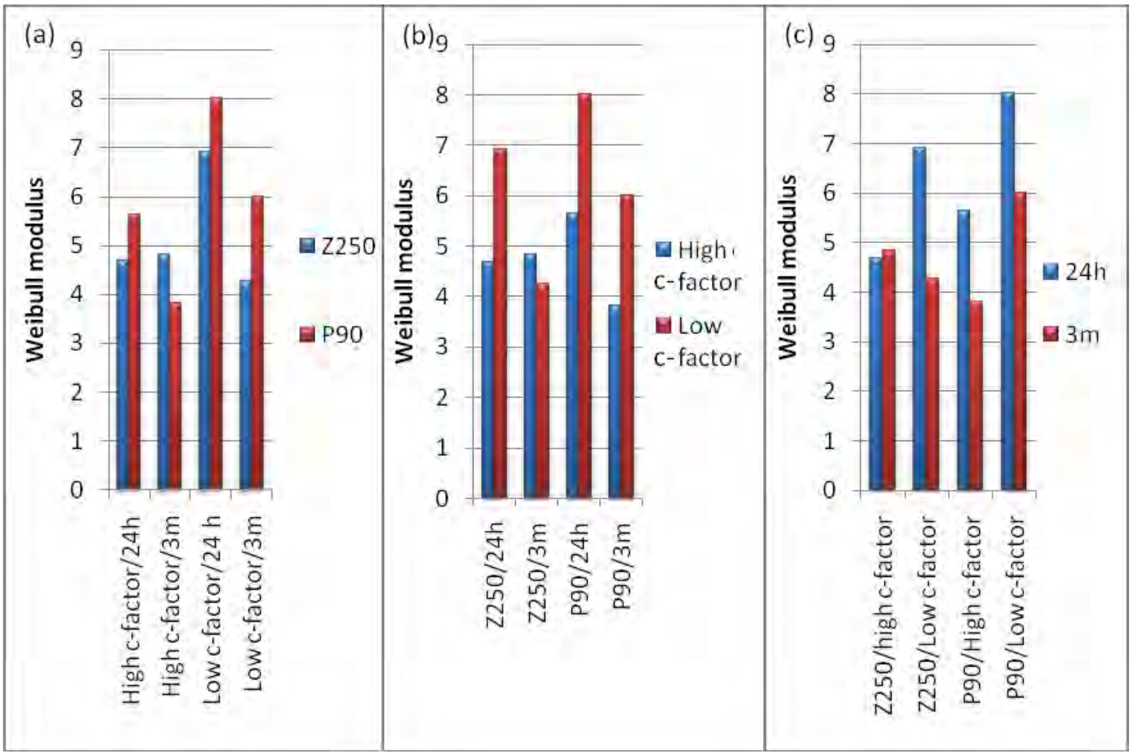


Figure 26. Survival probability vs the microtensile bond strength for Filtek P90® and Filtek Z250®

Comparison of the Weibull modulus between Filtek Z250® and Filtek P90® as shown in Figure 27(a). Weibull modulus of exhibited Filtek P90® higher than that of Filtek Z250®, except for the high c-factor cavity and 3 months of water storage group. When comparing the restoration which were applied to high and low c-factor cavities as shown in Figure 27(b), low c-factor presented higher Weibull modulus than high c-factor groups, except for Filtek Z250® at 3 months of water storage. Comparison of the Weibull modulus between various water storage times was shown in Figure 27(c). At 24 hours of water storage higher value of Weibull modulus compared to 3 month water storage groups, except Filtek Z250® high c-factor group.

Figure 27. Bar of Weibull modulus for microtensile bond strength affected by main factors (a) Type of material (b) c-factor (c) Water storage time.



CHAPTER V

DISCUSSIONS

In the present study, a newly introduced silorane based resin composite system was evaluated for its performances in high and low c-factor class I cavities. Restorations were also challenged under long term, 3 months, water storage. Combining high polymerization shrinkage of methacrylate-based resin composite and high c-factor at 3 month water storage time, a ‘worst-case scenario’ regarding shrinkage stress was created. The methacrylate-based resin composite (Filtek Z250® and Scotchbond Multipurpose®) was chosen as a control material to differentiate polymerization pathways; free radical polymerization of methacrylate-based resin composite compared to cationic polymerization for silorane-based resin composite system (Filtek P90® and Silorane system® adhesive).(18) The two adhesives used were produced by the same manufacturer having similar solvent (water/ethanol based) systems, however, one was considered a 3-step total etch system, whereas another was a 2-step self etch system. Data from previous studies indicated that the bonding efficacy of a 2-step self etch adhesive was comparable to a 3-step total etch adhesive in dentin.(24) The microtensile test was chosen because it is considered by majority of researchers in the bonding field as the most reliable technique for assessing “true” interfacial bond strength between an adhesive material and the substrate of interest.(41, 92)

In the present study, a smear layer was produced with diamond burs, as is routinely performed in daily dental practice. To produce a standardized smear layer, the diamond bur was discarded after every two preparations. The preparations were standardized in dimensions to minimize variability. Class I preparation were used in order to create a more clinically relevant cavity design, compared to flat enamel/dentin surface used in other studies.(15, 28, 79) Inside a tooth cavity preparation, shrinkage stress is generated during polymerization of the resin composite, pulling the adhesive away from cavity walls.(4, 10) High stresses may be imposed upon the early maturing dentin adhesive resin bonding system and this stress was detrimental to the long-term integrity of the adhesively bonded joint. (15)

Filtek Z250®, a methacrylate-based microhybrid resin composite, and Filtek P90®, a silorane-based microhybrid resin composite were used in the present study, of which data indicated that difference in type of resin composites provided significant differences in microtensile bond strengths. Therefore, the first null hypothesis was rejected because significant differences in bond strength values were observed among different type of materials. The manufacturer, 3M ESPE, reported 2% volumetric polymerization shrinkage of Filtek Z250®, and <1% for Filtek P90®. The difference may be due to different chemical nature of the Filtek P90®. Since silorane-based resins have been reported to produce minimal polymerization shrinkage (18, 106, 114), and with the application of additional coats of a filled adhesive layer (Silorane bond®) acting as an intermediate flexible layer, the interface is supposedly exposed to less stress.(115) It was reported that no statistical difference in bond strength was recorded between Filtek Silorane® and Filtek Z100®.(116) However, the present study showed better bond strength in class I cavities using Filtek Z250® with Scotchbond Multipurpose® compared to that of Filtek P90® with Silorane system® adhesive, using similar experimental conditions. This may be explained by the difference in characteristics of the adhesive agent.

