Distribution Statement

Distribution A: Public Release.

The views presented here are those of the author and are not to be construed as official or reflecting the views of the Uniformed Services University of the Health Sciences, the Department of Defense or the U.S. Government.

UNIFORMED SERVICES UNIVERSITY OF THE HEALTH SCIENCES

POSTGRADUATE DENTAL COLLEGE
SOUTHERN REGION OFFICE
2787 WINFIELD SCOTT ROAD, SUITE 220
JBSA FORT SAM HOUSTON, TEXAS 78234-7510
https://www.usuhs.edu/pdc

THESIS APPROVAL PAGE FOR MASTER OF SCIENCE IN ORAL BIOLOGY

Title of Thesis: “Comparing shear bond strength of universal bonding agents and PekkBond to Pekkton: Evaluating the effectiveness of traditional bonding agents and their respective cements against PekkBond when using Pekkton frameworks"

Name of Candidate: CPT Katelyn Sweet
Master of Science Degree
22 May 2023

THESIS/MANUSCRIPT APPROVED: DATE:

MASTERSON.ROBERT.EDWARD.1300590302 Digitally signed by MASTERSON.ROBERT.EDWARD.1300590302
Date: 2023.05.22 16:52:20 -05'00'

Dr. Robert E. Masterson
USUHS Graduate Dental Education, Advanced Education in General Dentistry- 2 year Program, Fort Hood, TX
Program Director

KREIDER.JOHN.KEITH.1152698816 Digitally signed by KREIDER.JOHN.KEITH.1152698816
Date: 2023.05.22 16:57:15 -05'00'

Dr. John K. Kreider
USUHS Graduate Dental Education, Advanced Education in General Dentistry- 2 year Program, Fort Hood, TX
Assistant Program Director

HU.ERIC.CHENHAO.1172052920 Digitally signed by HU.ERIC.CHENHAO.1172052920
Date: 2023.05.23 07:51:39 -05'00'

Dr. Eric C. Hu
USUHS Graduate Dental Education, Advanced Education in General Dentistry- 2 year Program, Fort Hood, TX
Research Mentor

COLTHIRST.PAUL.1062710990 Digitally signed by COLTHIRST.PAUL.1062710990
Date: 2023.06.27 12:53:42 -05'00'

Dr. Paul M. Colthirst
USUHS Graduate Dental Education, Advanced Education in General Dentistry- 2 year Program, Fort Hood, TX
Commander/ Local Dean

Comparing shear bond strength of universal bonding agents and Pekkbond to Pekkton: Evaluating the effectiveness of traditional bonding agents and their respective cements against Pekkbond when using Pekkton frameworks

Katelyn M. Sweet, DDS

CPT DC, USA

AEGD-2yr

Ft. Cavazos, TX

June 2023

The opinions or assertions contained herein are the private ones of the author(s) and are not to be construed as official or reflecting the view of the Department of the Army, Department of Defense DoD, The US Government or the USUHS

Acknowledgments:

1. I would like to thank the following individuals for their support and guidance through the completion of this research project: MAJ Eric Hu, COL Wen Lien, MAJ Robert Masterson, and the Fort Cavazos AEGD2 Lab Support.
2. Successful navigation through the eIRB process for this project was done with the assistance of Dr. Matthew Frazier and Ms. Dawn Beaver, who provided guidance and advice during research design, statistical analysis, and Institutional Review.

Katelyn M. Sweet, DDS
CPT, DC USARMY
Fort Cavazos AEGD-2 Residency

ABSTRACT

The aim of this study was to compare the effect of universal primers and methacrylate-based adhesives on shear bond strengths of resin cement to polyetherketoneketone (PEKK). Forty milled rectangular Pekkton ivory blocks (10x15x10mm) were divided into four groups and mounted into stone pucks. All block surfaces were particle abraded with 110µm AlO2 at 30psi and cleaned with isopropyl alcohol. Each group was assigned a primer/bonding adhesive and resin cement (Group A - Scotchbond Universal Adhesive with RelyX Ultimate; Group B - Monobond Plus and Visio.link with Multilink Hybrid Abutment; Group C - Clearfil Ceramic Primer Plus with Panavia V5; Group D - Pekkbond with Multilink Hybrid Abutment). Each bonding agent was applied to a block surface according to the manufacturer's instructions, and each cement transferred onto the block surface via gelatin pill capsules. Each block received 2 cement capsules, totaling 20 samples per bond group ($n = 20$/group). The stone pucks were then mounted onto a universal testing machine for shear bond strength testing. The values were recorded and compared using the Kruskal-Wallis test and the Steel-Dwass post-hoc comparisons. Group A (7.86 ± 4.11Mpa) and Group B (6.38 ± 3.38Mpa) had the highest shear bond strengths, and these two groups were not statistically different from each other ($p > 0.05$). Group A performed significantly better than Groups C and D ($p < 0.001$). Group C had the lowest overall shear bond strength (1.32 ± 0.72Mpa). This in vitro study found: 1) No statistical difference between the Scotchbond Universal and the visio.link primer group; 2) Universal adhesive tested can be a viable alternative for bonding to Pekkton; and 3) More research is needed to determine the bonding mechanism between universal and conventional primers/adhesives to PEKK.

