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Praca na stopień doktora nauk medycznych i nauk o zdrowiu

Wpływ różnych cementów samoadhezyjnych na siłę wiązania pomiędzy wybranymi ceramikami dentystycznymi przetwarzanymi w technologii CAD/CAM a ludzką zębiną

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I. STRESZCZENIE

WSTĘP

Odbudowy pełnoceramiczne, ze względu na możliwość uzyskania doskonałych efektów estetycznych, stanowią popularne rozwiązanie stosowane we współczesnej protetyce stomatologicznej. W szczególności, dzięki rozwojowi technologii wspomaganego komputerowo projektowania i wytwarzania (CAD/CAM), usprawniono proces wykonywania precyzyjnych ceramicznych prac protetycznych. Problemem, który nadal wzbudza zainteresowanie wielu grup badaczy jest konieczność poprawy wytrzymałości połączenia pomiędzy rekonstrukcjami ceramicznymi wytwarzanymi w technologii CAD/CAM, a tkankami twardymi zęba. Jej osiągnięcie jest możliwe dzięki zastosowaniu odpowiednich cementów dentystycznych.

Współczesne cementy na bazie żywic, oprócz właściciwości samoadhezyjnych, cechują się zdolnością samotrawienia łączonych powierzchni. Uważa się, że osiągnięte dzięki ich zastosowaniu uproszczone procedury aplikacji powinny skrócić czas zabiegu oraz doprowadzić do zwiększenia siły wiązania poprzez zminimalizowanie błędu operatora. Dzięki zdolności wiązania zarówno z odbudową ceramiczną jak i tkankami zęba, cementy na bazie żywic adhezyjnych mogą wzmocnić łączone struktury, zmniejszyć mikro przeciek i przebarwienie brzeżne na styku odbudowa-ząb oraz zmniejszyć wrażliwość po zabiegową.

Wytrzymałość połączenia cementu z ceramiką i tkankami zęba zależy zarówno od rodzaju zastosowanego cementu, jak również sposobu przygotowania powierzchni cementowanych zęba wraz z uzupełnieniem protetycznym. Istotnym parametrem bezpośrednio wpływającym na jakość odbudowy oraz wzmocnienie cementowanych struktur jest ich podatność na proces starzenia. Wpływ na degradację materiałów na bazie żywic w środowisku jamy ustnej mają bowiem czynniki fizyczne oraz chemiczne, takie jak zmiany temperatury, wilgotności oraz pH.

CEL PRACY

Ocena siły wiązania między wybranymi ceramikami dentystycznymi przetwarzanymi w technologii CAD/CAM a ludzką zębiną, połączonymi za pomocą samoadhezyjnych, samotrawiących cementów na bazie żywic oraz określenie wpływu procesu sztucznego starzenia na siłę tego wiązania.

Przeprowadzenie przeglądu systematycznego współczesnego piśmiennictwa podsumowującego najbardziej aktualne dowody dotyczące stosowania różnych metod modyfikacji powierzchni ceramik dentystycznych zwiększającą jej siłę wiązania do twardych tkanek zębów.

MATERIAŁY I METODY

Wybrano trzy samoadhezyjne, samotrawiące cementy (Panavia SA, RelyX U200, Maxcem Elite) oraz jeden popularny konwencjonalny cement adhezyjny traktowany jako grupa kontrolna (Panavia V5) do cementowania trzech powszechnie stosowanych materiałów ceramicznych przetwarzanych w technologii CAD/CAM (IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD). Badane cementy zostały użyte do połączenia ceramicznych cylindrów zfragmentami ludzkich zębów trzonowych o odsłoniętej zębinie. Do przeprowadzenia badań uzyskano zgodę Komisji Bioetycznej No. KB-37/2018. Przygotowano 288 próbek, po 12 próbek dla każdej kombinacji ceramiki i cementu, w dwóch grupach: poddanych i wytrzymałości na ścinanie po 24 godzinach inkubacji w cieplarce, natomiast druga grupa przed oceną wytrzymałości na ścinanie została poddana procesowi sztucznego starzenia w termocyklerze. Parametry przyspieszonego starzenia obejmowały 2000 cykli w temperaturze od 5°C do 55°C, z zanurzeniem próbek przez 40 sekund oraz czasem przeniesienia między pojemnikami wynoszącym 15 sekund. Wytrzymałość wiązania ceramiki z zębiną została oceniona za pomocą testu wytrzymałości na ścinanie przeprowadzonego za pomocą uniwersalnej maszyny testującej zgodnie z normą PN-EN ISO 29022: 2013-10. Dodatkowo, po wykonaniu testu wytrzymałości na ścinanie, za pomocą mikroskopu optycznego Zeiss Axio Lab A1 przeprowadzono analizę powstałych przełomów. W celu oceny istotności różnic pomiędzy wytrzymałością wiązania badanych cementów wykonano szczegółową analizę statystyczną uzyskanych wyników.

Przegląd systematyczny został przeprowadzony zgodnie z wytycznymi PRISMA służących dogromadzenia i raportowania danych. Przeszukano bazy danych Web of Science, Scopus i MEDLINE w celu zidentyfikowania odpowiednich artykułów opublikowanych w języku angielskim w okresie od 1 stycznia 2010 roku do 1 stycznia 2020 roku. Przeszukano literaturę, łącząc każde z następujących słów kluczowych w języku angielskim: (1) ceramika dentystyczna, (2) cement dentystyczny na bazie żywic, (3) cement dentystyczny (4) zęby, z każdym z następujących słów kluczowych: (A) modyfikacja powierzchni, (B) obróbka powierzchni, (C) kondycjonowanie powierzchni oraz z każdym z następujących dodatkowych słów kluczowych: (a) siła wiązania, (b) trwałość i (c) przyczepność. Bazy danych zostały uzupełnione o ręczne przeszukanie literatury wybranych artykułów mające na celu identyfikację potencjalnie odpowiednich badań.

WYNIKI

Badanie wykazało, że rodzaj zastosowanej ceramiki i cementu bezpośrednio wpływa na siłę wiązania. Najwyższą wytrzymałość na ścinanie uzyskano dla próbek łączonych za pomocą kontrolnego cementu Panavia V5, niższą dla Panavia SA i Maxcem Elite, a najniższą dla RelyX U200. Statystycznie najczęściej dochodziło do złamań adhezyjnych między ludzką zębiną a cementem. Ponadto, termocykling istotnie zmniejszył siłę wiązania cementów samoadhezyjnych, samotrawiących.

W przeglądzie systematycznym do którego ostatecznie włączono 19 artykułów w większości badań jest statystycznie istotna różnica w sile wiązania między próbkami poddanymi różnym metodom modyfikacji powierzchni ceramiki w stosunku do próbek niemodyfikowanych. Tylko dwa zakwalifikowane do przeglądu badania nie wykazały statystycznie istotnych różnic pomiędzy grupami eksperymentalnymi. Jednakże, w tych dwóch badaniach nie wykonano niemodyfikowanej grupy kontrolnej, a testowano wyłącznie rożne metody modyfikacji powierzchni łączenia ceramiki.

WNIOSKI

1. Konwencjonalny cement Panavia V5 wykazał znacznie wyższą siłę wiązania w porównaniu do samoadhezyjnych, samotrawiących cementów, zarówno po przyspieszonym starzeniu termicznym jak i bez jego zastosowania.

2. Niezależnie od badanego cementu najniższą siłę wiązania wśród badanej ceramiki uzyskano dla IPS e.max ZirCAD.

3. Właściwy dobór cementu do ceramiki ma kluczowe znaczenie dla trwałości połączenia, ponieważ różnice w wiązaniu dla badanych kombinacji wybranych cementów i ceramik były statystycznie istotne.

4. Statystyczne porównanie próbek poddanych przyśpieszonemu starzeniu z próbkami badanymi po 24 godzinach od cementowania uwidoczniło znaczne spadki sił wiązania na skutek termocyklingu, szczególnie dla cementów samoadhezyjnych, samotrawiących.

5. W oparciu o przeanalizowane wyniki, połączenie mechanicznej i chemicznej modyfikacji powierzchni ceramiki daje najskuteczniejszy sposób zwiększania siły wiązania pomiędzy ceramiką a tkankami twardymi zębów.

6. Przegląd dostępnej literatury podkreśla potrzebę standaryzacji metodyki modyfikacji powierzchni dla przyszłych badań ze względu na użycie różnych materiałów, protokołów i testów przez badaczy ponieważ, porównanie danych jest problematyczne ze względu na brak jednorodności.

7. Ponadto standaryzowane protokoły powinny próbować odtworzyć stan kliniczny poprzez: stosowanie różnych metod testowania, w tym testów zmęczeniowych, a także poprzez sztuczne starzenie próbek.

II. ABSTRACT

INTRODUCTION

It has becoming increasing popular for all-ceramic dental restoration due to dynamic developing technologies of manufacturing, especially computer aided design and manufacturing (CAD/CAM) and ceramics itself. Besides the known positives of all-ceramic materials, such as high biocompatibility, resistance to difficult physico-chemical conditions, aesthetics, these materials are sensitive to cracking and chipping. It is said that the quality of the bond between the ceramics and the hard tissues of the tooth significantly improves the mechanical properties of restoration. Moreover, durable bonding between tooth and reconstruction is important for avoiding detachment of restorations and for preventing microleakage, secondary dental caries, and tooth fractures.

Many modern resin based cements are characterized by self-adhesive and self-etch properties. It is well known that reliable bonding could be achieved by acid-etching the glass based ceramic surface and applying a silane coupling agent. Procedures takes many sensitive steps and simplifying the application procedures, should resolved in operator error limitation.

AIM OF THE STUDY

Assessment of the bond strength of self-adhesive self-etching cements based on resins used for luting selected dental ceramics processed in CAD/CAM technology to human dentin and evaluation of the influence of thermocycling on the bond strength.

Conducting a systematic review of modern literature summarizing the most recent evidence on the use of various surface modification methods of dental ceramics increasing its bond strength to hard dental tissues.

MATERIALS AND METHODS

Three self-adhesive, self-etching cements (Panavia SA, RelyX U200, Maxcem Elite) and one conventional adhesive resin-based cement as a control group (Panavia V5) were selected for the cementation of three commonly used CAD/CAM ceramics (IPS Empress CAD, IPS e. max CAD, IPS e.max ZirCAD). The tested cements were bond to ceramic cylinders and surface of human molars dentin. For this study prepared 288 samples, 12 samples for each combination of ceramic and cement. The first group of 144 samples was shear bond tested after 24 hours in incubator, while the second group was subjected to artificial aging in a thermocycler

before shearing. Shear bond strength tests were carried out with a universal testing machine in accordance with PN-EN ISO 29022: 2013-10. Additionally, after performing the shear bond strength test, an analysis of the fractures was carried out using the Zeiss Axio Lab A1 optical microscope. In order to assess the significance of differences between the bond strength of the tested cements, a statistical analysis of the obtained results was performed.

The systematic review was conducted in accordance with the PRISMA guidelines for data collection and reporting. The Web of Science, Scopus and MEDLINE databases were searched for relevant articles published in English between January 1, 2010 and January 1, 2020. The literature searched for each of the following English keywords: (1) dental ceramic, (2) dental resin cement, (3) dental luting cement, and (4) teeth; with each of the following keywords: (A) surface modification, (B) surface treatment, and (C) surface conditioning; and with each of the following additional keywords: (a) bond strength, (b) durability, and (c) adhesion. The database search was supplemented with a manual search of the bibliographic references of the retrieved articles aimed at the identification of potentially relevant papers.

RESULTS

The study showed that the combination of ceramics and cements directly affects the bond strength. The highest bond strength was observed in the Panavia V5 control cement, lower in Panavia SA and Maxcem Elite, and the lowest in the RelyX U200. Statistically, the adhesion fractures between human dentin and cement were the most common. Moreover, thermocycling significantly decreased the bond strength of self-adhesive, self-etching cements.

In the systematic review, which finally included 19 articles, most studies show a statistically significant difference in the bond strength between samples subjected to various surface modification methods of ceramic compared to the unmodified samples. Only two studies included in the review showed no statistically significant differences between the experimental groups. However, in these two studies, no untreated control group was performed, only different methods of modifying the ceramics bonding surface were tested.

CONCLUSIONS

1. Conventional Panavia V5 cement showed a much higher bond strength compared to self-adhesive, self-etching cements after accelerated thermal aging and before.

2. Regardless of the tested cement, the lowest bond strength among the tested ceramics was obtained for IPS e.max ZirCAD.

3. Proper selection of cement to ceramics is of key importance, because the differences in the setting of the tested combinations of selected cements and ceramics were statistically significant.

4. A statistical comparison of the samples subjected to accelerated aging with the samples tested 24 hours after cementing revealed a significant decrease in the bonding forces for self-adhesive, self-etching cements.

5. Based on the analyzed results of the systematic review, the combination of mechanical and chemical modification of the ceramic surface provides the most effective way to increase the bond strength between the ceramic and the hard tissues of the teeth.

6. Review of the available literature emphasizes the need to standardize the surface modification methodology for future research due to the use of different materials, protocols and tests by researchers, data comparison is quite difficult.

7. In addition, standardized protocols should attempt to reproduce the clinical state by: using a variety of testing methods, including fatigue testing, and by samples artificial aging.

III. WYKAZ UŻYWANYCH SKRÓTÓW

CAD/CAM - (ang. Computer Aided Design/Computer Aided Manufacturing) - Komputerowe Wspomaganie Projektowania/ Komputerowe Wspomaganie Wytwarzania

PRISMA - (ang. Preferred Reporting Items for Systematic Reviews and Meta-Analyses) - powstała w oparciu o dowody naukowe deklaracja zawierająca minimalny zestaw elementów niezbędnych do prawidłowego raportowania przeglądów systematycznych i meta-analiz oceniających korzyści i szkody wynikające z opieki zdrowotnej.

GRADE – (ang. Grading of Recommendations Assessment, Development and Evaluation) stanowi przejrzystą i uporządkowaną metodykę opracowywania i przedstawiania podsumowań dowodów naukowych na potrzeby przygotowania przeglądów systematycznych.

PICOS - (ang. Population, Intervention, Comparison, Outcomes and Study Design) Inclusion and Exclusion Criteria- model ten pomaga podzielić pytanie kliniczne na słowa kluczowe w celu wyszukiwania informacji. PICOS jest akronimem od słów: Pacjent/Problem, Interwencja, Porównanie, Wynik, Rodzaj Badania.

IV. WSTĘP

Dynamiczny postęp technologiczny w dziedzinie materiałów stomatologicznych wykorzystywanych do produkcji stałych rekonstrukcji protetycznych, jak również rozwój metod wspomaganego komputerowo projektowania i wytwarzania (CAD/CAM) doprowadziły do powstania uzupełnień niemal idealnie dopasowanych do naturalnego uzębienia, zarówno pod względem funkcjonalnym, jak i estetycznym [1,2]. Materiały do odbudowy uszkodzonych tkanek zębów powszechnie stosowane w technologii CAD/CAM to ceramika szklana (skaleniowa, na bazie miki, wzmocniona leucytem lub dwukrzemianem litu) oraz ceramiki polikrystaliczne, takie jak ceramika na bazie tlenku glinu i cyrkonu. Wymienione materiały ceramiczne cechuje wysoka biokompatybilność oraz obojętność chemiczna. Jednak, uwzględniając znaczne siły generowane podczas fizjologicznego funkcjonowania uzębienia oraz wymagające warunki panujące w środowisku jamy ustnej, uzupełnienia ceramicze winny cechować się odpowiednimi właściwościami mechanicznymi. Z tego powodu, w ostatnim czasie podjęto wiele wysiłków w celu opracowania materiałów, które cechuje nie tylko doskonała estetyka ale także wysoka wytrzymałość [2-4].

Oprócz właściwości samego materiału do odbudowy, na trwałość wykonanego uzupełnienia protetycznego w znacznym stopniu wpływa zastosowanie do osadzania odbudowy odpowiedniego cementu, zapewniającego silne wiązanie pomiędzy ceramiką i zębiną. Współcześnie w protetyce stomatologicznej powszechnie stosuje się cementy na bazie żywic, które można podzielić na trzy grupy: wymagające osobnych systemów adhezyjnych, konwencjonalne cementy samoadhezyjne oraz samoadhezyjne, samotrawiące [3-7]. Znaczący postęp technologiczny dokonany w celu opracowania cementów samotrawiących, samoadhezyjnych umożliwił osiągnięcie znacznego skrócenia czasu zabiegu [1,5,7].