The adhesive systems evaluated in the present study have different compositions and used different approaches, either by completely removing the smear layer (the total-etch technique; Scotchbond Multipurpose®) or by modifying it (the self-etch technique: Silorane system® adhesive).(24) The present study showed that silorane system adhesive presented lower bond strength value than that of Scotchbond Multipurpose®. This may be due to the two reasons. The first reason involved thickness of the interface because application of two layers of resin from silorane primer and silorane bond was suggested by the manufacture. According to D'Arcangelo et al., an excess of adhesive layer thickness could negatively influence the strength and quality of adhesion.(117) Therefore, this greater thickness of bonding layer may be a weak point of bonding interface resulting in lower µTBS values. Secondly, the mechanism of bonding to dentin of the silorane system® adhesive resembled that of a one-step self etch adhesive, because the actual bond to the tooth surface was realized by the silorane primer only.(118) As such ‘ultramild’ one-step self-etch adhesives only interacted superficially with the smear layer covered dentin, the bonding interface was much more dependent on the integrity of this smear layer.(119) This led to the creation of a hybrid layers in a one-step self-etch adhesive that could behave as semi-permeable membranes permitting water movements throughout the bonded interface even after the adhesive was polymerized.(120)

Decrease in μTBS value when bonding to high c-factor cavity was statistically significant for all groups ($p < 0.05$). Although Filtek P90® is a low-shrinking resin composite, and thus a better bond strength might be expected, a significant decrease in μTBS to high c-factor cavity was observed. Our findings are in agreement with those of Van Ende et al.(116), where the geometric configuration of specimens was an important factor affecting μTBS. The cavity configuration factor is a reasonable explanation for the decrease in bond strength observed when high c-factor cavity dentin was bonded.(15, 65, 121, 122) Consequently, the second null hypothesis was rejected, because significant differences in bond strength values were observed among the different c-factor cavities. As c-factor refers to the ratio of bonded to unbonded surfaces in a tooth preparation, Therefore, If two class I cavities had the same volume but different designs, an ellipsoidal shaped cavity would present a lower c-factor than the box shaped cavity.(2) An increased c-factor leads to a decreased flow capacity causing a higher rate of shrinkage stress development.(4) In the present study, ellipsoidal shaped class I cavities, prepared with a length of 7.0 mm., a width of 5.0 mm. and a depth of 3.0 mm., was computed to have a volume of 55 mm^3 and a c-factor of 1.4. Box shaped class I cavities, prepared with a length of 6.0 mm., a width of 3.0 mm. and a depth of 3.0 mm., was computed to have a volume of 54 mm^3 and a c-factor of 4.0. Low c-factor cavities provided less restraint of the material by cavity walls, providing a beneficial effect of high bond strength.(2) However, it is not easy to transfer the concept of c-factor directly to clinical situations, since tooth cavity preparations have a much more complex geometry than the specimens used in mechanical testing experimentally, thus tooth-adhesive-resin composite system exhibit a very heterogeneous stress distribution in clinical use.(8, 123) Moreover, photoelastic studies suggested that c-factors would be applicable to restorations only when comparing cavities with similar volumes.(124)

Three months of storage in distilled water at 37°C used in the present study is considered a brief period in comparison to the life expectancy of the restorations. However, direct exposure to water for three months resulted in a significant decrease of the µTBS of most groups (ZHC, ZLC and PLC groups), except PHC group ($p > 0.05$). Consequently, the third null hypothesis was rejected for the ZHC, ZLC and PLC groups, but not the PHC group. Similarly to studies previously mentioned, the present study showed that bond strength values of Scotchbond multipurpose® decreased over time.(39, 79) A study using silorane system® adhesive, particularly after aging, showed areas of possible hydrolytic degradation or phase separation of the adhesive layers.(125) Water can compete with the silorane monomer for oxonium ions, resulting in incomplete polymerization at the adhesive layer interface.(125) Once stressed by aging, or mechanical loading (not tested for our study), this incomplete polymerization zone potentially affects long-term bonding. Chemical reactions are responsible for degradation of resin-dentin bonds over time. Decrease in bond strength results in loss of stability of the adhesive systems,(75) and extraction of resin-material from the hybrid layer.(74) Also, a fall in bond strength has been ascribed to hydrolysis of adhesive and collagen fibrils at the base of hybrid layer(74, 75, 99, 126), thereby, weakening the physical properties of the resin-dentin bond.(99) Explanation for insignificant decrease over time (3 months of storage) in µTBS of PHC groups may be related to very large number of pretest failure for PHC at 3 months. In the present investigation, pretest failures were not included in the statistical analyses, therefore, there may have been an overestimation of the bonding potential for PHC3m group.

In this study, specimens fractured during trimming to produce hourglass shaped specimens were excluded from the statistical analysis, because the stress value at the interface during fracture would have been unknown. The relatively high incidence of pretest failure (28.57%-75.31%) observed can be explained by the stress generated by the hourglass specimen preparation.(90, 95) This finding corroborates the observations in previous study that also showed a great number of pretest failure (26%) during hourglass trimming of microtensile specimen.(94) Thickness (< 1.5 mm.) of the specimen affected its ability to survive the preparation procedures necessary for microtensile testing.(94) Shono et al., related the rate of pretest failures using a trimming technique to the mean microtensile bond strength measured and reported that groups with bond strength of 13 MPa or lower were less likely to survive preparation.(127) In addition, the reduction in bond strength may contribute to the increase in pretest failed specimens. The used of Filtek Z250® with Scotchbond Multipurpose® in low c-factor condition at 24 hour water storage time showed less pretest failure (3/23 or 28.57%), and also provided the highest bond strength value. The used of Filtek P90® with silorane system® adhesive in high c-factor condition at 3 month water storage showed the highest pretesting failure (61/81 or 75.31%), and also provided the lowest bond strength. It has been noted that when calculations are based on specimens surviving test preparation procedures, there may be an overestimation of the bonding potential.(92, 128) However, if zero values were attributed, bond strengths may be underestimated.(92, 128) Therefore, not only the bond strength values but also pre-test failures should be taken into consideration during interpretation of the bond strength of adhesives to dentin. Determination of type of failure from this study revealed that the majority of the specimens showed adhesive failures indicating that the µTBS method was an appropriate test to evaluate bond strength of adhesive.(23, 27)

The Weibull modulus (m) indicated reliability of the bond strength.(21) With this statistical approach, a more relevant clinical approximation for the risk level of failure can be made. The reliability of adhesives can be evaluated clinically using criteria from the American Dental Association, which stated that failure rate of class V restorations should be no more than 5% after 6 months for “provisional acceptance”, and 10% after 18 months for “full acceptance”.(98) However, minimum bond strength value for *in vitro* conditions has never been defined. Therefore, the stress levels which cause the first 5% and 10% of the specimens to fail were considered more clinically relevant to evaluate the reliability of the bond, rather than using the average strength values.(99) Ranking critical tensile bond strength corresponding to 5% and 10% of failed specimens from this study showed a very small difference, compared to the results of ranking using mean μTBS values. Probabilities of survival at 95% and 90% of PLC24h could withstand stress more than ZHC3m, while mean μTBS of ZHC3m was higher than that of PLC24h, but not statistically significant. The small difference in ranking of strength may be because ZHC3m and PLC24h provided quite similar mean bond strengths (19.97 MPa and 19.89 MPa, respectively), however, significantly different in reliability values.