Key words: *PEKK, high performance polymer, Pekkton, PEKKbond, visio.link, shear bond strength*

Introduction

Polymers have a long history of dental applications. Polymethylmethacrylate (PMMA) has traditionally been used for fabrication of complete dentures and full arch hybrid prostheses due to its low density, ease of use and processing, low cost, and life-like characteristics. However within the polymer group, PMMA falls at the bottom of the performance triangle, making it a standard polymer. In comparison, the high-performance polymers with polyetheretherketone (PEEK) and polyetherketoneketone (PEKK) are at the top of the triangle (Figure 1). PEEK has been studied and found to be an excellent option in dental and medical applications as it is shock-absorbing, has similar characteristics to that of human bone, and compressive strength similar to that of dentin and cortical bone. PEKK is biocompatible and metal free, with excellent stress distribution and higher flexural strength than PEEK. It can be used as a replacement for metal frameworks of partial dentures, complete dentures, custom or prefabricated post and cores, or implant supported fixed complete denture frameworks.[3]

One of the PEKK materials being used for framework fabrication is Pekkton® (Cendre + Metaux SA, Biel/Bienne, Switzerland). This PEKK material is incorporated with titanium dioxide to enhance its mechanical properties and esthetic appearance, and can be milled or pressed into any shape. Additionally, this material can be customized and layered with composite and ceramics.[4] Its low modulus of elasticity helps to reduce and distribute stress, and it is an effective shock absorber.[5] One disadvantage is its opaque white/gray color. Any modifications made to it (such as veneering materials and dyes) need to be bonded on. Surface treatment by chemical or mechanical means has been shown to improve retention to this material.[6] When PEKK is utilized as a full arch implant supported fixed complete denture framework, short titanium abutments rely on chemical bonding for retention. The company that manufactures

Pekkton has formulated a PMMA and composite bonding agent (PEKKbond) to be used when bonding the framework to the abutment. They also recommend another primer (visio.link, Bredent, Chesterfield, United Kingdom) for the same purpose.[7] However the effect on shear bond strength when using universal primers/adhesives as a surface treatment on PEKK has been minimally researched.

As the use of PEKK has gained popularity in dentistry, there have been increasing reports of complications related to titanium abutments debonding from the framework. The objective of this study is to compare and evaluate the shear bond strengths of resin cement to high performance polymer (PEKK) when utilizing various universal primers and bonding adhesives as surface treatments. The null hypothesis is that there will be no significant difference in shear bond strength between the various bonding agents when used on a PEKK framework.

MATERIALS AND METHODS

Forty rectangular specimens with dimensions of 10x15x10 ± .2 mm (ISO 6872) were designed using CAD software (Inlab, Dentsply Sirona, Charlotte, North Carolina), and milled from high performance polymer (PEKK) pucks (Pekkton ivory, Cendres Metaux, Switzerland) using a 5-axis mill (MCX5, Dentsply Sirona, Charlotte, North Carolina). The blocks were then mounted into mounting stone cylinders (Buff stone, Whip Mix, Louisville, Kentucky) (Figure 2). All block surfaces were particle abraded with 110µm AlO2 at 30psi and cleaned with isopropyl alcohol. This method was determined to be the best way to bond a Pekkton framework to titanium bases as per the recommendation from the Pekkton ivory CAD/CAM technology brochure instructions for use. Four test groups were formed based on 4 primers and their associated resin cement (Table 1). Each group contained 10 PEKK blocks, and 2 resin cement samples were placed on each block (Figure 3). The sample sizes comprised 20 specimens per group (n=20), totaling 80 samples. The surface of each PEKK block was primed with either: Group A – a universal adhesive (Scotchbond Universal, 3M, Saint Paul, Minnesota); Group B – universal primer (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) followed by methacrylate based primer (visio.link, Bredent, Chesterfield, United Kingdom); Group C – universal primer (Clearfil Ceramic Primer Plus, Kuraray, Tokyo, Japan) ; or Group D – methacrylate based bond (PEKKbond, Anaxdent, Stuttgart, Germany), according to the manufacturer's instructions. The four different types of cement were injected into gelatin capsules (Torpac®, Fairfield, New Jersey), and placed on the prepared PEKK surface, and cured according to the manufacturer's instructions (Table 1). All 40 cylinders were placed in a water bath for 24 hours. The specimens were mounted on an alignment jig (Ultradent, South Jordan, Utah) and loaded into a universal testing machine (Alliance RT/5, MTS) for notch-edge shear bond strength testing (Figure 4). The force was applied by a knife-edge wedge with 1mm/min crosshead speed and stopped at the point of failure (Figure 5). Bond strength was determined at the point at which bond failure occurred. **Specimen preparation** *Group A:*