Proces przyspieszonego postarzania ma za zadanie odzwierciedlić zachowanie badanego materiału w czasie. Pomaga przybliżyć rzeczywiste zachowanie i właściwości cementu i między innymi dlatego obecnie jest niezbędna podczas badań in vitro. Jednakże, jedna optymalna metoda przyspieszonego starzenia doskonale naśladująca warunki panujące w jamie ustnej do tej pory nie została opracowana. Opisano różne podejścia i protokoły w oparciu o starzenie termiczne starzenie termomechaniczne, obciążenie dynamiczne lub przetrzymywanie w wodzie o stałej temperaturze [7–22].W poniższym badaniu zastosowany został termocykling jako jedna z najczęstszych metod sztucznego starzenia materiałów stomatologicznych [5,14].

Większość badań przeprowadzana jest z zakresem temperatur 5°C–55°C zdefiniowane w ISO/TS 11405, ale badania różnią się czasem przebywania w wodzie oraz liczbą wykonanych cykli [5,6,14,16].

Zagadnienie związane z siłą łączenia się cementów samoadhezyjnych samotrawiących nowej generacji do tkanek zębów i ceramik dentystycznych spotyka się z zainteresowaniem wielu grup badaczy [9,10-17]. Niestety ze względu na znaczne różnice w metodologii badawczej dane z tych eksperymentów są trudno porównywalne, co uniemożliwia wyciągnięcie bardzo szczegółowych wniosków. Mnogość czynników wpływających na wyniki badań wytrzymałości połączeń, w tym właściwości zębiny, właściwości ceramiki i sposób ich przygotowania, oraz parametry związane z badaniem, wymagają aby projektowane badania przeprowadzać w sposób standaryzowany [22,27].

V. ZAŁOŻENIA I CEL PRACY

W szczególności, omawiana praca miała na celu realizację następujących zadań badawczych:

1) Określenie, który z wybranych cementów pozwala uzyskać największą siłę wiązania odbudowy ceramicznej do ludzkiej zębiny i czy jakość połączenia zależy od zastosowanej ceramiki CAD/CAM.

2) Określenie, który z wybranych cementów pozwala uzyskać największą siłę wiązania dla próbek poddanych procesowi sztucznego starzenia.

3) Porównanie siły wiązania wybranych cementów przed i po procesie sztucznego starzenia i ustalenie, który z wybranych cementów najlepiej zachowuje swoje właściwości łączące po przeprowadzeniu procesu sztucznego starzenia.

4) Przeprowadzenie mikroskopowej oceny rodzajów przełomów powstałych po wykonaniu testu wytrzymałości na ścinanie.

5) Przeprowadzenie przeglądu systematycznego współczesnego piśmiennictwa podsumowującego najbardziej aktualne dowody dotyczące stosowania różnych metod modyfikacji powierzchni ceramik dentystycznych.

VI. MATERIAŁ I METODY

Przygotowanie próbek

Do badania zastosowano cztery cementy na bazie żywic: jeden konwencjonalny, jako grupa kontrolna (Panavia V5) oraz trzy cementy samotrawiące, samoadhezyjne (Panavia SA, RelyX U200, Maxcem Elite) jako grupy badane. Wybrano trzy różne rodzaje ceramik dentystycznych przetwarzanych w technologii CAD/CAM: IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD. Dla każdego połączenia cementu i ceramiki przygotowano po 12 próbek. Zgodnie z PN-EN Norma ISO 29022:2013-10, zaprojektowano w sumie 288 cylindrów ceramicznych o średnicy 2,38 mm i wysokości 5 mm oraz wyfrezowano je w technologii CAD/CAM przy użyciu Sirona System Cerec inLAB SW 19.0 (Sirona, Nowy Jork, NY, USA). Cylindry zostały zacementowane do ludzkiej zębiny pociętej w plastry, uzyskanej z 67 świeżo usuniętych, wolnych od próchnicy ludzkich zębów trzonowych (badanie zostało zatwierdzone przez Komisję Bioetyczną Uniwersytetu Medycznego we Wrocławiu, nr KB-37/2018). Do przygotowania plastrów zębiny użyto systemu PetroThin z tarczą diamentową i chłodzeniem wodnym (Buehler, Lake Bluff, IL, USA). Do preparacji wybrano zębinę koronową tak aby uzyskać plastry zębiny o grubości 3mm. Przed zacementowaniem cylindra ceramicznego każdą przygotowaną próbkę zębiny oszlifowano papierem karborundowym o granulacji P 400 (Luna, Berno, Szwajcaria).

Cementowanie próbek

Cementowanie cylindrów ceramicznych na ludzkiej zębinie przeprowadzono zgodnie z wytycznymi producenta. W przypadku cementów samotrawiących, samoadhezyjnych przeprowadzono modyfikację wyłącznie powierzchni ceramicznej. IPS Empress CAD i IPS e.max CAD została wytrawiona 9% kwasem fluorowodorowym (3M ESPE, Maplewood, MN, USA) przez 1 min. Powierzchnię ceramiki IPS e.max ZirCAD poddano obróbce wstępnej przy użyciu CoJet System (3M ESPE, MN, USA). Konwencjonalny cement Panavia V5 wymagał dodatkowej modyfikacja powierzchni zębiny 37% kwasem ortofosforowym (3M ESPE, Maplewood, Minnesota, USA). Cementowanie ceramiki na zębinie przeprowadzono ze stałą siłą 10 N pod kontrolą dynamometru FB(C) (Axis, Gdańsk, Polska). Polimeryzację przeprowadzono przy użyciu lampy LED Elipar (3M ESPE, Maplewood, MN, USA) przez 20 sekund. 288 próbek podzielono na dwie grupy: poddaną i niepoddaną termocyklingowi.

Obie grupy przed badaniem siły wiązania metodą testu wytrzymałości na ścinanie przechowywano w wodzie destylowanej w temperaturze 37°C przez 24 godziny. Drugą grupę 144 próbek przed planowanym ścinaniem poddano symulowanemu sztucznemu starzeniu. W Tabeli 1 zawarto szczegółowe informacje o użytych materiałach.

Tabela 1. Materiały ceramiczne i cementy wykorzystane w badaniu.

Nazwa	**Rodzaj**	**Producent**
CEMENTY		
RelyX U200 A1	Samo-adhezyjny, samo-trawiący	3M ESPE (Maplewood, Minnesota, USA)
Maxcem Elite A1	Samo-adhezyjny, samo-trawiący	Kerr (Brea, California, USA)
Panavia SA Cement Universal A1	Samo-adhezyjny, samo-trawiący	Kuraray Noritake (Tokyo, Japan)
Panavia V5 A1	Samo-adhezyjny	Kuraray Noritake (Tokyo, Japan)
CERAMIKA		
IPS Empress CAD HT A1	Ceramika leucytowa	IvoclarVivadent (Schaan, Liechtenstein)
IPS e.Max CAD HT A1	Dwukrzemian litu	IvoclarVivadent (Schaan, Liechtenstein)
IPS e.Max ZirCAD	Dwutlenek cyrkonu	IvoclarVivadent (Schaan, Liechtenstein)

Przyśpieszone sztuczne starzenie

Proces sztucznego starzenia próbek przeprowadzono na maszynie Thermocycler THE-1100 (SD Mechatronik, Monachium, Niemcy). Próbki poddano termocyklingowi obejmującemu 2000 cykli w temperaturach od 5°C do 55°C z czasem przebywania 40 sekund i czasem transferu 15 sekund. Bezpośrednio po zakończeniu przyspieszonego starzenia próbki poddano testowi wytrzymałości na ścinanie.

Ocena wytrzymałości na ścinanie

Badania wytrzymałości wiązania na ścinanie przeprowadzono za pomocą uniwersalnej maszyny wytrzymałościowej (Thumler, Norymberga, Niemcy). Zgodnie z normą PN-EN ISO 29022:2013-10 nóż ścinający poruszał się z prędkością 1 mm/min.

Ocena mikroskopowa złamań

Rodzaj przełomów powstałych na skutek ścinania próbek obserwowano za pomocą mikroskopu świetlnego (Axio Lab. A1 MAT, Zeiss, Oberkochen, Niemcy) z obiektywem o powiększeniu ×5. Określono rodzaj złamania: złamanie adhezyjne pomiędzy ceramiką i cementem, złamanie adhezyjne pomiędzy zębiną i cementem, złamanie kohezyjne w cemencie, złamanie kohezyjne w ceramice, złamanie kohezyjne w zębinie lub złamanie mieszane.

Metodyka przeglądu systematycznego

Przegląd systematyczny literatury został dokonany zgodnie z wytycznymi PRISMA służącymi do gromadzenia i przetwarzania danych z piśmiennictwa [23,24]. Przegląd przeprowadzono, próbując odpowiedzieć na następujące pytania:

1) Czy modyfikacje powierzchni ceramiki znacząco wpływa na jej siłę wiązania do tkanek twardych zębów?

2) Która metoda modyfikacji powierzchni może najskuteczniej zwiększyć siłę wiązania ceramiki do tkanek zębów?

3) Jakie są inne czynniki (np. sztuczne starzenie), które znacząco wpływają na siłę wiązania cementów dentystycznych?

Przeszukano bazy danych Web of Science, Scopus i MEDLINE w celu zidentyfikowania odpowiednich artykułów opublikowanych w języku angielskim w okresie od 1 stycznia 2010 roku do 1 stycznia 2020 roku. Przeszukano literaturę, łącząc każde z następujących słów kluczowych w języku angielskim: (1) ceramika dentystyczna, (2) cement dentystyczny na bazie żywic, (3) cement dentystyczny (4) zęby, z każdym z następujących słów kluczowych: (A) modyfikacja powierzchni, (B) obróbka powierzchni, (C) kondycjonowanie powierzchni oraz z każdym z następujących dodatkowych słów kluczowych: (a) siła wiązania, (b) trwałość i (c) przyczepność. Bazy danych zostały uzupełnione o ręczne przeszukanie literatury wybranych artykułów mające na celu identyfikację potencjalnie odpowiednich badań [28,29]. Po usunięciu duplikatów ze wstępnie wybranych prac z baz danych, przejrzano tytuły oraz streszczenia wstępnie zakwalifikowanych artykułów na podstawie kryteriów włączenia i wykluczenia.

Aby uzyskać ostateczną ocenę kwalifikowalności, dwóch współautorów badania dokonało niezależnej oceny pełnego tekstu wybranych badań, które zostały następnie poddane krytycznej rewizji przez innego współautora.

VII. ANALIZA STATYSTYCZNA I WYNIKI

Porównanie wytrzymałości na ścinanie próbek poddanych i niepoddanych sztucznemu starzeniu

Przeprowadzono analizę statystyczną wyników badania z 2020 oraz z 2021 według tych samych norm [25,26].

Zastosowany został sparowany test *t* dla próbek, aby porównać siłę wiązania czterech rodzajów cementu (Panavia SA, Panavia V5, RelyX U200, Maxcem) testowanych w dwóch warunkach eksperymentalnych (z termocyklingiem lub bez). Analizy przeprowadzono oddzielnie dla trzech ceramik (IPS ZirCAD, IPS Empress, IPS e.max). Aby oszacować wielkość efektu obserwowanych różnic, obliczony został również d_z Cohena dla skorelowanych próbek [22].

Przeprowadzono jednokierunkową analizę ANOVA, aby sprawdzić, czy zmiany siły wiązania wynikające z termocyklingu (zmienna zależna) są statystycznie różne dla czterech rodzajów cementu (zmienna niezależna). Aby zbadać ten efekt bardziej szczegółowo i określić, które rodzaje cementu różnią się między sobą zmianą siły wiązania, przeprowadzono testy post-hoc (porównania parami za pomocą testu HSD Tukeya). Analizy uzupełniające przeprowadzono również oddzielnie dla każdej z trzech ceramik. Przeprowadzono mieszaną analizę wariancji (split-plot ANOVA) z ceramiką (tj. IPS e.max ZirCAD, IPS Empress, IPS e.max CAD) jako powtarzalny pomiar wewnątrz grupy oraz cementów (tj. Panavia SA, Panavia V5, RalyX U200, Maxcem) jako międzygrupowy czynnik wykorzystany do tego badania. Wartość prawdopodobieństwa $p < 0{,}05$ wskazywała na wyniki istotne statystycznie. Wszystkie analizy przeprowadzono w Statistica (system oprogramowania do analizy danych), wersja 10 (StatSoft Inc., Tulsa, OK, USA) oraz za pomocą narzędzi internetowych Psychometrica (Psychometrica - Alexandra Lenhard, Dettelbach, Niemcy) do obliczania wielkości efektu dla planowanej analizy kontrastów.

Siła wiązania po termocyklingu była statystycznie istotnie słabsza we wszystkich próbkach. Ta obserwacja dotyczy wszystkich czterech typów cementów na trzech materiałach ceramicznych. Jak pokazują obliczone wielkości efektów, wielkości obserwowanych różnic we wszystkich przypadkach były znaczne, co wskazuje na duży wpływ sztucznego starzenia (termocyklingu) na siłę wiązania.

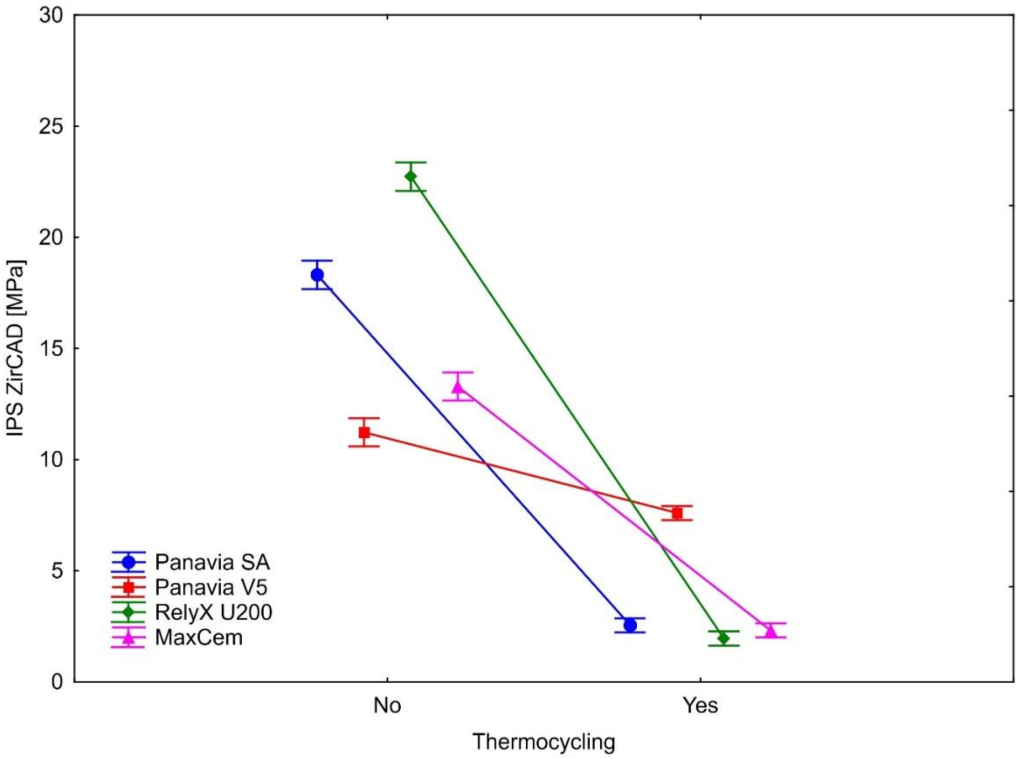
Analizy uzupełniające wykazały, że termocykling w różny sposób wpływał na wielkość zmian siły wiązania w zależności od rodzaju cementu. Efekt ten był statystycznie istotny dla wszystkich trzech ceramik (IPS e.max ZirCAD: $F(3,44) = 449{,}387$, $p < 0{,}0001$, $\eta2=0{,}97$); IPS Empress: $F(3,44) = 104{,}358$, $p < 0{,}0001$, $\eta2=0{,}88$); IPS e.max CAD: $F(3,44) = 35{,}795$, $p < 0{,}0001$, $\eta2=0{,}71$. W przypadku IPS ZirCAD najmniejszy spadek siły wiązania zaobserwowano dla cementu Panavia V5, większy w Maxcem i Panavia SA. Największy spadek siły wiązania zaobserwowano dla RelyX U200. Testy posthoc Tukeya wykazały, że różnice w spadku siły wiązania były statystycznie istotne między wszystkimi typami cementów (wszystkie $p = 0{,}0002$).