According to the results of Weibull modulus (Table 8), the most reliable bond strength group was PLC24h, followed by ZLC24h, PLC3m, PHC24h, ZHC3m, ZHC24h, ZLC3m and PHC3m, respectively. The differences of Weibull modulus between groups may be attributed by main factors (type of material, c-factor and water storage time) as shown in Figure 27. The used of Filtek Z250®, high c-factor condition and 3 months of water storage seem to be factors affecting to decrease in Weibull modulus or decrease reliability of microtensile bond strength. Six studies using the microtensile bond test (15, 96, 129-132) reported Weibull modulus values ranging from 1.7 to 5 for 16 adhesives bonded to dentin. Low Weibull modulus values,ranging from 3.81 to 8.02, of the μTBS tested in this study were likely to be due to inherent material properties, c-factor conditions and water storage times, indicating rather poor reliability. Furthermore, the performance reliability of bond strength data remained low, confirming the limitations of bond tests which were prone to high scattering from non-uniform stress states, as well as the variety of specimen preparation (hourglass trimming), and handling of materials, which may be difficult to control as already discussed.

A limitation of this study was the results were obtained under conditions different from those in clinical practice. In clinical practice the optimum bond strength of resin composites to dentin is not known. The bond strengths were dependent not only on the materials used, c-factor, or water storage time, but also other factors, such as quality of substrate, restorative technique, skills of the operator, type and technique of light curing, isolation method, and finishing technique, etc.,(24, 70) In addition, patient-related factors, such as age, oral hygiene, occlusal loading, and dentin sclerosis, may be more influential than any material property.(24, 70)

Conclusion

Silorane system® adhesive formed weak bond to dentin at the bottom of an occlusal cavity, especially in high c-factor condition after 3 months of water storage. While this new restorative material has been considered a low shrinkage material, it still has not been able to fulfill all the requirements for a long-term durable bonding. The need of an adhesive to achieve acceptable bond strengths restricts the universal application of silorane-based resin composites. Further *in vitro* and *in vivo* investigations are necessary to confirm the present finding and to test the bonding performance of a clinically relevant cavity wall such as that prepared on caries- affected dentin.

Within the limitation of this *in vitro* study, the results led to rejection of all null hypotheses. The bond strength was dependent on type of materials, c-factor and aging by water storage. The following conclusions may be drawn;

1. There were differences in microtensile bond strengths between Filtek P90® and Filtek Z250®, when used with their corresponding adhesive systems, in class I restorations.
2. C-factors affected the microtensile bond strengths of Filtek P90® and Filtek Z250®, when used with their corresponding adhesive systems, in class I restorations.
3. Artificial aging by water storage affected microtensile bond strengths of Filtek P90® and Filtek Z250®, except for Filtek P90® in high c-factor group, when used with their corresponding adhesive systems, in class I restoration.

A silorane-based resin composite provided lower bond strength when compared to a methacrylate-based resin composite, in spite of low-shrinkage property claimed by manufacturer, especially in a high c-factor cavity. When adding aging condition to the specimens, the bond furthermore deteriorated. Therefore, properties of resin composite alone did not determine performance of restoration. One must take into account other involved factors, including performance of bonding agents, configuration factors and aging process. Outcomes of the present study could not advocate the use of a silorane-based resin composite and its corresponding adhesive, especially in a high c-factor cavity under 3 month water storage. However, findings of the present study warrant further improvement of the system.

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APPENDIX

Table 1. Data of microtensile bond test (GROUP I: ZHC24h)

GROUP I: ZHC24h				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.24	38.23	38.54
2	0.8	1.09	39.74	45.57
3	0.8	1.02	27.91	34.20
4	0.8	1.25	35.01	35.01
5	0.8	1.04	27.02	32.48
6	0.8	1.03	30.24	36.70
7	0.8	1.02	31.77	38.93
8	0.8	1.02	23.51	28.81
9	0.8	0.91	20.79	28.56
10	0.8	1.02	34.12	41.81
11	0.8	0.76	27.91	43.37
12	0.8	1.02	20.59	25.23
13	0.8	1.01	13.34	16.51
14	0.8	1.25	29.39	29.39
15	0.8	1.25	31.3	31.30
16	0.8	1.01	21.97	27.19
17	0.8	1.05	18.64	22.19
18	0.8	1.2	36.7	38.23
19	0.8	1.03	25.17	30.55
20	0.8	0.92	20.59	27.98
Mean				32.63
Standard deviation				7.33

Table 2. Data of microtensile bond test (GROUP II: ZHC3m)

GROUP II: ZHC3m				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.18	18.63	19.74
2	0.8	1.02	9.53	11.68
3	0.8	1.1	19.93	22.65
4	0.8	1.19	19.91	20.91
5	0.8	1.17	14.25	15.22
6	0.8	1.18	22.26	23.58
7	0.8	0.85	8.44	12.41
8	0.8	0.97	17.37	22.38
9	0.8	1.02	12.5	15.32
10	0.8	1.25	17.63	17.63
11	0.8	1.23	21.31	21.66
12	0.8	1.25	28.7	28.70
13	0.8	1.22	18.91	19.38
14	0.8	1.24	26.45	26.66
15	0.8	1.22	14.98	15.35
16	0.8	1.2	16.26	16.94
17	0.8	1.24	22.31	22.49
18	0.8	0.97	18.23	23.49
19	0.8	0.99	17.21	21.73
20	0.8	1.07	18.41	21.51
			Mean	19.97
			Standard deviation	4.48

Table 3. Data of microtensile bond test (GROUP III: ZLC24h)

GROUP III: ZLC24h				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.22	67.82	69.49
2	0.8	1.17	70.19	74.99
3	0.8	0.92	35.57	48.33
4	0.8	0.98	35.12	44.80
5	0.8	1.03	50.99	61.88
6	0.8	1.24	58.6	59.07
7	0.8	0.84	31.38	46.70
8	0.8	0.97	50.79	65.45
9	0.8	0.87	36.27	52.11
10	0.8	0.89	30.17	42.37
11	0.8	1.25	46.63	43.37
12	0.8	1.03	41.54	50.41
13	0.8	1.23	55.95	56.86
14	0.8	1.25	48.8	48.80
15	0.8	1.03	39.4	47.82
16	0.8	1.24	49.12	49.52
17	0.8	1.02	40.69	49.87
18	0.8	1.25	57.93	57.93
19	0.8	1.25	63.26	63.26
20	0.8	1.24	50.32	50.73
			Mean	54.19
			Standard deviation	9.05

Table 4. Data of microtensile bond test (GROUP IV: ZLC3m)

GROUP IV: ZLC3m				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.23	30.55	31.05
2	0.8	1.07	21.24	24.81
3	0.8	1.16	37.48	40.39
4	0.8	1.13	25.05	27.71
5	0.8	1.22	35.46	36.33
6	0.8	1.25	22.64	22.64
7	0.8	1.25	21.31	21.31
8	0.8	1.04	20.19	24.27
9	0.8	1.23	20.12	20.45
10	0.8	1.25	36.71	36.71
11	0.8	1.18	33.12	35.08
12	0.8	1.23	28.42	28.88
13	0.8	1.18	26.17	27.72
14	0.8	1.21	40.84	42.19
15	0.8	1.14	15.2	16.67
16	0.8	1.22	21.26	21.78
17	0.8	1.24	27.97	28.20
18	0.8	1.25	42.8	42.80
19	0.8	1.06	34.23	40.37
20	0.8	1.08	25.67	29.71
			Mean	29.95
			Standard deviation	7.88

Table 5. Data of microtensile bond test (GROUP V: PHC24h)