Pekkton specimens were particle-abraded with 110um aluminum oxide. The surface was cleaned with isopropyl alcohol wipes. Scotchbond Universal bonding agent was scrubbed onto the surface for 10 seconds, and air thinned. The surface was light cured for 10 seconds per the manufacturer's instructions. Two gel capsules filled with RelyX Ultimate resin cement were placed on the Pekkton surface and light cured for 10 seconds.

Group B:

Pekkton specimens were air abraded with 110um aluminum oxide. The surface was cleaned with isopropyl alcohol wipes. Monobond Plus was applied to the Pekkton surface for 60 seconds. Visio.link was applied and light cured for 90 seconds. Two gel capsules filled with

Multilink Hybrid Abutment cement were placed on the surface, and then the specimen was held in place for 7 minutes.

Group C:

Pekkton specimens were air abraded with 110um aluminum oxide. Surface was cleaned with isopropyl alcohol wipes. A thin layer of Clearfil Ceramic Primer Plus was scrubbed onto surface and entire surface dried with a mild, oil-free air flow. Two gel capsules filled with Panavia V5 were placed onto prepared surface and light cured for 10 seconds.

Group D:

Pekkton specimens were air abraded with 110um aluminum oxide. The surface was cleaned with isopropyl alcohol wipes. A layer of Pekkbond was applied to the surface and light cured for 90 seconds. Two gel capsules were filled with Multilink Hybrid Abutment cement, placed on the surface, and held for 7 minutes.

TABLES

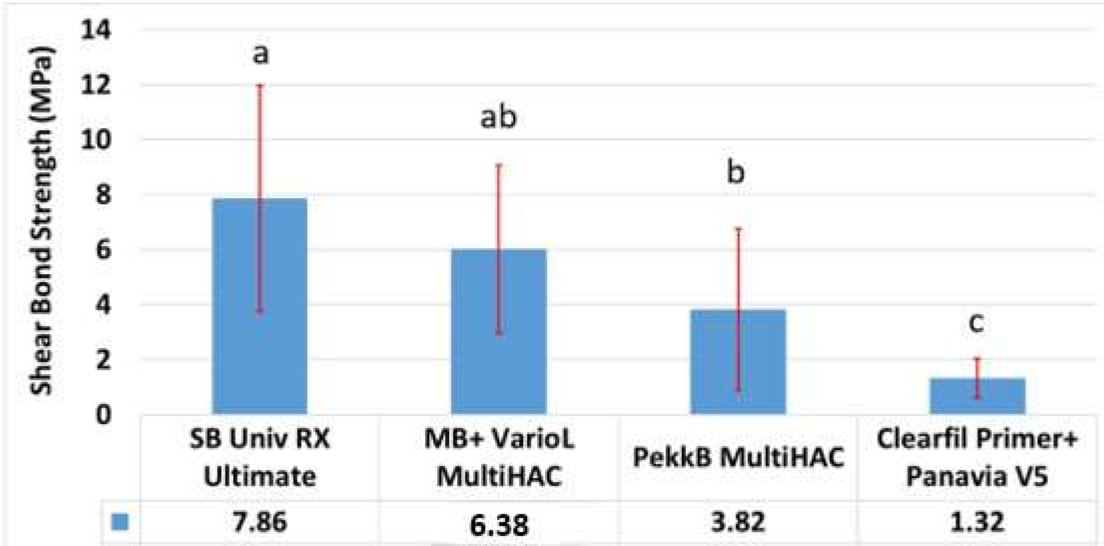
Table 1 Primer/Cement groups and manufacturer's bonding protocol