Podobnie w przypadku IPS Empress, Panavia V5 miała najmniejszy spadek siły wiązania. Większe wartości odchyleń (w porządku rosnącym) zaobserwowano dla Relyx U200, Maxcem i Panavia SA. Podczas gdy Relyx U200 i Maxcem nie różniły się statystycznie istotnie pod względem wielkości spadku siły wiązania ($p = 0{,}95$), inne porównania wykazały, że wielkości zmiany siły wiązania były statystycznie istotne pomiędzy analizowanymi typami cementu (Panavia SA vs Panavia V5 : $p = 0{,}0002$; Panavia SA vs Relyx U200: $p=0{,}008$; Panavia SA vs Maxcem $p = 0{,}03$; Panavia V5 vs Relyx U200: $p = 0{,}0002$; Panavia V5 vs Maxcem: $p = 0{,}0002$).

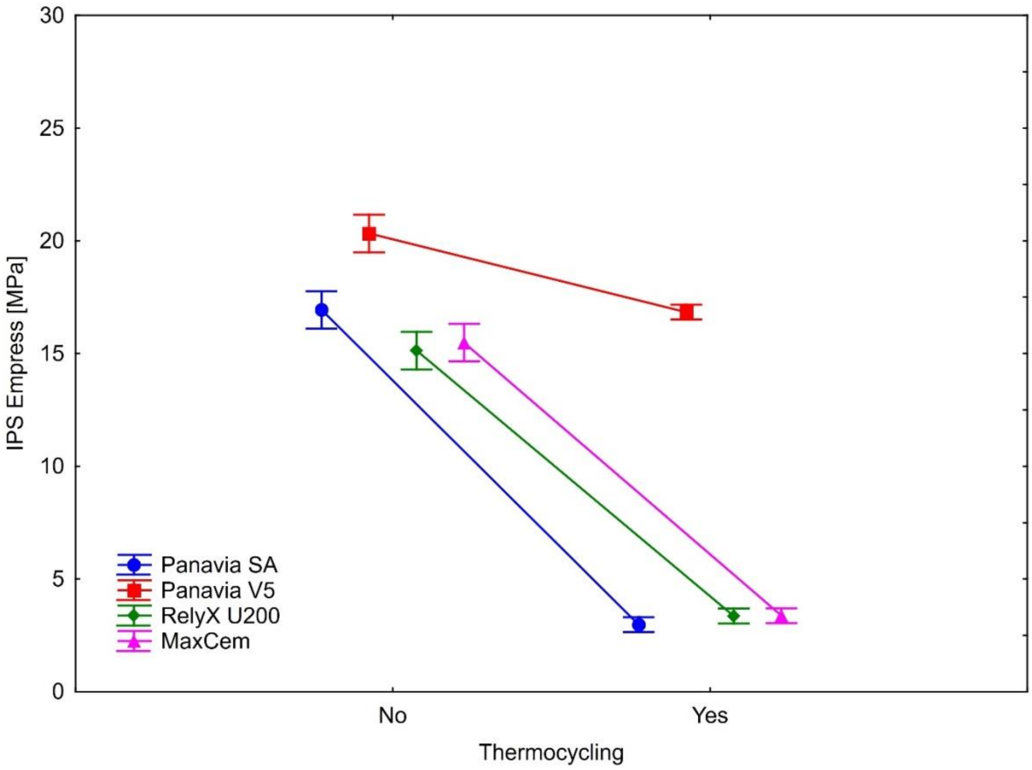
W przypadku IPS e.max CAD przebieg zmian siły wiązania był analogiczny jak w IPS Empress. Ponownie najniższy spadek zanotowano dla Panavii V5. Większe wartości (w kolejności rosnącej) zaobserwowano dla Relyx U200, Maxcem i Panavia SA (Tabela 5). Wielkość spadku siły wiązania nie różniła się istotnie statystycznie między Panavia SA i Maxcem ($p = 0{,}86$). Statystycznie istotne różnice zaobserwowano między innymi rodzajami cementu (Panavia SA vs Panavia V5: $p = 0{,}0002$; Panavia SA vs Relyx U 200: $p = 0{,}001$; Panavia V5 vs RelyX U200: $p = 0{,}0002$; Panavia V5 vsMaxcem: $p = 0{,}0002$; RelyX U200 vs Maxcem: $p = 0{,}01$). Szczegółowe wyniki przedstawiono w Tabeli 2 oraz na wykresach 1, 2, 3.

Tabela 2. Porównanie statystyczne siły wiązania dla próbek niepoddanych i poddanych sztucznemu starzeniu.

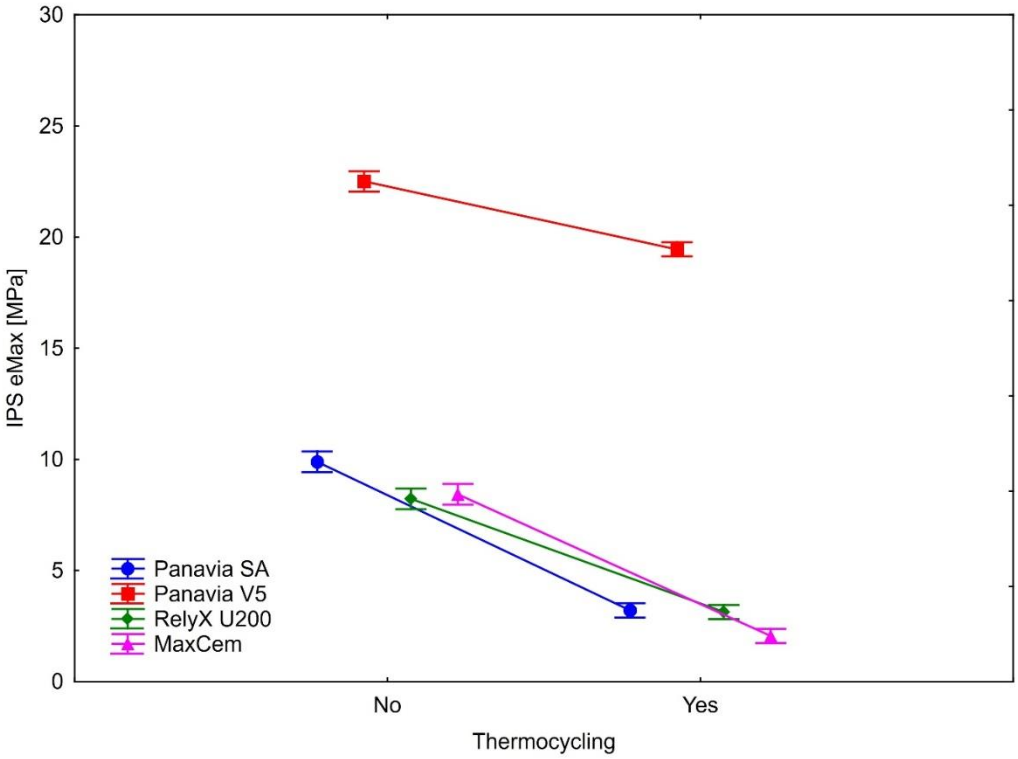
	Bez sztucznego starzenia (N)		**Ze sztucznym starzeniem (T)**		**N-T**		**t-test (df=11)**		**Wielkość efektu**
Cement	M(MPa)	SD	M(MPa)	SD	M(MPa)	SD	*t*-value	*p*-value	Cohen d_z
IPS e.max ZirCAD									
Panavia SA	18.31	1.357	2.55	0.558	15.77	1.424	38.36	<.0001	11.07
Panavia V5	11.23	0.475	7.60	0.765	3.632	0.875	14.37	<.0001	4.15
RelyX U200	22.73	1.148	1.95	0.520	20.78	1.196	60.18	<.0001	17.37
Maxcem	13.29	1.185	2.32	0.182	10.97	1.212	31.35	<.0001	9.05
IPS Empress									
Panavia SA	16.94	1.533	2.98	0.667	13.96	1.924	25.13	<.0001	7.25
Panavia V5	20.33	0.787	16.84	0.844	3.488	1.041	11.61	<.0001	3.35
RelyX U200	15.13	1.953	3.36	0.260	11.77	1.895	21.51	<.0001	6.21
Maxcem	15.48	1.158	3.37	0.229	12.11	1.274	32.92	<.0001	9.50
IPS e.max CAD									
Panavia SA	9.888	0.926	3.209	0.588	6.679	1.162	19.91	<.0001	5.75
Panavia V5	22.50	0.690	19.45	0.564	3.059	0.952	11.13	<.0001	3.21
RelyX U200	8.228	0.696	3.135	0.595	5.092	0.908	19.42	<.0001	5.61
Maxcem	8.428	0.867	2.058	0.445	6.370	0.739	29.87	<.0001	8.62



Wykres 1. Wpływ termocyklingu na siłę wiązania cementów użytych do połączenia IPS ZirCAD z zębiną. Wąsy oznaczają przedziały ufności 0,95 wokół wartości średnich.



Wykres 2. Wpływ termocyklingu na siłę wiązania cementów użytych do połączenia IPS Empress z zębiną. Wąsy oznaczają przedziały ufności 0,95 wokół wartości średnich.



Wykres 3. Wpływ termocyklingu na siłę wiązania cementów użytych do połączenia IPS eMax z zębiną. Wąsy oznaczają przedziały ufności 0,95 wokół wartości średnich.

Ocena mikroskopowa przełomów

Próbki po teście na maszynie wytrzymałościowej poddano obserwacji za pomocą mikroskopu świetlnego. Wyniki oceny ilościowej częstości występowanie określonego rodzaju przełomów zostały zawarte w Tabeli 3 dla próbek bez sztucznego starzenia oraz w Tabeli 4 dla próbek poddanych termocyklingowi.

Tabela 3. Ilościowa ocena częstości występowania określonego rodzaju przełomów po przeprowadzeniu testu wytrzymałości na ścinanie dla próbek bez sztucznego starzenia.

		Rodzaje złamań [%]			
Ceramika	**Cement**	**Adhezyjne złamanie pomiędzy zębiną/cementem**	**Adhezyjne złamanie pomiędzy cementem/ceramiką**	**Kohezyjne złamanie w cemencie**	**Mieszane złamanie**
IPS Empress CAD	RelyX U200	75	0	25	0
	Maxcem Elite	75	0	25	0
	Panavia SA	83.33	0	0	16.67
	Panavia V5	0	0	50	50
IPS e.max CAD	RelyX U200	83.33	0	0	16.67
	Maxcem Elite	91.66	0	8.34	0
	Panavia SA	100	0	0	0
	Panavia V5	0	91.66	0	8.34
IPSe.maxZirCAD	RelyX U200	100	0	0	0
	Maxcem Elite	91.66	8.34	0	0
	Panavia SA	75	25	0	0
	Panavia V5	50	50	0	0

Tabela 4. Ilościowa ocena częstości występowania określonego rodzaju przełomów po przeprowadzeniu testu wytrzymałości na ścinanie dla próbek poddanych przyspieszonemu starzeniu.

		Rodzaje złamań [%]			
Ceramika	**Cement**	**Adhezyjne złamanie pomiędzy zębiną/cementem**	**Adhezyjne złamanie pomiędzy cementem/ceramiką**	**Kohezyjne złamanie w cemencie**	**Mieszane złamanie**
IPS Empress CAD	RelyX U200	58.4	0	0	41.6
	Maxcem Elite	66.7	0	33.3	0
	Panavia SA	75	0	25	0
	Panavia V5	0	0	33.3	66.7
IPS e.max CAD	RelyX U200	75	0	8.3	16.7
	Maxcem Elite	83.3	0	0	16.7
	Panavia SA	75	0	25	0
	Panavia V5	58.4	0	0	41.6
IPS e.maxZirCAD	RelyX U200	50	0	25	25
	Maxcem Elite	66.7	16.6	0	16.6
	Panavia SA	50	25	0	25
	Panavia V5	58.4	41.6	0	0

VIII. DYSKUSJA

Długoterminowy sukcesu kliniczny pełnoceramicznych uzupełnień protetycznych wiąże się nie tylko z wytrzymałością mechaniczną ceramiki, ale także z trwałością i silną adhezja materiału ceramicznego do twardych tkanek zęba zapewnione przez właściwe cementowanie. Niezawodność i odporność na wyjątkowo trudne środowisko jamy ustnej jest szczególnie istotnym aspektem stomatologii odtwórczej [3,7–9]. Trwałe połączenie ceramiki z tkanką zęba opiera się na wiązaniu chemicznym i mikromechanicznym blokujące. Współcześnie temat łączenia nowoczesnej ceramiki wysokowytrzymałej przetwarzanej w cyfrowych technologiach CAD/CAM, jest intensywnie badany w wielu aspektach. Wiele badań skupia się na modyfikacjach powierzchni ceramiki w celu uzyskania najwyższej możliwej siły wiązania [1–8,12–15]. W przeciwieństwie do tego punktu widzenia jest podejście podkreślające potencjał zastosowania samotrawiących, samoadhezyjnych cementów na bazie żywic, znacznie zmniejszających liczbę etapów związanych z przygotowaniem powierzchni do cementowania, co w rezultacie zmniejsza również możliwe błędy operatora [19].

Symulacja zachowania się materiałów dentystycznych w czasie odbywa się za pomocą sztucznego starzenia. Niestety nie istnieje ujednolicony protokół do symulacji tego procesu. W literaturze opisano symulacje dynamiczne [17,18] lub przechowywanie próbek w łaźniach wodnych o stałej temperaturze [6,14,21], czy termocykling [19]. Termocykling jest obecnie jedną z najczęstszych metod starzenia materiału [5], jednak jego parametry również nie zostały jeszcze jednoznacznie ustandaryzowane. Comino-Garayoa i wsp. przeprowadzili przegląd systematyczny analizując 45 różnych badań, dochodząc do wniosku, że sztuczne starzenie powinno być oparte na 5000 termicznych cykli lub 30 dniach ciągłej kąpieli wodnej [12]. Inne przeglądy systematyczne [8-10] porównujące parametry sztucznego starzenia pozwalają wyciągnąć ogólny wniosek, że a należy zastosować gradient temperatur od 5° C do 55° C. Te temperatury są również zgodnie ze Specyfikacją Techniczną ISO TS 11405 do badania przyczepności do struktur zęba. Z drugiej strony nadal istnieją znaczne różnice w liczbie wykonanych cykli, co sugeruje, że jest to parametr oparty na predykcji.

Przeprowadzone badanie wraz z analizą statystyczną wykazały, że siły wiązania wybranych cementów samotrawiących, samoadhezyjnych były silnie uzależnione od zastosowania metody sztucznego starzenia.

Zauważalny, choć znacznie mniejszy, był również spadek siły wiązania konwencjonalnego cementu Panavia V5 stosowanego jako grupa kontrolna [27]. Niektóre badania in vitro dowiodły, że cementy samotrawiące, samoadhezyjne mogą być bardziej korzystne dla tkanek zęba, ponieważ mogą wykazywać mniejszą toksyczność niż cementy konwencjonalne [21]. Z drugiej strony jest to sprzeczne z wynikami Sawada i wsp., w których nie zaobserwowano istotnych różnic między cementami samotrawiącymi, samoadhezyjnymi [13,27]. Jako że, metody przygotowania próbek badawczych i oceny ich siły wiązania prezentowane we współczesnej literaturze różnią się znacznie, niemożliwe jest przeprowadzenie rzetelnej metaanalizy, pozwalającej na wyciągnięcie szerszych wniosków [27–31]. Metodologia opisana w tym badaniu jest ustandaryzowana w oparciu o wytyczne ISO, które dają możliwość ich reprodukcji oraz porównania w przypadku przeprowadzenia innych badań według tych samych wytycznych. Głównym ograniczeniem tego badania jest zastosowanie, najpopularniejszej, aczkolwiek prostej metody sztucznego przyspieszonego starzenia. Dalsze badania są niezbędne, biorąc pod uwagę również inne metody starzenia próbek. Ogólnie trzeba wziąć pod uwagę że cementy na bazie żywic, podobnie jak inne materiały stosowane do odbudowy twardych tkanek zębów, muszą wytrzymać szereg czynników bezpośrednio i pośrednio wpływających na siłę i stabilność połączenia. Należy zauważyć, że środowisko jamy ustnej zależy nie tylko od zmian temperatury. Zmiany wilgotności, pH, enzymów w ślinie lub sił fizycznych działające w trzech osiach również znacząco wpływają na utrzymanie rekonstrukcji [19]. Shahin i wsp. Zastosowali przyspieszone starzenie w oparciu o kombinowany termocykling i dynamiczne ładowanie materiału próbek, które wydaje się lepiej odzwierciedlać rzeczywiste warunki w jamie ustnej [16]. Podążanie w tym kierunku byłoby cenną kontynuacją dla obecnego badania.