GROUP V: PHC24h				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.25	7.82	7.82
2	0.8	1.22	8.49	8.70
3	0.8	1.17	6.34	6.77
4	0.8	1.04	5.23	6.29
5	0.8	1.2	9.87	10.28
6	0.8	1.12	5.58	6.23
7	0.8	1.25	7.98	7.98
8	0.8	1.12	7.17	8.00
9	0.8	1.03	7.98	9.68
10	0.8	1.23	7.26	7.38
11	0.8	1.21	6.04	6.24
12	0.8	1.2	10.45	10.89
13	0.8	1.16	7.89	8.50
14	0.8	1.2	10.85	11.30
15	0.8	1.24	8.1	8.17
16	0.8	1.11	5.98	6.73
17	0.8	1.24	10.5	10.58
18	0.8	1.22	10.89	11.16
19	0.8	1.23	8.34	10.67
20	0.8	1.25	7.64	7.64
			Mean	8.55
			Standard deviation	1.76

Table 6. Data of Microtensile bond test (GROUP VI: PHC3m)

GROUP VI: PHC3m				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.08	6.33	7.33
2	0.8	1.25	7.65	7.65
3	0.8	1.24	5.31	5.35
4	0.8	1.23	4.8	4.88
5	0.8	1.21	5.21	5.38
6	0.8	1.23	4.72	4.80
7	0.8	1.17	10.04	10.73
8	0.8	1.25	10.65	10.65
9	0.8	1.23	4.9	4.98
10	0.8	1.25	9.9	9.90
11	0.8	1.24	6.24	6.29
12	0.8	1.25	6.77	6.77
13	0.8	1.25	9.68	9.68
14	0.8	1.24	7.47	7.53
15	0.8	1.23	7.09	7.21
16	0.8	1.24	6.7	6.76
17	0.8	1.11	5.08	5.72
18	0.8	1.23	4.29	4.36
19	0.8	1.23	4.27	4.34
20	0.8	1.23	8.45	8.59
			Mean	6.94
			Standard deviation	2.07

Table 7. Data of microtensile bond test (GROUP VII: PLC24h)

GROUP VII :PLC24h				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.23	20.43	20.76
2	0.8	1.25	20.55	20.55
3	0.8	1.23	19.11	19.42
4	0.8	1.24	19.33	19.49
5	0.8	1.01	19.46	24.08
6	0.8	1.18	18.75	19.86
7	0.8	1.24	23.84	24.03
8	0.8	1.21	18.21	18.81
9	0.8	1.25	17.85	17.85
10	0.8	1.2	18.04	18.79
11	0.8	1.16	15.41	16.61
12	0.8	1.2	18.38	19.15
13	0.8	1.23	15.71	15.97
14	0.8	1.25	17.53	17.53
15	0.8	1.23	20.56	20.89
16	0.8	1.2	18.82	19.60
17	0.8	1.3	14.86	14.29
18	0.8	1.25	21.27	21.27
19	0.8	1.23	24.56	24.96
20	0.8	1.17	22.34	23.87
			Mean	19.89
			Standard deviation	2.82

Table 8. Data of microtensile bond test (GROUP VIII: PLC3m)

GROUP VIII :PLC3m				
Sample No.	Thick (mm.)	Width (mm.)	Force (N)	Microtensile bond strength (MPa)
1	0.8	1.25	17.45	17.45
2	0.8	1.23	16.22	16.48
3	0.8	1.21	13.85	14.31
4	0.8	1.24	15.04	15.16
5	0.8	1.25	17.36	17.36
6	0.8	1.19	12.85	13.50
7	0.8	1.22	13.01	13.33
8	0.8	1.25	18.85	18.85
9	0.8	1.19	14.26	14.98
10	0.8	1.19	11.49	12.07
11	0.8	1.22	12.47	12.78
12	0.8	1.23	10.65	10.82
13	0.8	0.97	11.57	14.91
14	0.8	1.03	9.8	11.90
15	0.8	1.02	7.42	9.09
16	0.8	1.03	8.06	9.78
17	0.8	1.25	12.47	12.47
18	0.8	1.09	13.09	15.01
19	0.8	1.07	10.79	12.61
20	0.8	1.12	10.22	11.41
			Mean	13.71
			Standard deviation	2.60

Table 9. The Shapiro-Wilk test for normality

	Type*c-factor*storage time	Shapiro-Wilk		
		Statistic	df	Sig.
microtensile bond strength	PHC24h	.921	20	.101
	PHC3m	.919	20	.096
	PLC24h	.963	20	.595
	PLC3m	.982	20	.952
	ZHC24h	.981	20	.948
	ZHC3m	.966	20	.665
	ZLC24h	.915	20	.081
	ZLC3m	.949	20	.350

a. Lilliefors Significance Correction

*. This is a lower bound of the true significance.

Table 10. The 3-way ANOVA test of between subjects effects

Tests of Between-Subjects Effects

Dependent Variable: microtensile bond strength

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	34013.536[a]	7	4859.077	162.183	.000
Intercept	86504.943	1	86504.943	2887.300	.000
c-factor	6198.821	1	6198.821	206.900	.000
Storage time	5029.536	1	5029.536	167.872	.000
type	19378.393	1	19378.393	646.798	.000
c-factor * storage time	663.646	1	663.646	22.151	.000
c-factor * type	446.284	1	446.284	14.896	.000
aging * type	2176.624	1	2176.624	72.650	.000
c-factor * storage time * type	120.231	1	120.231	4.013	.047
Error	4553.995	152	29.960		
Total	125072.474	160			
Corrected Total	38567.531	159			

a. R Squared = .882 (Adjusted R Squared = .876)

Table 11. The Levene test of homogeneity of variances

Test of Homogeneity of Variances

microtensile bond strength

Levene Statistic	df1	df2	Sig.
13.393	7	152	.000

Table 12. The Brown-Forsythe test of equality of means

Robust Tests of Equality of Means

microtensile bond strength

	Statistic[a]	df1	df2	Sig.
Brown-Forsythe	162.183	7	78.893	.000

a. Asymptotically F distributed.

Table 13. The Tamhane multiple comparison test of different between groups

Tamhane Multiple Comparisons

(I) groups	(J) groups	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
PHC24h	PHC3m	1.49712	.59613	.374	-.5066	3.5009
	PLC24h	-11.44800*	.73435	.000	-13.9508	-8.9452
	PLC3m	-5.27187*	.69337	.000	-7.6249	-2.9188
	ZHC24h	-24.31320*	1.72820	.000	-30.4784	-18.1480
	ZHC3m	-11.53014*	1.07032	.000	-15.2742	-7.7861
	ZLC24h	-45.90908*	2.01933	.000	-53.1391	-38.6790
	ZLC3m	-21.51213*	1.80257	.000	-27.9496	-15.0747
PHC3m	PHC24h	-1.49712	.59613	.374	-3.5009	.5066
	PLC24h	-12.94511*	.78178	.000	-15.5827	-10.3076
	PLC3m	-6.76899*	.74342	.000	-9.2698	-4.2682
	ZHC24h	-25.81031*	1.74888	.000	-32.0109	-19.6097
	ZHC3m	-13.02725*	1.10340	.000	-16.8443	-9.2102
	ZLC24h	-47.40620*	2.03705	.000	-54.6649	-40.1475