Group #	Primer	Composition	Indication	Light cure/scrub	Resin Cement
A	Scotchbond Universal	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond Copolymer, filler, ethanol, water, initiator, silane.	Glass ceramics, zirconia, alumina, metals, composites	10 seconds	RelyX Ultimate – light cured
B	visio.link	Methyl methacrylate (MMA), 2-propenoic acid reaction products with pentaerythritol, diphenyl (2, 4, 6,-trimethylbenzoyl)-phosphineoxide.	Composite, PMMA, High-performance polymers	90 seconds	Multilink Hybrid Abutment – self cured (7 min)
	Monobond Plus	Silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate, 10-MDP	Glass and oxide ceramics, metal, composites, fiber-reinforced composite	Vaporize for 60 seconds	
C	Clearfil Ceramic Primer Plus	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate	Ceramic, hybrid ceramics, composite resin, and metal	Apply to surface, air dry completely	Panavia V5 – light cured
D	PEKKBond	Urethandimethacrylate, monomer methacrylate, stabilizers, initiators	Pekkton/PEEK materials and composites	60-120 seconds	Multilink Hybrid Abutment – self cured (7 min)

RESULTS

All data is presented in Tables 2 and 3. Group A had the highest shear bond strength of 7.86 ± 4.11Mpa, and Group C had the lowest shear bond strength at 1.32 ± 0.72Mpa. Group A and Group B were not statistically significant from each other. Group A was significantly higher than Groups C and D. The Shapiro-Wilk test for normality showed that the two groups were not normally distributed and, along with the Leven test, demonstrated that the data sets were nonparametric. Because of this, the Kuskal-Wallis test and the Steel-Dwass post hoc test were conducted (Table 3).

Table 2 Comparison of shear bond strength (MPa) to Pekkton using various bonding primers/agents and their respective cements. Groups with the same case letter are not significantly different (p>0.05).



DISCUSSION

The null hypothesis was rejected as Group A had a statistically significant bond strength increase compared to Groups C and D, and Groups B and C were significantly higher than Group D. In this in vitro study since all four test groups underwent the same pre-primer/adhesive surface treatment on the PEKK blocks, the only independent variables were the primer/adhesive and resin cement used.

All of the shear bond strengths were lower than previous studies of bonding to zirconia utilizing various surface treatments.[8] According to literature by Davidson et al., to adequately fight the contraction forces of polymerization, you ideally want to achieve a bond strength of 20Mpa or more.[9] None of the four groups in this study had acceptable bond strengths of at least 20Mpa. Group A, the highest bond strength, only achieved an average of 7.86Mpa.

In this in vitro study, group A and group B performed the best while group C performed the worst. The manufacturers of Pekkton Ivory recommend using visio.link as the primer of choice and pairing it with Multilink Hybrid Abutment cement. They also promote the use of PEKKbond. PEKKbond is described as creating a strong chemical bond between Pekkton and composites, acrylics and cements and visio.link is described as being ideal for priming the surface of Pekkton to bond with acrylic, composite and resin cements. Because of this recommendation, it would be assumed that the PEKKbond and visio.link testing groups would have performed the best and given strong shear bond results. However, this study showed that no statistically significant difference between Scotchbond and visio.link, and both performed better than Pekkbond.

A study by Lee et al. found that regardless of surface treatment, bonding agents that contained methyl methacrylate (MMA) performed as well as those containing 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP). They attributed this to the fact that MMA-containing bonding materials exhibit a similar effect as MDP materials on roughened PEKK surfaces.[10] This study supports the findings in our current study where the 10-MDP containing universal adhesive (Scotchbond Universal) performed similarly to the MMA-containing primer (visio.link). Unfortunately, we did not get the same result from Clearfil Ceramic Primer Plus, even though this product contains MDP as well. One explanation could be that Scotchbond Universal was used as a bonding agent in this study, whereas Clearfil Ceramic Primer Plus was used as a primer.

According to Song et al., one of the main disadvantages of PEKK was its difficulty bonding due to its low surface energy and resistance to surface modification by chemical treatment.[11] The study by Fuhrmann et al. was considered one of the first to examine bonding to

PEKK, and they determined that the most durable bonding to amorphous and crystalline PEKK and fiber-reinforced PEEK was achieved when applying silica coating, a universal primer, and a resin primer. They also found that no adequate bonding was achieved using dental universal composite resin cement compared to adhesive composite systems.[12] In the current study, no silica coating was used on any of the surfaces. Still, the use of a universal primer (Monobond Plus) and a resin primer (visio.link) did result in one of the higher shear bond strengths, mimicking the results of Fuhrman's study.