Z przeprowadzonego przeglądu systematycznego wynika, że połączenie modyfikacji mechanicznej i chemicznej powierzchni ceramiki są niezbędne dla uzyskania dobrej przyczepności. Jednak obecnie brak jest dowodów na poparcie uniwersalnego protokołu adhezji [17,18]. Przegląd systematyczny skupiał się przede wszystkim na wpływie modyfikacji powierzchni na siłę wiązania między ceramiką a podłożami dentystycznymi. Zdecydowana większość spośród wybranych badań obejmowała modyfikację cyrkonu w celu uzyskania trwałej metody cementowania tego materiału. Oprócz oceny skuteczności metod modyfikacji powierzchni, w przeglądzie zwrócono również uwagę na efekty działania sztucznego starzenia.

Kolejną interesującą kwestią, którą należy zbadać w przyszłości, są ograniczenia badania wytrzymałości z wykorzystaniem testu na ścinanie ponieważ, nie odzwierciedlają w pełni rzeczywistej sytuacji klinicznej ze złożonym wzorcem rozkładu naprężeń. W związku z tym wykonywanie badań zmęczeniowych pod obciążeniem cyklicznym, wydaje się lepiej symulować funkcję żucia.

IX. WNIOSKI

1. Konwencjonalny cement Panavia V5 wykazywał znacznie wyższą siłę wiązania do każdego rodzaju ceramiki dentystycznej w porównaniu z samoadhezyjnymi, samotrawiącymi cementami zarówno po przyspieszonym starzeniu termicznym, jak i bez jego zastosowania.

2. Niezależnie od badanego cementu, najniższą siłę wiązania spośród badanych ceramik uzyskano dla próbek wykonanych z IPS e.maxZirCAD.

3. Właściwy dobór cementu do ceramiki ma kluczowe znaczenie, ponieważ różnice w wiązaniu badanych kombinacji wybranych cementów i ceramiki były istotne statystycznie.

4. Statystyczne porównanie próbek poddanych przyspieszonemu starzeniu z próbkami badanymi 24 godziny po zacementowaniu wykazało największy spadek sił wiązania dla cementów samoadhezyjnych, samotrawiących.

5. W oparciu o przeanalizowane wyniki, połączenie mechanicznej i chemicznej modyfikacji powierzchni ceramiki daje najskuteczniejszy sposób zwiększania siły wiązania pomiędzy ceramiką a tkankami twardymi zębów.

6. Przegląd dostępnej literatury podkreśla potrzebę standaryzacji metodyki modyfikacji powierzchni dla przyszłych badań ze względu na użycie różnych materiałów, protokołów i testów przez badaczy ponieważ, porównanie danych jest problematyczne ze względu na brak jednorodności.

7. Ponadto standaryzowane protokoły powinny próbować odtworzyć stan kliniczny poprzez: stosowanie różnych metod testowania, w tym testów zmęczeniowych, a także poprzez sztuczne starzenie próbek.

X. PIŚMIENNICTWO

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28. Małysa, A.; Weżgowiec, J.;Orzeszek, S.; Florjański, W.; Ziętek, M.; Więckiewicz, M. Effect of different surface treatment methods on bond strength of dental ceramics to dental hard tissues: a systematic review. *Mol.* 2021, 26(5), pp. 1210-1223.

XI. OŚWIADCZENIA WSPÓŁAUTORÓW

Prof. dr hab. n. med. Mieszko Więckiewicz Wrocław, 11.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor prac wchodzących w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracach:

1. Małysa, A.; Weżgowiec, J.; Danel, D.; Boening, K.; Walczak, K.; Więckiewicz, M. Bond strength of modern self-adhesive resin cements to human dentin and different CAD/CAM ceramics. *Acta Bioeng Biomech.* 2020, 22(2), pp. 25-34.
2. Małysa, A.; Weżgowiec, J.; Grzebieluch, W.; Danel, D.; Więckiewicz, M. Effect of thermocycling on the bond strength of self-adhesive resin cements used for luting CAD/CAM ceramics to human dentin. *Int J Mol Sci.* 2022, 23(2), pp. 714-745.
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mój udział jako współautora wymienionych publikacji, polegał na pomocy przy metodologii, analizie statystycznej, korekty manuskryptów.

Wyrażam zgodę na włączenie wyżej wymienionych publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

Prof. dr hab. n. med. Marek Ziętek Wrocław, 11.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor pracy wchodzących w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracy:

1. Małysa, A.; Weżgowiec, J.; Orzeszek, S.; Florjański, W.; Ziętek, M.; Więckiewicz, M. Effect of different surface treatment methods on bond strength of dental ceramics to dental hard tissues: a systematic review. *Mol.* 2021, 26(5), pp. 1210-1223.

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Wyrażam zgodę na włączenie wyżej wymienionej publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

Marek Ziętek

Prof. Dr. med. dent. Klaus Boening Wrocław, 07.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor prac wchodzących w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracy:

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Prof. Dr. med. dent. Klaus W. Böning
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Podpis

Dr inż. Joanna Weżgowiec Wrocław, 10.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor prac wchodzących w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracach:

4. Małysa, A.; Weżgowiec, J.; Danel, D.; Boening, K.; Walczak, K.; Więckiewicz, M. Bond strength of modern self-adhesive resin cements to human dentin and different CAD/CAM ceramics. *Acta Bioeng Biomech.* 2020, 22(2), pp. 25-34.
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Wyrażam zgodę na włączenie wyżej wymienionych publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Joanna Weżgowiec

Podpis

INSTYTUT IMMUNOLOGII I TERAPII DOŚWIADCZALNEJ
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Wrocław, 11/10/2022

Dariusz Danel, dr hab.
Zakład Antropologii IITD PAN

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

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Wyrażam zgodę na włączenie wyżej wymienionych publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

Dr. med. Dent. Katarzyna Kresse-Walczak Wrocław, 11.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

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Wyrażam zgodę na włączenie wyżej wymienionej publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

Dr n. med. Wojciech Florjański Wrocław, 24.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor pracy wchodzącej w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracy:

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Wyrażam zgodę na włączenie wyżej wymienionej publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

W. Florjański

Lek. dent. Sylwia Orzeszek Wrocław, 25.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

Jako współautor pracy wchodzącej w skład rozprawy doktorskiej lek. dent. Andrzeja Małysy oświadczam, że w pracy:

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Wyrażam zgodę na włączenie wyżej wymienionych publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

Sylwia Orzeszek

Dr n. med. Wojciech Grzebieluch Wrocław 20.10.2022

OŚWIADCZENIE

Dotyczy przewodu doktorskiego lek. dent. Andrzeja Małysy

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Wyrażam zgodę na włączenie wyżej wymienionych publikacji do rozprawy doktorskiej lek. dent. Andrzeja Małysy.

Podpis

XII. ZGODA KOMISJI BIOETYCZNEJ

1

KOMISJA BIOETYCZNA
przy
Uniwersytecie Medycznym
we Wrocławiu
ul. Pasteura 1; 50-367 WROCŁAW

OPINIA KOMISJI BIOETYCZNEJ Nr KB – 37/2018

Komisja Bioetyczna przy Uniwersytecie Medycznym we Wrocławiu, powołana zarządzeniem Rektora Uniwersytetu Medycznego we Wrocławiu nr 133/XV R/2017 z dnia 21 grudnia 2017 r. oraz działająca w trybie przewidzianym rozporządzeniem Ministra Zdrowia i Opieki Społecznej z dnia 11 maja 1999 r. (Dz.U. nr 47, poz. 480) na podstawie ustawy o zawodzie lekarza z dnia 5 grudnia 1996 r. (Dz.U. nr 28 z 1997 r. poz. 152 z późniejszymi zmianami) w składzie:

dr hab. Jacek Daroszewski (endokrynologia, diabetologia)
prof. dr hab. Krzysztof Grabowski (chirurgia)
dr Henryk Kaczkowski (chirurgia szczękowa, chirurgia stomatologiczna)
mgr Irena Knabel-Krzyszowska (farmacja)
prof. dr hab. Jerzy Liebhart (choroby wewnętrzne, alergologia)
ks. dr hab. Piotr Mrzygłód (duchowny)
mgr Luiza Műller (prawo)
dr hab. Sławomir Sidorowicz (psychiatria)
dr hab. Leszek Szenborn (pediatria, choroby zakaźne)
Danuta Tarkowska (pielęgniarstwo)
prof. dr hab. Anna Wiela-Hojeńska (farmakologia kliniczna)
dr hab. Andrzej Wojnar (histopatologia, dermatologia) przedstawiciel Dolnośląskiej Izby Lekarskiej)
dr hab. Jacek Zieliński (filozofia)

pod przewodnictwem
prof. dr hab. Jana Kornafela (ginekologia i położnictwo, onkologia)

Przestrzegając w działalności zasad Good Clinical Practice oraz zasad Deklaracji Helsińskiej, po zapoznaniu się z projektem badawczym pt.:

„Ocena siły połączenia między wybranymi ceramikami dentystycznymi a zębiną z wykorzystaniem samoadhezyjnych samotrawiących cementów na bazie żywic" zgłoszonym przez **lek dent. Andrzeja Małysę** zatrudnionego w Katedrze i Zakładzie Stomatologii Doświadczalnej Uniwersytetu Medycznego we Wrocławiu oraz złożonymi wraz z wnioskiem dokumentami, w tajnym głosowaniu postanowiła **wyrazić zgodę** na przeprowadzenie badania w Katedrze i Zakładzie Stomatologii Doświadczalnej Uniwersytetu Medycznego we Wrocławiu.

Uwaga: Badanie to zostało objęte ubezpieczeniem odpowiedzialności cywilnej Uniwersytetu Medycznego we Wrocławiu z tytułu prowadzonej działalności.

Pouczenie: W ciągu 14 dni od otrzymania decyzji wnioskodawcy przysługuje prawo odwołania do Komisji Odwoławczej za pośrednictwem Komisji Bioetycznej UM we Wrocławiu.

Opinia powyższa dotyczy projektu badawczego będącego podstawą działalności statutowej.

Uniwersytet Medyczny we Wrocławiu
KOMISJA BIOETYCZNA
przewodniczący
prof. dr hab. Jan Kornafel

Wrocław, dnia 6 lutego 2018 r.

XIII. PUBLIKACJE STANOWIĄCE CYKL PRACY DOKTORSKIEJ

PRACE ORYGINALNE

I. **Bond strength of modern self-adhesive resin cements to human dentin and different CAD/CAM ceramics.** [AUT.] ANDRZEJ MAŁYSA, [AUT. KORESP.] JOANNA WEŻGOWIEC, [AUT.] DARIUSZ DANEL, KLAUS BOENING, KATARZYNA WALCZAK, MIESZKO WIĘCKIEWICZ. *Acta Bioeng Biomech.* 2020 Vol.22 no.2 s.25-34, ryc., tab., bibliogr. 25 poz., summ. DOI: 10.37190/ABB-01526-2019-02. Impact Factor: 1,073; punktacja MEiN: 100.

II. **Effect of thermocycling on the bond strength of self-adhesive resin cements used for luting CAD/CAM ceramics to human dentin.** [AUT.] ANDRZEJ MAŁYSA, JOANNA WEŻGOWIEC, WOJCIECH GRZEBIELUCH, DARIUSZ P. DANEL, [AUT. KORESP.] MIESZKO WIĘCKIEWICZ. *Int J Mol Sci.* 2022 Vol.23 no.2 art.745 [14 s.], ryc., tab., bibliogr. 31 poz., summ. DOI: 10.3390/ijms2302074. Impact Factor: 6,208 ; punktacja MEiN: 140.

PRACE POGLĄDOWE

III. **Effect of different surface treatment methods on bond strength of dental ceramics to dental hard tissues: a systematic review.** [AUT.] ANDRZEJ MAŁYSA, [AUT. KORESP.] JOANNA WEŻGOWIEC, [AUT.] SYLWIA ORZESZEK, WOJCIECH FLORJAŃSKI, MAREK ZIĘTEK, MIESZKO WIĘCKIEWICZ. *Mol.* 2021 Vol.26 no.5 art.1223 [15 s.], ryc., tab., bibliogr. 46 poz., summ. DOI: 10.3390/molecules26051223. Impact Factor: 4,927; punktacja MEiN: 140.

Acta of Bioengineering and Biomechanics
Vol. 22, No. 2, 2020

Original paper
DOI: 10.37190/ABB-01526-2019-02

Bond strength of modern self-adhesive resin cements to human dentin and different CAD/CAM ceramics

ANDRZEJ MAŁYSA[1], JOANNA WEŻGOWIEC[1]*, DARIUSZ DANEL[2],
KLAUS BOENING[3], KATARZYNA WALCZAK[3], MIESZKO WIĘCKIEWICZ[1]

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Purpose: The aim of the study was to evaluate the shear bond strength of CAD/CAM ceramics to dentin after cementation with conventional or self-adhesive resin cements. *Methods*: Three self-adhesive, self-etching cements (Panavia SA, RelyX U200, Maxcem Elite), and one conventional cement (Panavia V5), were selected to lute three CAD/CAM ceramics (IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD) onto the dentin. The bond strength was evaluated using a shear strength test according to the PN-EN ISO 29022:2013-10. Evaluation of the differences was performed using the Statistica software. Failure modes were analyzed using a light microscope. *Results*: All the studied cements differed (regardless of the ceramic type) in the bond strength. The highest bond strength was observed in Panavia V5, lower – in RelyX U200 and Panavia SA, and the lowest – in Maxcem. For IPS e.max ZirCAD, it was observed that compared to Panavia V5, the other cements were characterized by a significantly higher bond strength. For the IPS Empress CAD and the IPS e.max CAD, Panavia V5 displayed the highest bond strength. For all the studied self-adhesive cements, the failure of adhesion between the cement and dentin was predominant mode. *Conclusions*: Significant differences were found in the shear bond strengths of the CAD/CAM ceramics luted to dentin using tested self-adhesive and conventional cements. The bond strength depended on the combination of ceramic and cement. The IPS e.max ZirCAD had the highest bond strength to dentin after cementation with RelyX U200, while the IPS Empress CAD and IPS e.max CAD – with Panavia V5.

Key words: *shear bond strength, self-adhesive resin cement, dentin, CAD/CAM ceramic, dental materials, adhesion*

1. Introduction

A remarkable technical development in the manufacturing of dental materials allowed for the creation of restorations that almost perfectly match the natural ones in terms of both function and esthetics. For this purpose, modern prosthodontics uses computer-aided design/computer-aided manufacturing (CAD/CAM) technology, which has several advantages, such as ease of application, minimal invasiveness, standardized manufacturing process, and long-term clinical success [23].

The indirect restorative materials commonly used in CAD/CAM technology are glass ceramics (feldspathic ceramics, mica-based ceramics, leucite- or lithium disilicate-reinforced ceramics, glass-infiltrated alumina or zirconia ceramics) and polycrystalline ceramics, such as alumina and zirconia. These are highly biocompatible and esthetic; however, taking the huge amount of load in the oral environment into account, dental restorations should be ensured to pos-

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sess appropriate mechanical properties to avoid their fracture or loss of contact with dentin. For this reason, many efforts have been recently made to develop materials that are characterized by not only excellent esthetics but also high strength in addition to good bond strength to the dental substrates [2], [4], [16].