Tamhane Multiple Comparisons

(I) groups	(J) groups	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
PLC24h	PHC24h	11.44800*	.73435	.000	8.9452	13.9508
	PHC3m	12.94511*	.78178	.000	10.3076	15.5827
	PLC3m	6.17612*	.85823	.000	3.2988	9.0534
	ZHC24h	-12.86520*	1.80069	.000	-19.1658	-6.5646
	ZHC3m	-.08214	1.18380	1.000	-4.1052	3.9409
	ZLC24h	-34.46108*	2.08170	.000	-41.7998	-27.1224
	ZLC3m	-10.06413*	1.87218	.000	-16.6291	-3.4992
PLC3m	PHC24h	5.27187*	.69337	.000	2.9188	7.6249
	PHC3m	6.76899*	.74342	.000	4.2682	9.2698
	PLC24h	-6.17612*	.85823	.000	-9.0534	-3.2988
	ZHC24h	-19.04133*	1.78437	.000	-25.3088	-12.7738
	ZHC3m	-6.25826*	1.15883	.000	-10.2137	-2.3028
	ZLC24h	-40.63721*	2.06760	.000	-47.9495	-33.3249
	ZLC3m	-16.24026*	1.85649	.000	-22.7741	-9.7064
ZHC24h	PHC24h	24.31320*	1.72820	.000	18.1480	30.4784
	PHC3m	25.81031*	1.74888	.000	19.6097	32.0109
	PLC24h	12.86520*	1.80069	.000	6.5646	19.1658
	PLC3m	19.04133*	1.78437	.000	12.7738	25.3088
	ZHC3m	12.78306*	1.96184	.000	6.0949	19.4712
	ZLC24h	-21.59588*	2.60402	.000	-30.3388	-12.8529
	ZLC3m	2.80107	2.43977	1.000	-5.3758	10.9779

Tamhane Multiple Comparisons

(I) groups	(J) groups	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
ZHC3m	PHC24h	11.53014*	1.07032	.000	7.7861	15.2742
	PHC3m	13.02725*	1.10340	.000	9.2102	16.8443
	PLC24h	.08214	1.18380	1.000	-3.9409	4.1052
	PLC3m	6.25826*	1.15883	.000	2.3028	10.2137
	ZHC24h	-12.78306*	1.96184	.000	-19.4712	-6.0949
	ZLC24h	-34.37895*	2.22257	.000	-42.0273	-26.7306
	ZLC3m	-9.98199*	2.02765	.001	-16.9118	-3.0522
ZLC24h	PHC24h	45.90908*	2.01933	.000	38.6790	53.1391
	PHC3m	47.40620*	2.03705	.000	40.1475	54.6649
	PLC24h	34.46108*	2.08170	.000	27.1224	41.7998
	PLC3m	40.63721*	2.06760	.000	33.3249	47.9495
	ZHC24h	21.59588*	2.60402	.000	12.8529	30.3388
	ZHC3m	34.37895*	2.22257	.000	26.7306	42.0273
	ZLC3m	24.39695*	2.65395	.000	15.4944	33.2995
ZLC3m	PHC24h	21.51213*	1.80257	.000	15.0747	27.9496
	PHC3m	23.00925*	1.82240	.000	16.5384	29.4801
	PLC24h	10.06413*	1.87218	.000	3.4992	16.6291
	PLC3m	16.24026*	1.85649	.000	9.7064	22.7741
	ZHC24h	-2.80107	2.43977	1.000	-10.9779	5.3758
	ZHC3m	9.98199*	2.02765	.001	3.0522	16.9118
	ZLC24h	-24.39695*	2.65395	.000	-33.2995	-15.4944

*. The mean difference is significant at the 0.05 level.

Table 14. Weibull analysis (GROUP I: ZHC24h)

WEIBULL ZHC 24h		
Beta (or shape parameter)	4.686220138	
Alpha (or characteristic life)	35.79781743	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	3.8293E-06	0.999996
5	9.85807E-05	0.999901
7.5	0.000658981	0.999341
10	0.002534868	0.997465
12.5	0.007195826	0.992804
15	0.016827852	0.983172
17.5	0.03434544	0.965655
20	0.063253902	0.936746
22.5	0.107276029	0.892724
25	0.169665191	0.830335
27.5	0.252195461	0.747805
30	0.353976625	0.646023
32.5	0.470475793	0.529524
35	0.593334632	0.406665
37.5	0.711542125	0.288458
40	0.814045907	0.185954
42.5	0.893006559	0.106993
45	0.94614644	0.053854
47.5	0.976807579	0.023192
50	0.991659826	0.00834
52.5	0.997561386	0.002439
55	0.999436784	0.000563
57.5	0.999900426	9.96E-05
60	0.999986973	1.3E-05
62.5	0.999998784	1.22E-06
65	0.999999922	7.79E-08
67.5	0.999999997	3.28E-09
70	1	8.71E-11
72.5	1	1.39E-12
75	1	1.25E-14
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 15. Weibull analysis (GROUP II: ZHC3m)

WEIBULL ZHC 3m		
Beta (or shape parameter)	4.833537	
Alpha (or characteristic life)	21.78412	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	2.85E-05	0.999971
5	0.000814	0.999186
7.5	0.00576	0.99424
10	0.022938	0.977062
12.5	0.065959	0.934041
15	0.151864	0.848136
17.5	0.29319	0.70681
20	0.484	0.516
22.5	0.689373	0.310627
25	0.857093	0.142907
27.5	0.954226	0.045774
30	0.990873	0.009127
32.5	0.999007	0.000993
35	0.99995	5.05E-05
37.5	0.999999	1.01E-06
40	1	6.41E-09
42.5	1	1.04E-11
45	1	3.33E-15
47.5	1	0
50	1	0
52.5	1	0
55	1	0
57.5	1	0
60	1	0
62.5	1	0
65	1	0
67.5	1	0
70	1	0
72.5	1	0
75	1	0
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 16. Weibull analysis (GROUP III: ZLC24h)

WEIBULL ZLC 24h		
Beta (or shape parameter)	6.920967482	
Alpha (or characteristic life)	58.10953989	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	3.49809E-10	1
5	4.23886E-08	1
7.5	7.01409E-07	0.999999
10	5.1365E-06	0.999995
12.5	2.40644E-05	0.999976
15	8.49908E-05	0.999915
17.5	0.000246986	0.999753
20	0.000622232	0.999378
22.5	0.001405422	0.998595
25	0.002911824	0.997088
27.5	0.00562408	0.994376
30	0.010246475	0.989754
32.5	0.01776276	0.982237
35	0.029489133	0.970511
37.5	0.047106876	0.952893
40	0.072649214	0.927351
42.5	0.108405097	0.891595
45	0.156692873	0.843307
47.5	0.219457921	0.780542
50	0.297675476	0.702325
52.5	0.390611335	0.609389
55	0.495118047	0.504882
57.5	0.605295768	0.394704
60	0.712932556	0.287067
62.5	0.809002649	0.190997
65	0.886025108	0.113975
67.5	0.940395481	0.059605
70	0.973407888	0.026592
72.5	0.990188823	0.009811
75	0.997111674	0.002888
77.5	0.999348757	0.000651
80	0.999892664	0.000107
82.5	0.999987735	1.23E-05
85	0.999999085	9.15E-07
87.5	0.999999958	4.17E-08
90	0.999999999	1.07E-09
92.5	1	1.44E-11
95	1	9.14E-14
97.5	1	0
100	1	0
102.5	1	0