More research is needed on the topic of bonding to PEKK. This study had a few limitations that could have affected the results. First, the sample size was limited—this could have been increased to achieve a better distribution. Secondly, there was no control group as there is not yet a bonding system proven clinically superior over the long term. In addition, there was no system in place to accurately standardize the samples to ensure uniform treated surfaces and to ensure that there was homogenous cement contact on the PEKK surface. It would be worth looking into the mode of failure and the exact bonding mechanism between various primers, PEKK, and resin cement in future studies.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions can be drawn: 1) There was no significant difference between the Scotchbond Universal with RelyX Ultimate group and the Monobond and vario.link with Multilink Hybrid Abutment cement groups; 2) The universal adhesive tested (Scotchbond Universal) may be a viable alternative for bonding to Pekkton; and 3) More research is needed to determine the bonding mechanism between universal primers/adhesives to PEKK.

REFERENCES

1. O'Brien, W. J. (1997). Polymers and Polymerization: Denture Base Polymers. In *Dental Materials and their selection* (2nd ed., pp. 79–93). essay, Quintessence Publ.
2. Zafar MS. Prosthodontic Applications of Polymethyl Methacrylate (PMMA): An Update. Polymers (Basel). 2020 Oct 8;12(10):2299. doi: 10.3390/polym12102299. PMID: 33049984; PMCID: PMC7599472.
3. Alqurashi, H., Khurshid, Z., Syed, A. U., Rashid Habib, S., Rokaya, D., & Zafar, M. S. (2021). Polyetherketoneketone (PEKK): An emerging biomaterial for oral implants and dental prostheses. *Journal of Advanced Research*, *28*, 87–95. https://doi.org/10.1016/j.jare.2020.09.004
4. Alsadon, O., Wood, D., Patrick, D., & Pollington, S. (2020). Fatigue behavior and damage modes of high performance poly-ether-ketone-ketone pekk bilayered crowns. *Journal of the Mechanical Behavior of Biomedical Materials*, *110*, 103957. https://doi.org/10.1016/j.jmbbm.2020.103957
5. Han, K.-H., Lee, J.-Y., & Shin, S. (2016). Implant- and tooth-supported fixed prostheses using a high-performance polymer (Pekkton) framework. *The International Journal of Prosthodontics*, *29*(5), 451–454. https://doi.org/10.11607/ijp.4688
6. Fokas, G., Guo, C. Y., & Tsoi, J. K. H. (2019). The effects of surface treatments on tensile bond strength of polyether-ketone-ketone (PEKK) to veneering resin. *Journal of the Mechanical Behavior of Biomedical Materials*, *93*, 1–8. https://doi.org/10.1016/j.jmbbm.2019.01.015
7. Cendres + Metaux. (2021). *Instructions for Use Pekkton ivory Milling blank*.

8. Sharafeddin, F., & Shoale, S. Effects of universal and conventional MDP primers on the shear bond strength of zirconia ceramic and nanofilled composite resin. *Journal of Dentistry*. 2018, *19*(1), 48.
9. Davidson, A.J., de Gee, A.J., Feilzer, A. The competition between the composite-dentin bond strength and the polymerization contraction stress. Journal of Dental Research. 1984 Dec; 63(12): 1396-1399.
10. Lee KS, Shin MS, Lee JY, Ryu JJ, Shin SW. Shear bond strength of composite resin to high performance polymer PEKK according to surface treatments and bonding materials. J Adv Prosthodont. 2017 Oct;9(5):350 -357.
11. Song C-H, Choi J-W, Jeon Y-C, Jeong C-M, Lee S-H, Kang E-S, Yun M-J, Huh J-B. Comparison of the Microtensile Bond Strength of a Polyetherketoneketone (PEKK) Tooth Post Cemented with Various Surface Treatments and Various Resin Cements. *Materials*. 2018; 11(6):916. https://doi.org/10.3390/ma11060916
12. Fuhrmann, Gyde, Steiner, Martin, Freitag-Wolf, Sandra, Kern, Matthias. (2014) Resin bonding to three types of polyaryletherketones (PAEKs)—Durability and influence of surface conditioning, Dental Materials. Volume 30, Issue 3.