To achieve a strong bonding between the ceramic and dentin, a proper luting cement should be applied. Several types of cements are used in dentistry: zinc phosphate cement, polycarboxylate cement, glass ionomer cement, resin-modified glass ionomer cement, and resin cement [14]. Resin cements can be divided into three groups: total-etch and self-etch cements, which require to be bonded to the tooth surface; and self-adhesive resin cements. Significant technological advancements made in the manufacturing of self-adhesive self-etching resin cements have enabled achieving unprecedented speed and ease of operation [1]. The issues related to bond strength that are encountered with the new-generation self-adhesive cements are currently investigated by many research groups [1], [5], [7], [8], [14], [21], [24]. Unfortunately, due to differences in the method, the data from these studies are hardly comparable, and therefore, it is very difficult to draw a general conclusion. As many factors influence the results of bond strength tests, including the properties of dentin, properties of ceramics and their preparation, and parameters related to testing, research methods should be designed in a more standardized way [22].

This study evaluated and compared the shear bond strengths of CAD/CAM ceramics to dentin after cementation with the modern self-adhesive resin cements following the PN-EN ISO 29022:2013-10 standard to ensure the reproducibility of the results. In addition, a complex statistical analysis was performed to evaluate the differences in the bond strengths after cementation with the conventional and modern self-adhesive self-etching cements to propose the most strong combination of CAD/CAM ceramic and resin cement. The tested null hypothesis was that there would be no difference in the shear bond strengths between the CAD/CAM ceramics luted to dentin using the modern self-adhesive and conventional resin cements.

2. Materials and methods

2.1. Materials

A total of 144 ceramic samples in a shape of a cylinder were prepared and cemented into the dentin (12 samples for each ceramic – cement combination). Three types of ceramics (IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD) possessing different physical and chemical properties were milled using CAD/CAM technology. For each type of ceramic, 48 cylinders were prepared and divided into four groups consisting of 12 each, depending on the type of resin cement used for cementing the ceramic onto the dentin. Three resin-based cements (Panavia SA, RelyX U200, Maxcem Elite) characterized by similar physicochemical properties and binding type (self-etching, self-adhesive) were used as study materials, while one resin-based adhesive cement (Panavia V5) was selected as a control sample. The different types of ceramics and resin cements used in this study are summarized in Table 1. Ceramics were cemented into the cylinders prepared from 53 human molars, which were freshly removed and cut into 144 plasters, in accordance with the PN-EN ISO 29022:2013-10 standard.

Table 1. CAD/CAM ceramics and resin cements used in this study

Name	Manufacturer	Type
CERAMICS		
IPS Empress CAD HT A1	Ivoclar Vivadent (Schaan, Liechtenstein)	Leucite glass
IPS e.max CAD HT A1	Ivoclar Vivadent (Schaan, Liechtenstein)	Lithium disilicate
IPS e.max ZirCAD	Ivoclar Vivadent (Schaan, Liechtenstein)	Zirconia
RESIN CEMENTS		
Panavia V5 A1	Kuraray Noritake (Tokyo, Japan)	Adhesive
Panavia SA Cement Universal A1	Kuraray Noritake (Tokyo, Japan)	Self-adhesive, self-etching
RelyX U200 A1	3M ESPE (Maplewood, Minnesota, USA)	Self-adhesive, self-etching
Maxcem Elite A1	Kerr (Brea, California, USA)	Self-adhesive, self-etching

2.2. Tooth preparation

Immediately after the extraction procedure (Wroclaw Medical University Bioethical Committee approval No. KB-37/2018), the teeth were stored in a 10% methanol solution (CHEMPUR, Piekary Sl., Poland) at 4 °C for approximately 24 h. The soft tissue residues were removed from the teeth using a scalpel, and their surfaces were cleaned. The preprepared teeth were sectioned using the PetroThin Thin Sectioning System with diamond cutting and water cooling features (Buehler, Lake Bluff, Illinois, USA). First, the root was cut off, and then, the crown of the teeth was cut into 3-mm slices to reveal the healthy dentin. The slices were embedded in a transparent Villacryl H Plus fast-curing acrylic (Zhermack, Öhlmühle, Germany) in a silicone mold of dimensions of 35 mm × 25 mm × 4 mm. Before cementing, the surfaces of the samples to which the ceramic materials were intended to be attached were ground with a carborundum paper of P 400 granularity (Luna, Berno, Switzerland) under a stream of running water to obtain a flat surface.

2.3. Ceramic samples preparation

For each resin cement, 12 cylinders of CAD/CAM ceramics, with a diameter of 2.38 mm and a height of 5 mm, were prepared following the PN-EN ISO 29022:2013-10 standard. In the first stage, the cylinders were designed in Sirona Cerec inLAB using a silicone mold and the BlueCam Sirona Cerec scanner (Sirona, New York, USA). Then, the cylinders were milled and the dimensions of the ceramic samples were checked using 150 mm Limit 144550100 digital caliper (Limit, Wrocław, Poland). The IPS e.max CAD and IPS e.max ZirCAD samples were heat-treated in accordance with the manufacturer's instructions in the Ivoclar Vivadent Programat CS and Ivoclar Vivadent Programat S1 furnaces (Schaan, Liechtenstein). The parameters of the crystallization process and sintering for the IPS e.max CAD and IPS e.max ZirCAD ceramics are presented in Table 2.

Table 2. The parameters of crystallization process and sintering

	IPS e.max CAD	IPS e.max ZirCAD
Furnace	Programat CS	Programat S1
Standby mode [°C]	403	403
Closing time [min]	6:00	4:00
Temperature increase [°C]	90	40
Holding temperature T1 [°C]	820	960
Holding time H1 [min]	0:10	01:00
Holding temperature T2 [°C]	840	–
Holding time H2 [min]	7:00	–
Vacuum on [°C]	550	450
Vacuum off [°C]	820	959
Long-term cooling [°C]	700	0

2.4. Cementing ceramics into dentin

Cementation was carried out in accordance with the manufacturers' recommendations. In the case of Panavia V5 cement, both the ceramics and dentin surfaces were required to be prepared. After digestion with 37% orthophosphoric acid (3M ESPE, Maplewood, Minnesota, USA), the dentin surface was rinsed with distilled water and dried. The surfaces of the IPS Empress CAD and IPS e.max CAD ceramic samples were subjected to etching with 9% hydrofluoric acid (3M ESPE, Maplewood, Minnesota, USA) for 1 min, rinsed with distilled water, and dried with air jet, while the surface of the IPS e.max ZirCAD ceramic was sandblasted with alumina of 110-μm diameter using the CoJet System (3M ESPE, Maplewood, Minnesota, USA). In the case of Panavia SA, RelyX U200, and Maxcem Elite cements, the joined surfaces should be dried. Therefore, after rinsing under a stream of distilled water, the sample surfaces were dried with compressed air.

The process of cementation was carried out under the control of the FB(C) dynamometer (Axis, Gdańsk, Poland) with a fixed compression force of 10 N for each sample. Firstly, resin cements were applied to both surfaces and the samples were pressed. Then, the excess resin cement was removed and cement in the joined surfaces was polymerized using an Elipar LED lamp (3M ESPE, Maplewood, Minnesota, USA) for 20 s. Before performing the shear bond strength test, the prepared samples were stored in distilled water at 37 °C for 24 h.

2.5. Evaluation of the shear bond strength between ceramic and dentin

The laboratory shear bond strength tests were carried out using a universal testing machine (Thumler, Nurnberg, Germany). The schematic illustration of a sample subjected to shear force is presented in Fig. 1. The crosshead with a 1-mm/min speed of shear knife movement and a maximum force of 3000 N were applied in the tests in accordance with the PN-EN ISO 29022:2013-10 standard. Each of the 12 tested groups representing a ceramic–cement combination had 12 samples.

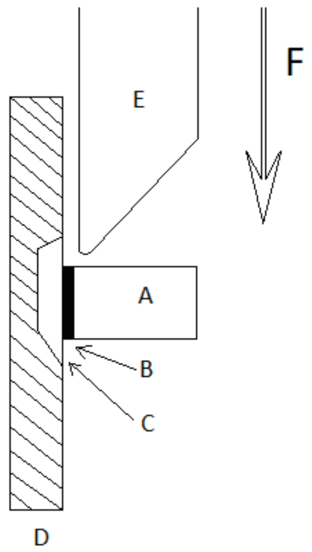

Fig. 1. Schematic illustration of a sample subjected to shear force: *A* – ceramic cylinder, *B* – resin cement, *C* – slice of human tooth, *D* – acrylic used for mounting the prepared tooth, *E* – shear blade of the testing machine, and *F* – direction of the applied force

2.6. Statistical analysis

A mixed-design analysis of variance (split-plot ANOVA) was carried out, considering ceramics (i.e., IPS e.max ZirCAD, IPS Empress CAD, IPS e.max CAD) as a within-group repeated measure and the dental cement (i.e., Panavia SA, Panavia V5, RelyX U200, Maxcem Elite) as a between-group factor. A post hoc analysis was conducted to identify and statistically test the differences between the compared groups. Additionally, a planned contrast analysis was performed. A probability value of $p < 0.05$ indicated statistically significant results. All the statistical analyses were conducted in Statistica version 13 using online tools for calculating the effect sizes as described by Lenhard and Lenhard [15].

2.7. Analysis of failure mode under a light microscope

The fractured specimens were observed under ×5 magnification using a light microscope (Axio Lab. A1 MAT, Zeiss, Oberkochen, Germany) to determine the failure mode: failure of adhesion between ceramic and cement, failure of adhesion between dentin and cement, failure of cohesion in cement, failure of cohesion in ceramic, failure of cohesion in dentin, or mixed failure.

3. Results

3.1. Evaluation of the shear bond strength between ceramic and dentin

The results of the comparative analysis of the shear bond strength between the selected types of ceramics and dentin connected using selected resin cements are presented in Fig. 2.

All four types of dental cements examined differed (regardless of the ceramic type) in the bond strength ($F(3, 44) = 161.03$, $p < 0.0001$, $\eta_p^2 = 0.92$). The highest bond strength was observed in Panavia V5 (M = 18.02), lower – in RelyX U200 (M = 15.36) and Panavia SA (M = 15.05), and the lowest – in Maxcem (M = 12.40). The post hoc analysis by Tukey's honestly significant difference (HSD) test showed statistically significant differences (all $p_s < 0.0002$) in all but one pairwise comparison of the dental cements. Only in the case of comparison between Panavia SA and RelyX U200, a statistically nonsignificant difference in the bond strength was observed ($p = 0.61$).

Regardless of the dental cements, the examined ceramics differed in the bond strengths ($F(2, 88) = 235.18$, $p < 0.0001$, $\eta_p^2 = 0.84$). The highest bond strength was observed in IPS Empress CAD (M = 16.97), lower – in IPS e.max ZirCAD (M = 16.39), and the lowest – in IPS e.max CAD (M = 12.26). Tukey's post hoc tests showed that the differences observed in all the comparisons were statistically significant (IPS Empress CAD vs. IPS e.max ZirCAD: $p = 0.04$; IPS Empress CAD vs. IPS e.max CAD: $p = 0.0001$; IPS e.max ZirCAD vs. IPS e.max CAD: $p = 0.0001$).

The statistically significant results observed in the analysis of the interaction between the ceramics and the resin cements ($F(6, 88) = 284.02$, $p < 0.0001$, $\eta_p^2 = 0.95$) indicated that the bond strength depends on the combination of both materials (Fig. 2). While the bond strength for RelyX U200 and Panavia SA cements decreased from IPS e.max ZirCAD through IPS Empress CAD to IPS e.max CAD, a reverse pattern of results was observed for Panavia V5 (i.e., the lowest bond strength was observed for IPS e.max ZirCAD, higher – for IPS Empress CAD, and the highest – for

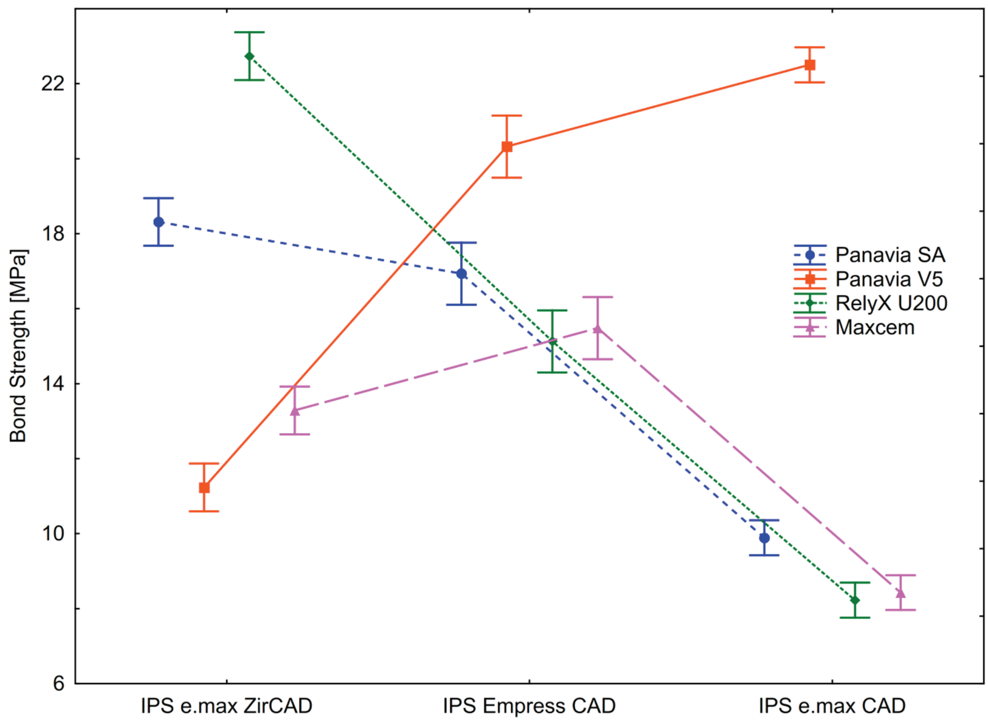

Fig. 2. Shear bond strength of the examined samples. Vertical bars denote 0.95 confidence intervals

Table 3. Planned contrast analysis of the differences in the bond strength between Panavia V5 and Panavia SA, RelyX U200, Maxcem, respectively. The Cohen's d value for groups with equal size was calculated according to the method described by Lenhard and Lenhard [15]

	IPS e.max ZirCAD			IPS Empress CAD			IPS e.max CAD		
Panavia V5 vs.	*t*(44)	*p*	Cohen's *d*	*t*(44)	*p*	Cohen's *d*	*t*(44)	*p*	Cohen's *d*
Panavia SA	15.85	<0.0001	6.97	–5.82	<0.0001	2.78	–38.56	<0.0001	15.46
RelyX U200	25.75	<0.0001	13.01	–8.93	<0.0001	3.49	–43.63	<0.0001	20.61
Maxcem	4.60	<0.0001	2.28	–8.32	<0.0001	4.90	–43.02	<0.0001	17.97

IPS e.max CAD). Maxcem had the highest bond strength for IPS Empress CAD, lower – for IPS e.max ZirCAD, and the lowest – for IPS e.max CAD. The post hoc analysis by Tukey's HSD test showed that only seven (out of 66) pairwise comparisons did not show statistically significant differences, while the differences in bond strength observed in all the other comparisons were statistically significant (all $p_s < 0.018$).

In order to directly test the null hypothesis, a series of planned contrast analyses were performed (detailed results are presented in Table 3). Firstly, the study focused on the IPS e.max ZirCAD samples and their bond strength to Panavia V5 was compared to that of Panavia SA, RelyX U200, and Maxcem, respectively. All the planned contrasts were statistically significant, indicating that compared to Panavia V5, the other types of resin cements examined were characterized by a significantly higher bond strength (in ascending order: Maxcem, Panavia SA, RelyX U200). The analogous analysis performed for the IPS Empress CAD samples also showed that all the planned contrasts were statistically significant. In this case, Panavia V5 was characterized by a significantly higher bond strength compared to the other types of resin cements (in descending order: Panavia SA, Maxcem, RelyX U200). In the last series of planned contrasts, the study focused on the IPS e.max CAD samples and their bond strength to Panavia V5 was compared to that of other types of cements. As observed in the other cases, all the planned contrasts were statistically significant. Compared to Panavia V5, all the other types of cements were characterized by a significantly lower bond strength (in descending order: Panavia SA, Maxcem, RelyX U200).