Table 17. Weibull analysis (GROUP IV: ZLC3m)

WEIBULL ZLC3m		
Beta (or shape parameter)	4.259062862	
Alpha (or characteristic life)	32.91002354	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	1.70786E-05	0.999983
5	0.000326957	0.999673
7.5	0.001837154	0.998163
10	0.00624177	0.993758
12.5	0.016065725	0.983934
15	0.03459612	0.965404
17.5	0.065633133	0.934367
20	0.112979406	0.887021
22.5	0.179618552	0.820381
25	0.266635	0.733365
27.5	0.372106603	0.627893
30	0.490410283	0.50959
32.5	0.612486227	0.387514
35	0.727421997	0.272578
37.5	0.825149023	0.174851
40	0.899289379	0.100711
42.5	0.948787229	0.051213
45	0.97742424	0.022576
47.5	0.991540924	0.008459
50	0.997362113	0.002638
52.5	0.999330654	0.000669
55	0.99986508	0.000135
57.5	0.99997894	2.11E-05
60	0.999997522	2.48E-06
62.5	0.999999786	2.14E-07
65	0.999999987	1.31E-08
67.5	0.999999999	5.52E-10
70	1	1.55E-11
72.5	1	2.81E-13
75	1	3.11E-15
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 18. Weibull analysis (GROUP V: PHC24h)

WEIBULL PHC24h		
Beta (or shape parameter)	5.639507	
Alpha (or characteristic life)	9.126531	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	0.000673578	0.999326
5	0.033031015	0.966969
7.5	0.281485776	0.718514
10	0.81257711	0.187423
12.5	0.99724297	0.002757
15	0.99999993	6.97E-08
17.5	1	0
20	1	0
22.5	1	0
25	1	0
27.5	1	0
30	1	0
32.5	1	0
35	1	0
37.5	1	0
40	1	0
42.5	1	0
45	1	0
47.5	1	0
50	1	0
52.5	1	0
55	1	0
57.5	1	0
60	1	0
62.5	1	0
65	1	0
67.5	1	0
70	1	0
72.5	1	0
75	1	0
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 19. Weibull analysis (GROUP VI: PHC3m)

WEIBULL PHC24h		
Beta (or shape parameter)	3.814783	
Alpha (or characteristic life)	7.68897	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	0.013667	0.986333
5	0.176056	0.823944
7.5	0.59725	0.40275
10	0.934463	0.065537
12.5	0.998311	0.001689
15	0.999997	2.77E-06
17.5	1	9.84E-11
20	1	0
22.5	1	0
25	1	0
27.5	1	0
30	1	0
32.5	1	0
35	1	0
37.5	1	0
40	1	0
42.5	1	0
45	1	0
47.5	1	0
50	1	0
52.5	1	0
55	1	0
57.5	1	0
60	1	0
62.5	1	0
65	1	0
67.5	1	0
70	1	0
72.5	1	0
75	1	0
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 20. Weibull analysis (GROUP VII: PLC24h)

WEIBULL PLC24h		
Beta (or shape parameter)	8.018565	
Alpha (or characteristic life)	21.08393	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	3.75593E-08	1
5	9.73965E-06	0.99999
7.5	0.000251472	0.999749
10	0.002522469	0.997478
12.5	0.015002889	0.984997
15	0.063137605	0.936862
17.5	0.201073707	0.798926
20	0.480527599	0.519472
22.5	0.814391837	0.185608
25	0.980158138	0.019842
27.5	0.99977907	0.000221
30	0.999999955	4.52E-08
32.5	1	1.11E-14
35	1	0
37.5	1	0
40	1	0
42.5	1	0
45	1	0
47.5	1	0
50	1	0
52.5	1	0
55	1	0
57.5	1	0
60	1	0
62.5	1	0
65	1	0
67.5	1	0
70	1	0
72.5	1	0
75	1	0
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Table 21. Weibull analysis (GROUP VIII: PLC3m)

WEIBULL PLC3m		
Beta (or shape parameter)	6.005254	
Alpha (or characteristic life)	14.75695	
Microtensile bond strength (MPa)	Reliability	Survival probability
2.5	0.013667	0.986333
5	0.176056	0.823944
7.5	0.59725	0.40275
10	0.934463	0.065537
12.5	0.998311	0.001689
15	0.999997	2.77E-06
17.5	1	9.84E-11
20	1	0
22.5	1	0
25	1	0
27.5	1	0
30	1	0
32.5	1	0
35	1	0
37.5	1	0
40	1	0
42.5	1	0
45	1	0
47.5	1	0
50	1	0
52.5	1	0
55	1	0
57.5	1	0
60	1	0
62.5	1	0
65	1	0
67.5	1	0
70	1	0
72.5	1	0
75	1	0
77.5	1	0
80	1	0
82.5	1	0
85	1	0
87.5	1	0
90	1	0
92.5	1	0
95	1	0
97.5	1	0
100	1	0
102.5	1	0

Figure 1. ln (MPa) Line Fit Plot of Weibull analysis (GROUP I: ZHC24h)

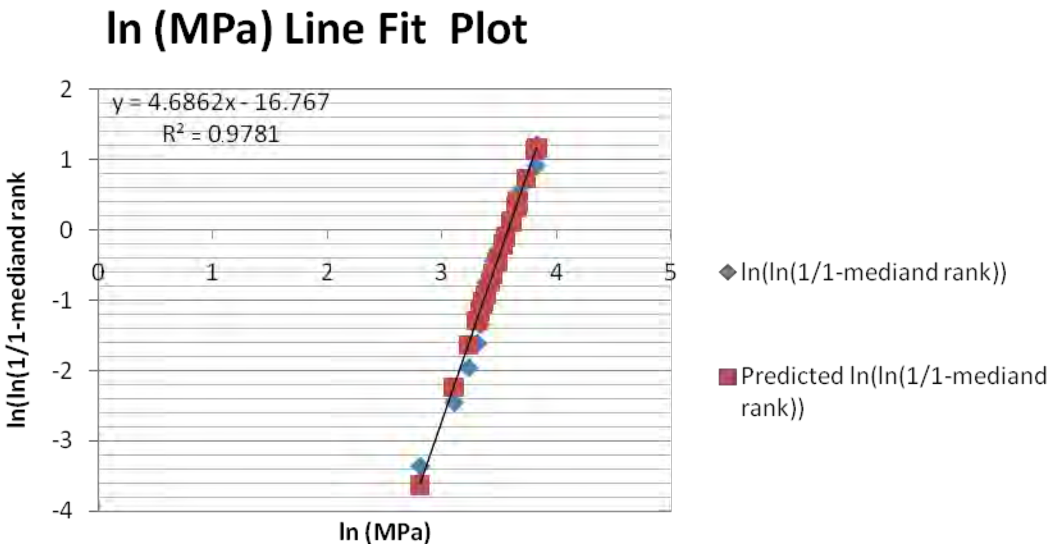


Figure 2. ln (MPa) Line Fit Plot of Weibull analysis (GROUP II: ZHC3m)

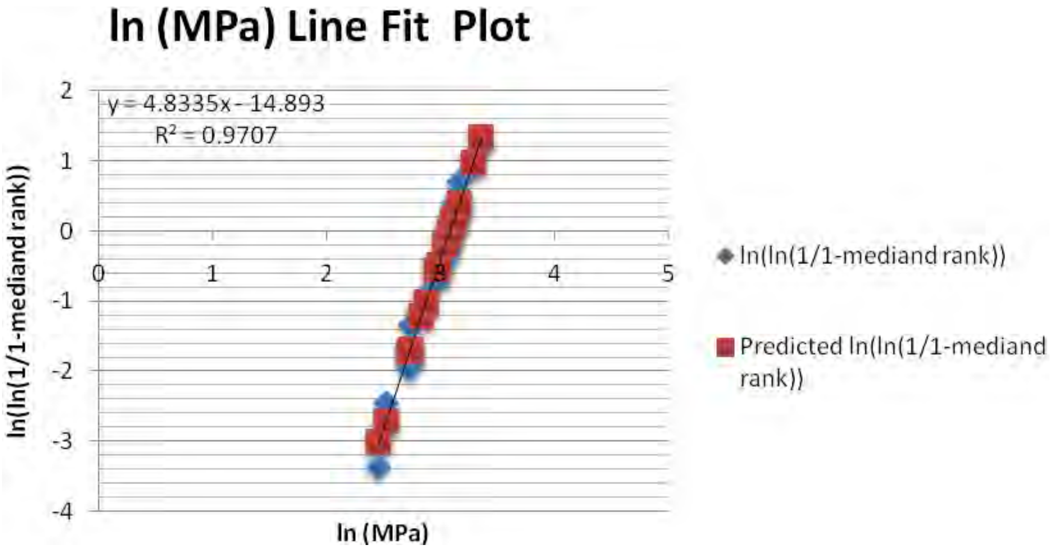


Figure 3. ln (MPa) Line Fit Plot of Weibull analysis (GROUP III: ZLC24h)

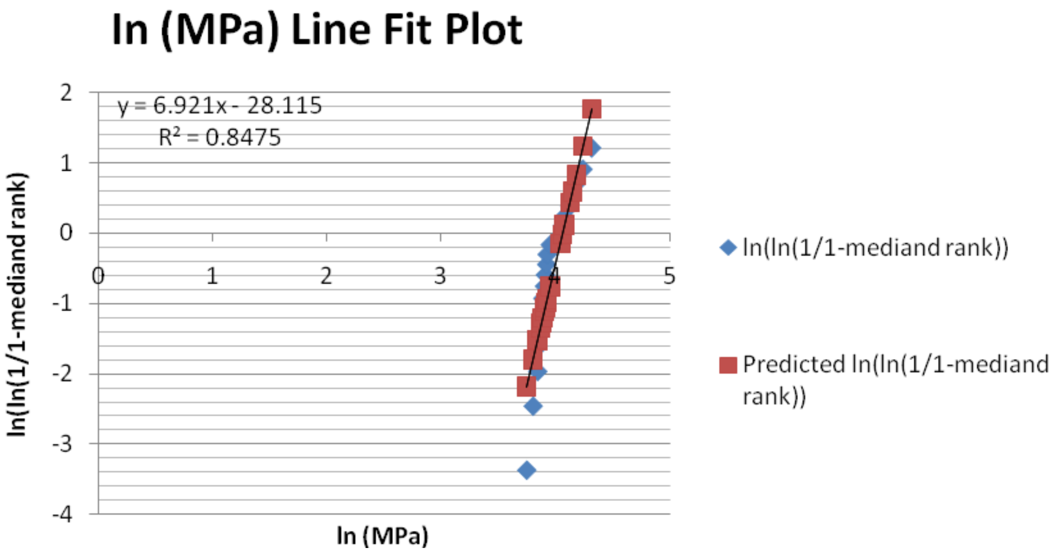


Figure 4. ln (MPa) Line Fit Plot of Weibull analysis (GROUP IV: ZLC3m)

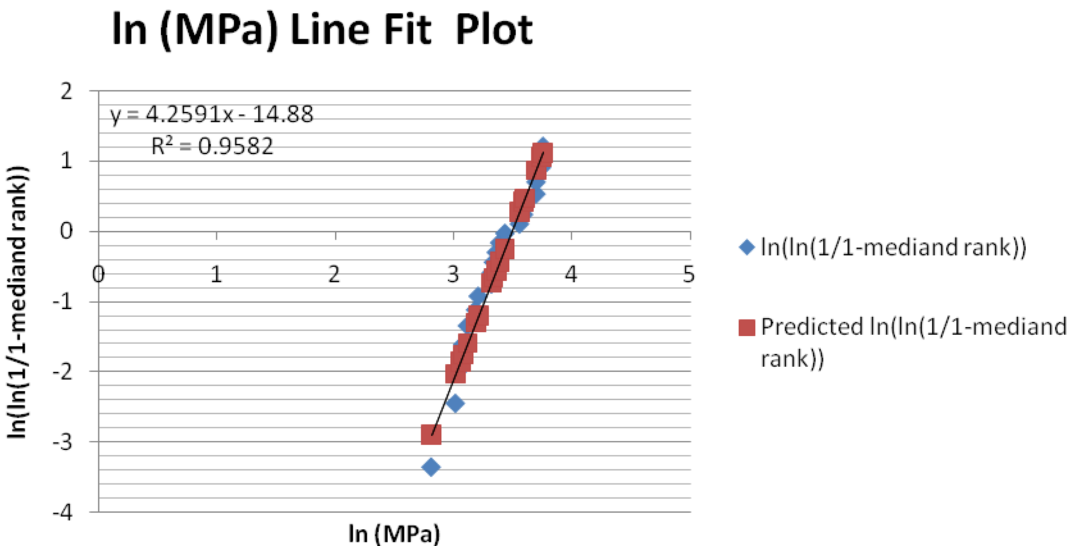


Figure 5. In (MPa) Line Fit Plot of Weibull analysis (GROUP V: PHC24h)