3.2. Analysis of failure mode under a light microscope

Representative photographs obtained in the analysis of failure mode are presented in Figs. 3 (photographs of ceramic surfaces) and 4 (photographs of dentin surfaces), and the quantitative results are presented in Table 4. In the case of all the self-adhesive cements studied (Panavia SA, Maxcem, RelyX U200), the predominant failure mode was the failure of adhesion between the cement and dentin was. However, for Panavia V5, different modes of failure were observed for different ceramics used: for IPS e.max ZirCAD ceramic, the failure mode identified was failure of adhesion between dentin and cement or ceramic and cement; for IPS Empress CAD, the mode was failure of cohesion in cement or mixed failure; and for IPS e.max CAD, it was failure of adhesion between ceramic and cement.

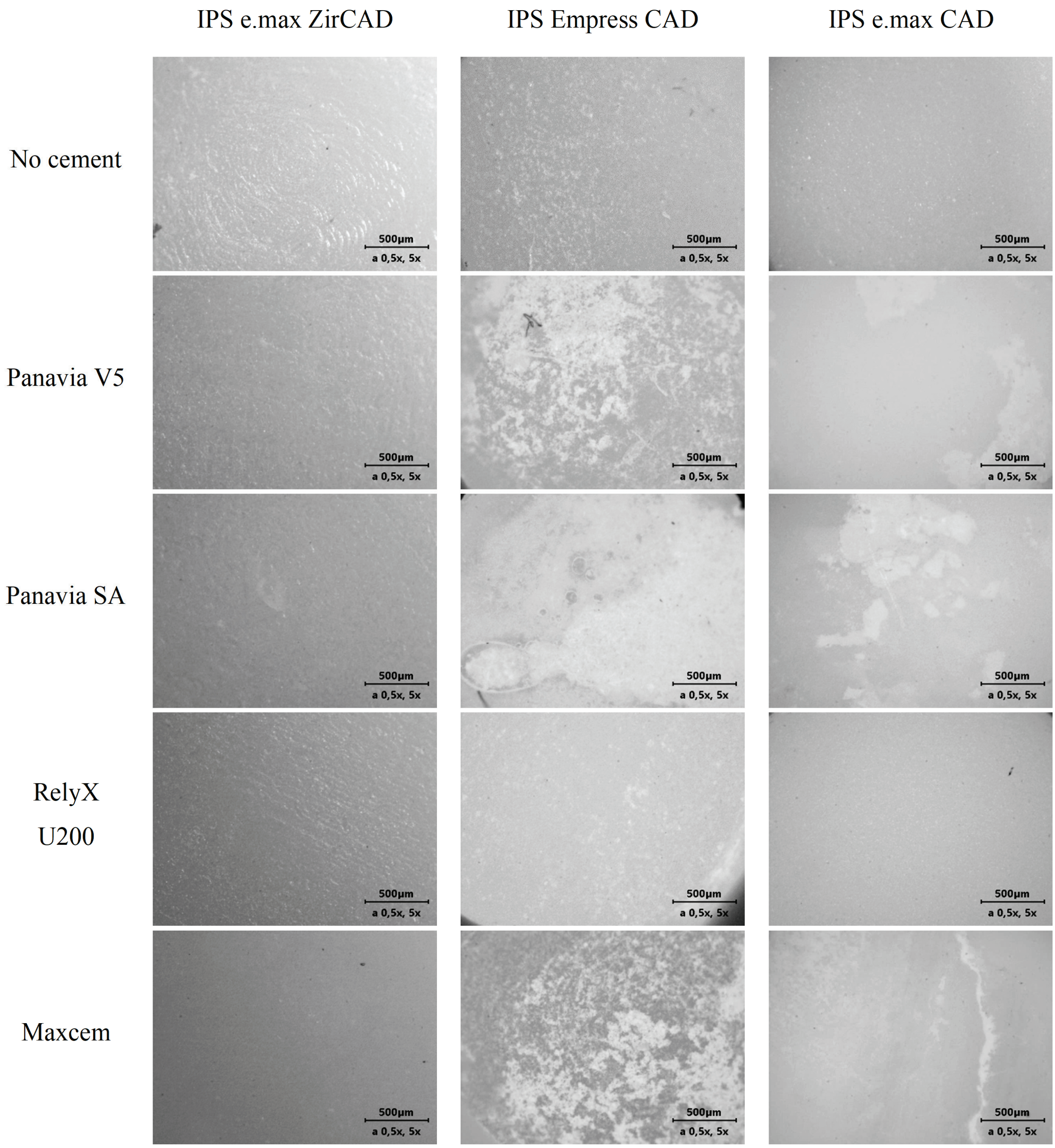

Fig. 3. Light microscope photographs of different failure modes observed on ceramic surfaces

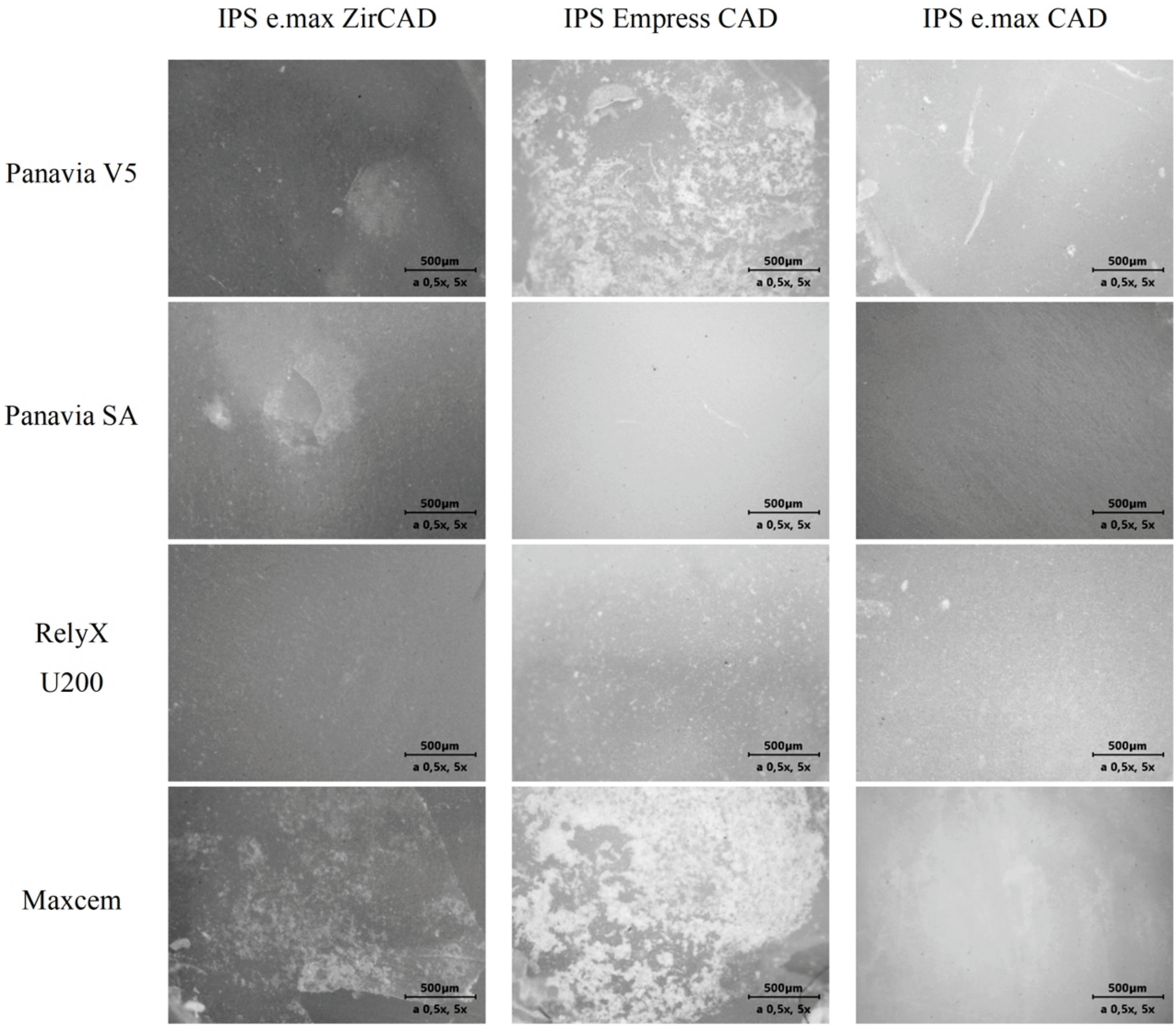

Fig. 4. Light microscope photographs of different failure modes observed on dentin surfaces

Table 4. Quantitative results of the analysis of failure mode

Ceramic	Resin cement	Failure mode [%]			
		Adhesive failure at dentin/cement interface	Adhesive failure at ceramic/cement interface	Cohesive failure in cement	Mixed failure
IPS e.max ZirCAD	Panavia V5	50	50	0	0
	Panavia SA	75	25	0	0
	RelyX U200	100	0	0	0
	Maxcem	91.66	8.34	0	0
IPS Empress CAD	Panavia V5	0	0	50	50
	Panavia SA	83.33	0	0	16.67
	RelyX U200	75	0	25	0
	Maxcem	75	0	25	0
IPS e.max CAD	Panavia V5	0	91.66	0	8.34
	Panavia SA	100	0	0	0
	RelyX U200	83.33	0	0	16.67
	Maxcem	91.66	0	8.34	0

4. Discussion

Due to their very good biocompatibility, esthetics, and cost-effectiveness, CAD/CAM ceramics are widely gaining attention for application in permanent prosthetic reconstructions. The clinical success of a bonded ceramic restoration strongly depends on its durable bonding to dentin, ability of optimal marginal adaptation after cementation, and overall strength [3], [12], [24].

In order to achieve a strong bonding of a ceramic material to the supporting tooth structure, it is necessary to apply an appropriate cement. The new-generation self-adhesive self-etching resin cements seem to be an ideal option for this purpose since they do not need a separate etching, priming, or bonding process, and are, therefore, easier to handle and less technique-sensitive. However, a previous study indicated that self-adhesive resin cements have a lower bonding ability compared to adhesive cements [18].

The present study revealed that, in general, ceramics cemented to dentin using the conventional Panavia V5 cement displayed a significantly higher shear bond strength than those cemented using the self-adhesive self-etching cements. Similar results were reported in the study by Ab-Ghani et al. [1], which demonstrated that the multistep etch-and-rinse adhesive bonding (Variolink II cement) of CAD/CAM ceramics to dentin was characterized by a statistically significantly higher shear bond strength than the bonding achieved using the self-adhesive cement (RelyX U200). In addition, Gundogdu et al. [8] revealed that the self-etch adhesive resin cements exhibited a higher shear bond strength than the self-adhesive resin cements. Their study was performed following the ISO/TS 11405:2003 standard, but instead of the CAD/CAM ceramics, they used two types of pressable ceramic materials (IPS e.max Press and Prettau Zirconia). The lower shear bond strength of the self-adhesive resin cements noted in the study may be explained by the fact that this type of material interacts superficially with mineralized tissue and cannot demineralize the dentin completely. Thus, the smear layer cannot be completely removed, and hence, it is impossible to achieve the full formation of resin tags in the hybrid layer [5], [13], [20], [24].

Nevertheless, in the analysis of the bonding ability of the individual cements to the individual ceramics in the current study, one of the ceramics – IPS e.max ZirCAD – was found to show the highest adhesion to dentin (22.74 MPa) after cementation with RelyX U200 among all the cements tested, even higher than that observed after cementation with Panavia V5. In a study by Lee et al. [14], the shear bond strength of the combination of zirconia (Zirtooth, HASS, Kangneung, Korea) and RelyX U200 was reported at an average level of 2.84 MPa. Moreover, the shear bond strength measured by Lee et al. [14] for another resin cement – Maxcem Elite – was 2.86 MPa, while in the present study it was 13.29 MPa. The discrepancies in the obtained results may be due to the use of different ceramic materials and different methods for the preparation of zirconia samples.

One of the possible reasons for the differences in the measured bonding forces of the selected cements can be attributed to the different compositions of these materials. Additionally, Arango et al. [2] reported that the shear bond strength of the cements was dependent on the nature of the prosthodontic substrate. In the present study, three CAD/CAM ceramics, differing in composition and crystal structure, were studied. IPS e.max ZirCAD MO is a 3Y-TZP generation of dental zirconia (yttria-stabilized tetragonal zirconia polycrystal) which has a typical crystal size of 0.5 μm and a crystal phase volume of 98% [9]. IPS e.max CAD is a glass ceramic containing lithium disilicate crystals at a volume of approximately 70% and a typical size of 1.5 μm [10]. IPS Empress CAD is a leucite-reinforced glass ceramic with a crystal phase volume of 35–45% and a crystal diameter of 1–5 μm [11]. In this study, it was found that, notwithstanding the dental cements, IPS Empress CAD had the highest bond strength. This observation can be explained by the biggest size of the crystals present in this material.

The analysis of failure mode revealed that in the case of all the studied ceramics luted with self-adhesive cements, the most frequently noted mode of failure was adhesive failure at the dentin/cement interface. The dentin/resin cement interface was weaker than the resin cement/ceramic interface, which corresponds to the observations described previously by the other researchers and could be explained by the limited ability of the self-adhesive cements to demineralize dentin [8], [17]–[19], [25]. In order to improve the bonding of self-adhesive resin cements to dentin, either the dentin should be properly pretreated or the chemistry of the cements should be modified [6].

The current study was performed in laboratory conditions in order to determine some basic relationships, however, the associated simplifications should be first understood. One of the limitations is related to the simplified structure of the tested samples, since only one surface of dentin was cemented, whereas in the case of crowns or bridges the restoration is cemented into five surfaces of the prepared teeth. Additionally, in further studies, some important phenomena, such as the degradation of resin cements after long periods of exposure to oral fluids and cyclic mechanical fatigue occurring during chewing should also be taken into account in order to simulate the clinical conditions more realistically. In a study by Sathish et al. [21], it was demonstrated that thermocycling, which was used for artificial aging, affected the bond strength of the different resin cements used. Moreover, additional physical properties, such as tensile strength and flexural strength, should be examined. Application of several methods of surface treatment, such as hydrofluoric acid etching, silanization, and treatment with adhesives or laser, that can improve the bond strength of dental restorations, is also worth investigating [3]. In the future, the preliminary results reported in this research should be verified by a clinical study evaluating the *in vivo* performance of the self-adhesive resin cements.

5. Conclusions

The present study revealed significant differences between the shear bond strengths of the CAD/CAM ceramics luted to dentin using different self-adhesive and conventional resin cements, and so the tested null hypothesis was rejected.

The following conclusions can be drawn on the basis of the obtained results:

1. Notwithstanding the dental ceramic, the examined cements differed in the bond strengths; the highest bond strength was observed in Panavia V5, lower – in RelyX U200 and Panavia SA, and the lowest – in Maxcem.
2. Similarly, notwithstanding the dental cements, the examined ceramics differed in the bond strengths; the highest bond strength was observed in IPS Empress CAD, lower – in IPS e.max ZirCAD, and the lowest – in IPS e.max CAD.
3. The bond strength depends on the combination of ceramic and cement. IPS e.max ZirCAD displayed the highest bond strength to dentin after cementation with RelyX U200, while IPS Empress CAD and IPS e.max CAD showed the highest strength after cementation with Panavia V5.
4. In the case of all the studied self-adhesive cements (Panavia SA, Maxcem, RelyX U200), the predominant mode of failure observed was the failure of adhesion between the cement and dentin.

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Effect of Thermocycling on the Bond Strength of Self-Adhesive Resin Cements Used for Luting CAD/CAM Ceramics to Human Dentin

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Abstract: The aim of the study was to evaluate the influence of thermocycling on the shear bond strength of self-adhesive, self-etching resin cements luted to human dentin and computer-aided design/computer-aided manufacturing (CAD/CAM) ceramics. Three modern self-adhesive dental cements (Maxcem Elite, RelyX U200, Panavia SA) were used to lute three CAD/CAM ceramics (IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD) onto the dentin. One conventional cement (Panavia V5) served as a control. After preparation, the samples were subjected to thermocycling as a method of artificial aging of dental materials applied to simulate long-term use in oral conditions. Shear bond strength was evaluated according to PN-EN ISO 29022:2013-10 and failure modes were observed under a light microscope. Statistical analysis was performed. The study demonstrated that a combination of ceramics and cements directly impacts the bond strength. The highest bond strength was observed in Panavia V5, lower in Panavia SA and Maxcem Elite and the lowest–in RelyX U200. Adhesive failure between human dentin and cements was the most common failure mode. Moreover, thermocycling highly decreased bond strength of self-adhesive, self-etching cements.

Keywords: shear bond strength; self-adhesive resin cement; adhesion; luting agents; tooth; dentin; CAD/CAM ceramic; thermocycling; artificial aging

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1. Introduction

The use of all-ceramic restorations in modern dental prosthetics is still increasing due to the development of innovative ceramic materials and computer-aided design/computer-aided manufacturing (CAD/CAM) technologies. One of the most important clinical aspects of prosthetic ceramic reconstruction is providing a suitable bond strength between ceramic surface and tooth tissues [1–3]. The strong adhesion achieved due to the application of proper cements directly translates into the longevity of the prosthetic restoration, especially in the case of minimally invasive restorations such as veneers, inlays or overlays [4]. In particular, stable cementation of ceramic materials strengthens the marginal adaptation, protects against micro-leakage and significantly reduces the risks of cracking and fracturing of the restoration [5,6]. As a result, it is crucial for functional properties of the restoration, influencing both its mechanical and biological behavior.

The choice of cement is one of the most important steps to obtain a strong bond between dental ceramics and the hard tissues of the teeth [3–6]. The use of conventional resin-based cements with well-developed methods of surface modification allows us to obtain a high-quality bond of ceramic restoration to a tooth [7–10]. However, due to the multitude of surface modification techniques, duration and complexity of the procedure, as well as the dependence on the operator skills, simplification of the methodology has become necessary to reduce the risk of error. For this reason, contemporary technology, in order to meet the growing expectations, has developed self-etching, self-adhesive cements.

Using 10-methacryloyloxydecyl dihydrogen phosphate (MDP) or long carbon-chain silane (LCSi) coupling agent monomers in modern self-adhesive, self-etch cements is claimed to ensure high durability of the joined surfaces and elimination of additional steps needed to obtain proper bond strength of the prosthetic reconstruction to the tooth tissues [11,12]. On the other hand, research shows large discrepancies in the bond strengths of modern self-adhesive cements based on MDP monomers, particularly in the cases when they are used for cementation of zirconium materials [8–13]. The selection of an appropriate cementing protocol or modifications of ceramic surface directly affects the obtained bond strength results [14–16].

Our previous research evaluated the potential of self-adhesive, self-etching cements for the cementation of the selected CAD/CAM ceramic materials from various groups (glass ceramics, lithium disilicate and zirconium) [17]. The obtained shear forces depended on the selection of cement in relation to the cemented ceramic material. The study revealed much a higher bond strength of conventional cement than self-adhesive, self-etching cements. To obtain a broader view concerning long-term behavior, the bond strength analysis of dental materials should also apply some type of accelerated aging method [14–20].

The artificial decrease of bond strength over time better reflects the real behavior and characteristics of the cement and, therefore, is necessary during in vitro studies. However, a single optimal method of accelerated aging perfectly mimicking intraoral conditions has not yet been developed. Various approaches and protocols have been described, based on thermocycling, thermomechanical aging, dynamic load or water storage [18–23]. In the current study, we applied thermocycling as one of the most common methods of artificial aging of dental materials [5,14]. Most of the research follows the temperature range of 5–55 °C defined in ISO/TS 11405, but they differ in terms of dwell time and number of cycles performed [5,6,14,16].

The present study aimed to assess the shear bond strength of self-adhesive, self-etching resin cements used for luting CAD/CAM ceramics to human dentine when then samples were subjected to artificial aging (thermocycling). The following research hypothesis was tested: there is no significant difference between the shear bond strength of the modern self-adhesive resin cements and the conventional luting agent (Panavia V5). The second hypothesis was formulated as follows: there is no significant difference between the shear bond strength obtained for thermocycled and non-thermocycled samples.

2. Results

2.1. Shear Bond Strength between Ceramic and Dentin

The four types of dental cement differed (regardless of the ceramics type) in the shear bond strength ($F(3, 44) = 5763.53$, $p < 0.0001$, $\eta^2 = 0.997$). The highest shear bond strength was observed in Panavia V5 (M = 14.63); this was lower in Panavia SA (M = 2.91) and RelyX U200 (M = 2.82) and the lowest in Maxcem (M = 2.58). The post-hoc Tukey's HSD test showed statistically significant differences between Panavia V5 and all other cements (all p's < 0.0002), as well as between Panavia SA and Maxcem ($p = 0.024$). The differences between RelyX U200 and Panavia SA ($p = 0.83$) and Maxcem ($p = 0.17$) were not statistically significant.

Regardless of the dental cements used, the examined ceramics differed in their shear bond strengths ($F(2, 88) = 467.81$, $p < 0.0001$, $\eta^2 = 0.91$). The highest shear bond strength was observed in IPS e.max CAD (M = 6.96); this was lower in IPS Empress CAD (M = 6.64) and the lowest in IPS e.max ZirCAD (M = 3.61). The Tukey's post-hoc tests showed that all differences between ceramics were statistically significant (IPS Empress CAD vs. IPS e.max ZirCAD: $p = 0.0001$; IPS Empress CAD vs. IPS e.max CAD: $p = 0.02$; IPS e.max ZirCAD vs. IPS e.max CAD: $p = 0.0001$).

The statistically significant effect of the interaction between ceramics and cement ($F(6, 88) = 297.84$, $p < 0.0001$, $\eta^2 = 0.95$) indicated that the shear bond strength depends on the combination of both materials (Figure 1 and Table A1). The shear bond strength for Panavia V5 and Panavia SA increased from IPS e.max ZirCAD through IPS Empress CAD to IPS e.max CAD. For RelyX U200, the shear bond strength increased from IPS e.max ZirCAD through IPS e.max CAD to IPS Empress CAD. In the case of Maxcem, that lowest shear bond strength was observed of IPS e.max CAD, higher for IPS e.max ZirCAD and the highest for IPS Empress CAD. The post-hoc analysis by Tukey's HSD test showed that 20 (out of 66) pairwise comparisons did not reach the statistical significance level (all p's ≥ 0.14). The differences in shear bond strength between all other groups were statistically significant (all p's ≤ 0.016). Detailed results of the post-hoc analysis are presented in Table A2.

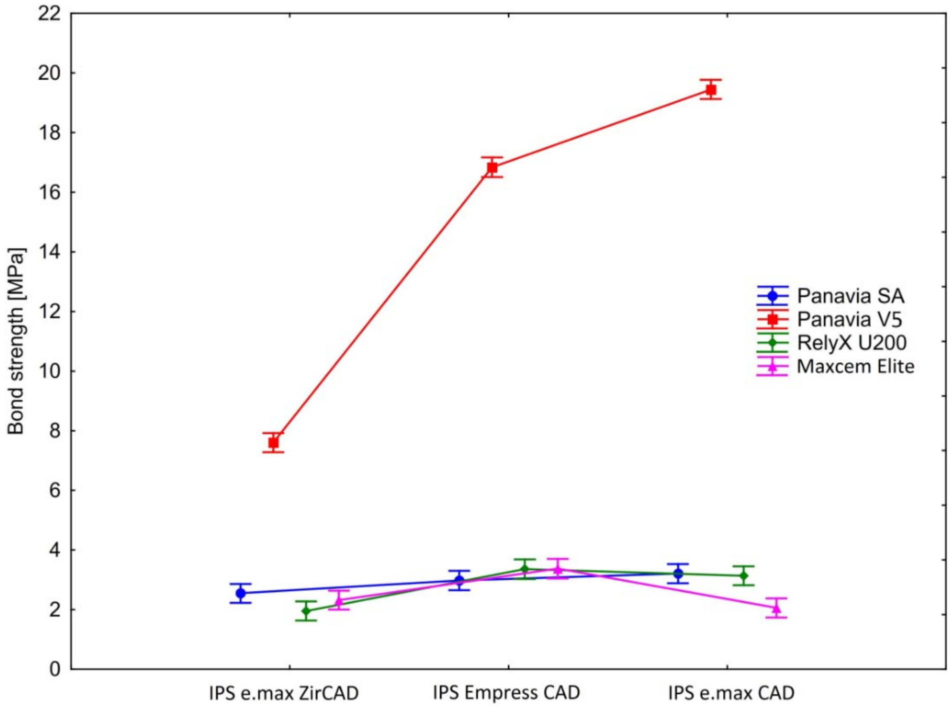

Figure 1. Shear bond strength in the examined samples. Vertical bars denote 0.95 confidence intervals.

In order to directly test our research hypothesis, we ran a series of planned contrast analysis (detailed results are presented in Table 1 and Figure 1). Firstly, we focused on IPS e.max ZirCAD samples and compared shear bond strength in Panavia V5 to Panavia SA, RelyX U200 and Maxcem, respectively. All planned contrasts were statistically significant, indicating that, when compared to Panavia V5, other examined types of cement were characterized by significantly lower shear bond strength (in descending order: Panavia SA, Maxcem, RelyX U200). Analogous analysis for IPS Empress CAD samples also showed that all planned contrasts were statistically significant. In this case, the Panavia V5 was characterized by the highest shear bond strength and statistically significantly different than other types of cement (in descending order: Maxcem, RelyX U200, Panavia SA). In the last series of planned contrasts, we concentrated on the IPS e.max CAD samples and compared the shear bond strength in Panavia V5 to other types of cement. As in the previous cases, all planned contras were statistically significant. Comparing to Panavia V5 all other types of cement were characterized by significantly lower shear bond strength (in descending order: Panavia SA, RelyX U200, Maxcem).

Table 1. Planned contrast analysis for differences in the shear bond strength between Panavia V5 and Panavia SA, RelyX U200, Maxcem respectively. The Cohen's *d* for groups with equal size was calculated according to Lenhard, W. & Lenhard, A [24].

	IPS e.max ZirCAD			IPS Empress CAD			IPS e.max CAD		
Panavia V5	***t* (44)**	***p***	**Cohen's *d***	***t* (44)**	***p***	**Cohen's *d***	***t* (44)**	***p***	**Cohen's *d***
Panavia SA	22.61	<0.0001	7.542	60.07	<0.0001	18.221	72.13	<0.0001	28.188
RelyX U200	25.26	<0.0001	8.638	58.41	<0.0001	21.586	72.46	<0.0001	28.135
Maxcem	23.63	<0.0001	9.496	58.35	<0.0001	21.783	77.25	<0.0001	34.233

2.2. Microscopic Evaluation of a Failure Mode

The microscopic photographs of the sheared samples surfaces are presented in Figure 2 (surfaces of ceramic cylinders) and in Figure 3 (surfaces of human dentin). Quantitative results of the failure mode analysis were summarized in Table 2. It was found that the adhesive failure between dentin and cement were predominant mode among the self-etching, self-adhesive cements with the selected ceramics. Application of Panavia V5 cement in case of IPS Empress CAD ceramics most often resulted in a mixed failure. However, in the case of IPS e.max ZirCAD and IPS e.max CAD, an adhesive failure at cement/dentin interface was a predominant mode, similarly to the studied self-adhesive, self-etching cements (Table 2).

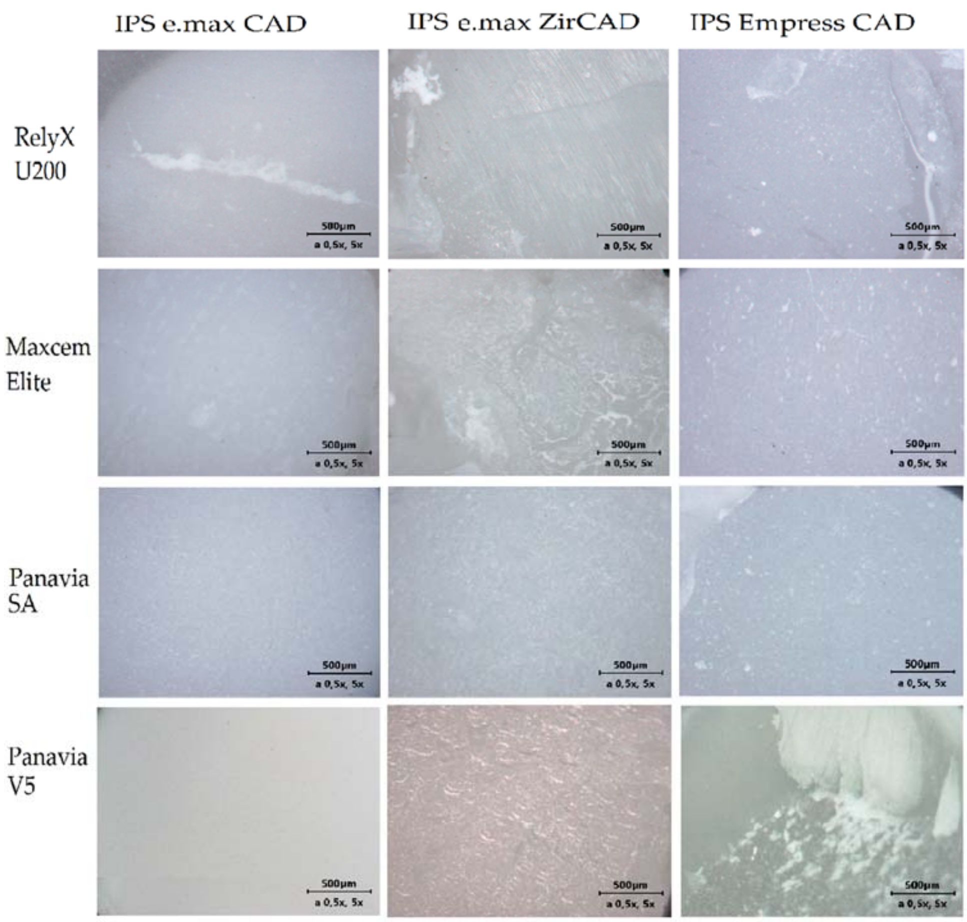

Figure 2. Light microscope photographs of different failure modes observed on ceramic surfaces.

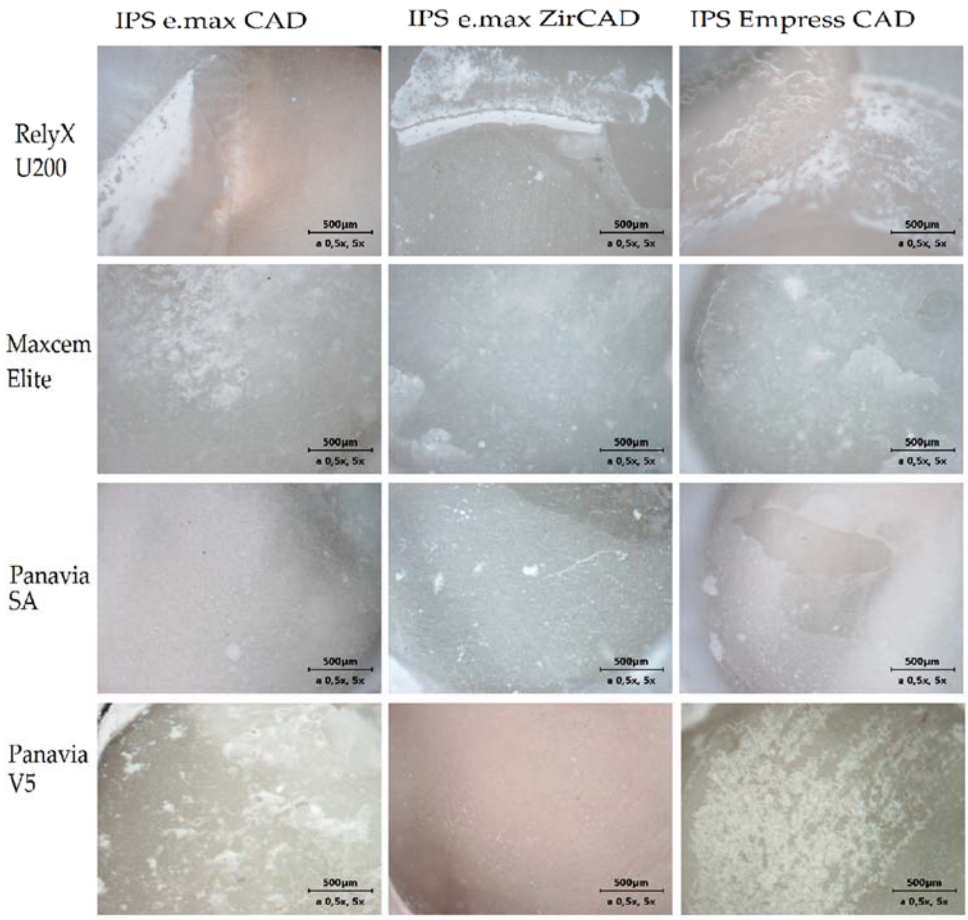

Figure 3. Light microscope photographs of different failure modes observed on dentin surfaces.