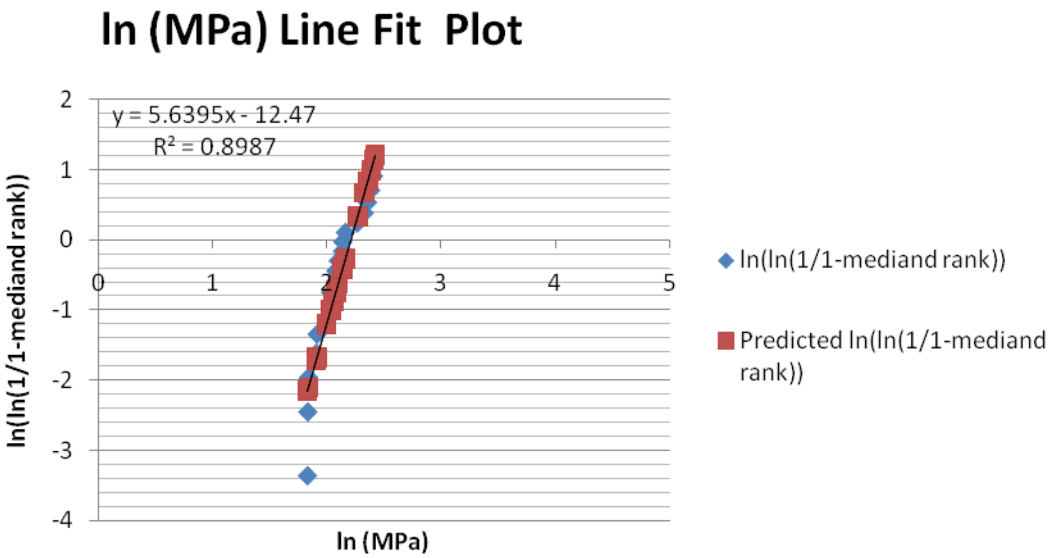


Figure 6. In (MPa) Line Fit Plot of Weibull analysis (GROUP VI: PHC3m)

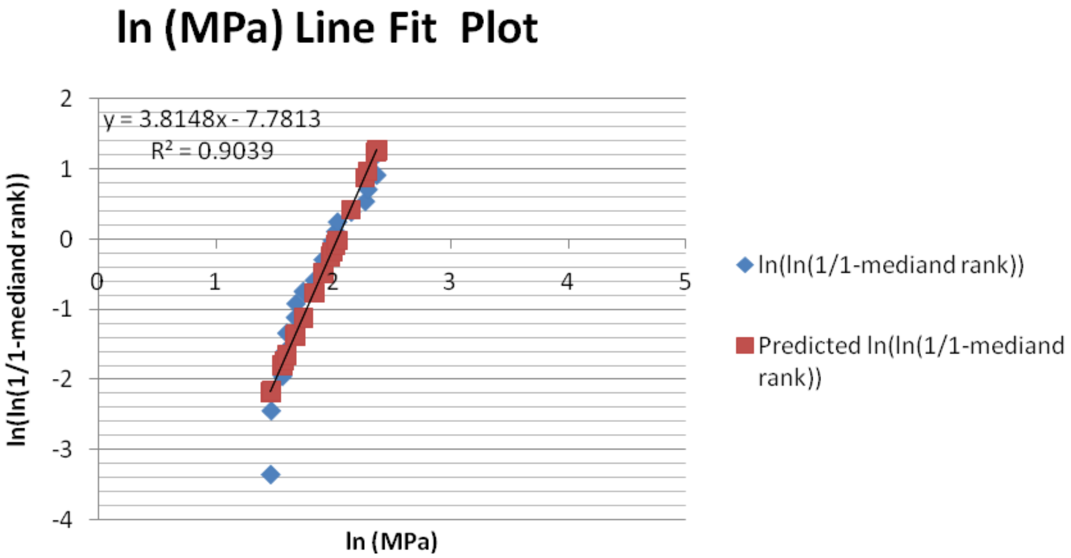


Figure 7. ln (MPa) Line Fit Plot of Weibull analysis (GROUP VII: PLC24h)

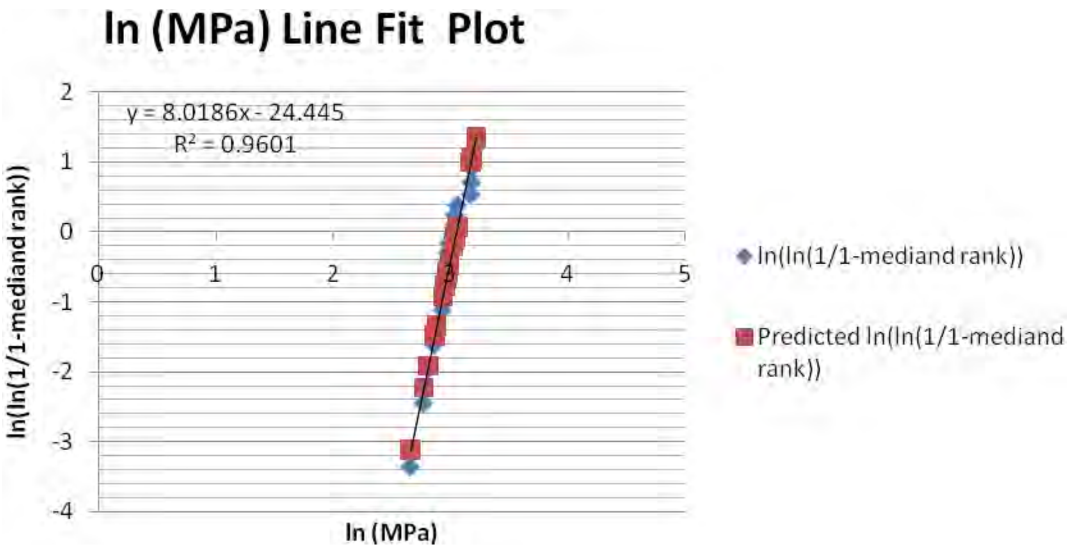
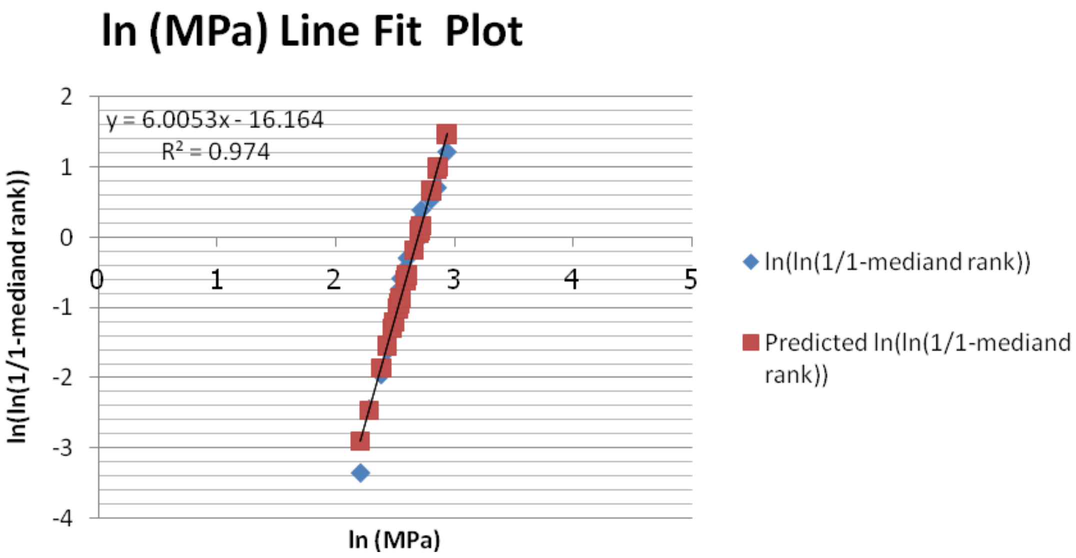


Figure 8. ln (MPa) Line Fit Plot of Weibull analysis (GROUP VIII: PLC3m)



BIOGRAPHY

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