Table 2. Quantitative results of failure mode analysis.

Ceramic	Resin Cement	Failure Mode [%]			
		Adhesive Failure at Dentin/Cement Interface	Adhesive Failure at Ceramic/Cement Interface	Cohesive Failure in Cement	Mixed Failure
IPS Empress CAD	RelyX U200	58.4	-	-	41.6
	Maxcem Elite	66.7	-	33.3	-
	Panavia SA	75	-	25	-
	Panavia V5	-	-	33.3	66.7
IPS e.max CAD	RelyX U200	75	-	8.3	16.7
	Maxcem Elite	83.3	-	-	16.7
	Panavia SA	75	-	25	-
	Panavia V5	58.4	-	-	41.6
IPS e.max ZirCAD	RelyX U200	50	-	25	25
	Maxcem Elite	66.7	16.6	-	16.6
	Panavia SA	50	25	-	25
	Panavia V5	58.4	41.6	-	-

2.3. Comparison of Shear Bond Strength for Thermocycled and Non-Thermocycled Samples

The results of the current study were compared with the results of the previous research carried out using exactly the same materials and methodology, but without thermal accelerated aging [17].

The bond strength after thermocycling was statistically significantly weaker in all samples. This observation applies to all four types of cement on three ceramics. As shown by the calculated effect sizes, the magnitudes of the observed differences in all cases were substantial, indicating the large effect of aging (thermocycling) on the bond strengths. Detailed results are presented in Table 3 and Figures 4–6.

Table 3. Statistical comparation of shear bond strength for thermocycled and non-thermocycled samples.

	Without Thermocycling		With Thermocycling		Bond Strength Decline		*t*-Test (df = 11)		Effect Size
Cement	**M (MPa)**	**SD**	**M (MPa)**	**SD**	**M (MPa)**	**SD**	***t*-Value**	***p*-Value**	**Cohen's d_z**
					IPS e.max ZirCAD				
Panavia SA	18.31	1.357	2.55	0.558	15.77	1.424	38.36	<0.0001	11.07
Panavia V5	11.23	0.475	7.60	0.765	3.632	0.875	14.37	<0.0001	4.15
RelyX U200	22.73	1.148	1.95	0.520	20.78	1.196	60.18	<0.0001	17.37
Maxcem	13.29	1.185	2.32	0.182	10.97	1.212	31.35	<0.0001	9.05
					IPS Empress CAD				
Panavia SA	16.94	1.533	2.98	0.667	13.96	1.924	25.13	<0.0001	7.25
Panavia V5	20.33	0.787	16.84	0.844	3.488	1.041	11.61	<0.0001	3.35
RelyX U200	15.13	1.953	3.36	0.260	11.77	1.895	21.51	<0.0001	6.21
Maxcem	15.48	1.158	3.37	0.229	12.11	1.274	32.92	<0.0001	9.50
					IPS e.max CAD				
Panavia SA	9.888	0.926	3.209	0.588	6.679	1.162	19.91	<0.0001	5.75
Panavia V5	22.50	0.690	19.45	0.564	3.059	0.952	11.13	<0.0001	3.21
RelyX U200	8.228	0.696	3.135	0.595	5.092	0.908	19.42	<0.0001	5.61
Maxcem	8.428	0.867	2.058	0.445	6.370	0.739	29.87	<0.0001	8.62

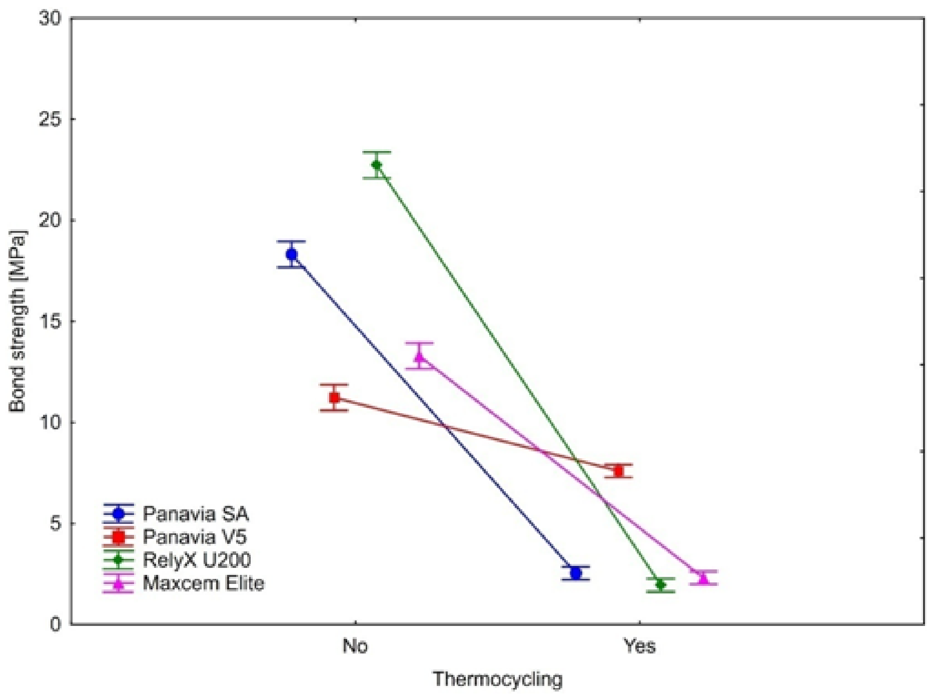

Figure 4. The effect of thermocycling on luting cement bond strength to IPS e.max ZirCAD. Vertical bars denote 0.95 confidence intervals around the mean values.

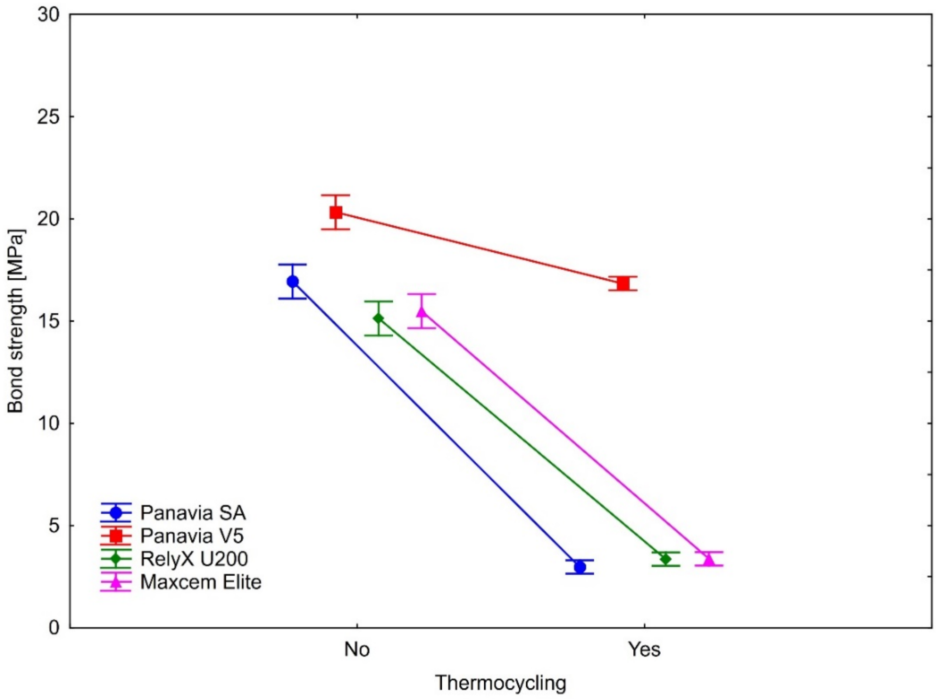

Figure 5. The effect of thermocycling on luting cements bond strength to IPS Empress CAD. Vertical bars denote 0.95 confidence intervals around the mean values.

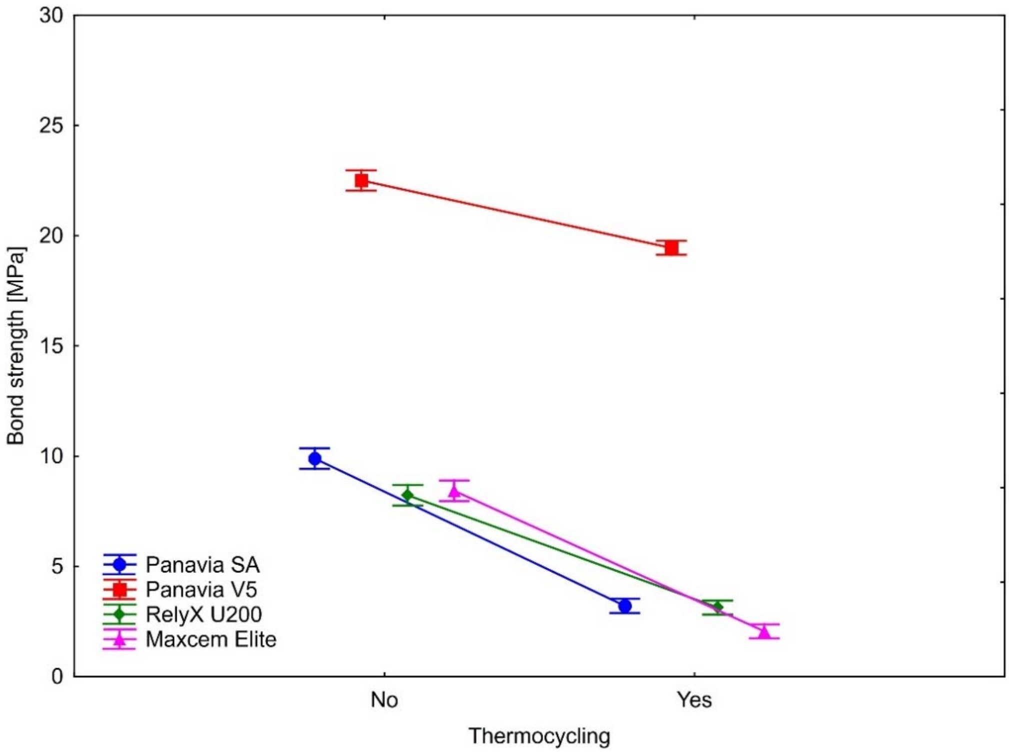

Figure 6. The effect of thermocycling on luting cements bond strength to IPS e.max CAD. Vertical bars denote 0.95 confidence intervals around the mean values.

Complimentary analyses showed that thermocycling affected the magnitude of changes in the bond strength differently depending on the type of cement. This effect was statistically significant for all three ceramics (IPS e.max ZirCAD: $F(3, 44) = 449.387$, $p < 0.0001$, $\eta^2 = 0.97$); IPS Empress CAD: $F(3, 44) = 104.358$, $p < 0.0001$, $\eta^2 = 0.88$); IPS e.max CAD: $F(3, 44) = 35.795$, $p < 0.0001$, $\eta^2 = 0.71$).

In the case of IPS e.max ZirCAD, the lowest decline in the bond strength was observed for Panavia V5, larger in Maxcem and Panavia SA. The largest drop in the bond strength was observed for RelyX U200 (Table 3 and Figure 4). Tukey's post-hoc tests showed that the differences in the bond strength decline were statistically significant between all cement types (all p's = 0.0002).

Similarly, for the IPS Empress CAD, Panavia V5 had the lowest decline in the bond strength (Table 3 and Figure 5). Larger decline values were observed (in ascending order) for RelyX U200, Maxcem and Panavia SA. While the RelyX U200 and Maxcem were not statistically significantly different in the magnitude of bond strength decline ($p = 0.95$), the other comparisons showed that the magnitudes of change in the bond strength were statistically significant between analyzed types of cement (Panavia SA vs. Panavia V5: $p = 0.0002$; Panavia SA vs. RelyX U200: $p = 0.008$; Panavia SA vs. Maxcem: $p = 0.03$; Panavia V5 vs. RelyX U200: $p = 0.0002$; Panavia V5 vs. Maxcem: $p = 0.0002$).

In the case of IPS e.max CAD, the pattern of changes in the bond strength were analogous as in IPS Empress CAD. Again, the lowest decline was observed for Panavia V5. Larger values were observed (in ascending order) for RelyX U200, Maxcem and Panavia SA (Table 3 and Figure 6). The magnitude of the bond strength declines did not statistically significantly differ between Panavia SA and Maxcem ($p = 0.86$). Statistically significant differences were observed between other types of cement (Panavia SA vs. Panavia V5: $p = 0.0002$; Panavia SA vs. RelyX U200: $p = 0.001$; Panavia V5 vs. RelyX U200: $p = 0.0002$; Panavia V5 vs. Maxcem: $p = 0.0002$; RelyX U200 vs. Maxcem: $p = 0.01$).

3. Discussion

The essence of the clinical long-term success of all-ceramic prosthetic restoration is not only related to the mechanical strength of ceramic, but also to the durable and strong adhesion of ceramics material to the tooth's hard tissues provided by the proper cementation. A strong need for a reliability and resistance to the oral cavity environment is a particularly crucial aspect of minimally invasive reconstructions [3,7–9]. Especially the durability of veneers, overlays, inlays, etc., directly depends on the surface adhesion [7]. Durable ceramic-tooth tissue connection relies on chemical bonding and micromechanical interlocking [12,15].

Nowadays, the subject of adhesion of modern high-strength ceramics, and also those processed in digital CAD/CAM technologies, is intensively researched in many aspects. A large number of studies focuses on the modification of the ceramic surface in order to obtain the highest possible bond strength [1–8,12–15]. In opposition to this point of view is the approach emphasizing the potential of the use of self-adhesive self-etching cements, which significantly reduces the number of steps involved in preparation of the surfaces to be joined and, as a result, also reduces possible operator errors [19]. Despite these obvious advantages, the possible risks associated with the use of self-adhesive cements still require investigation, particularly in terms of its effect on the long-term durability of restoration.

To simulate the wear of dental materials over time, artificial aging should be performed and its effect must be assessed, e.g., during the evaluation of the strength of adhesive materials applied between tooth tissue and ceramics. These challenges remain since there is no unified protocol for simulating accelerated aging. The literature describes dynamic simulations [23,25,26] or the storage of samples in water baths at constant temperature [6,14,26]. Thermocycling is one of the most common methods to age the material; however, its parameters have not yet been unified. Comino-Garayoa et al. conducted a systematic review analyzing 45 different papers, concluding that artificial aging should be based on 5000 thermal cycles or 30 days of continuous water bath [26]. Other systematic reviews [5,9,10,26] comparing parameters of thermocycling enable us to draw the general conclusion that a gradient of temperatures from 5 °C to 55 °C should be applied. These temperatures are also in accordance with the ISO TS 11405 Technical Specification for testing the adhesion to tooth structure. On the other hand, there are still significant differences in the number of cycles performed, suggesting that this is a prediction-based parameter [10,22,23].

Our study revealed that thermocycling significantly reduced a bond strength of dental cements. The performed statistical analysis showed that the bond strengths of self-etching, self-adhesive cements were strongly dependent on the use of this method of artificial aging (or not). Noticeable, though much smaller, decrease of bond strength was also demonstrated for the conventional Panavia V5 cement used as a control group.

Results of different preclinical and clinical studies concerning self-adhesive, self-etch cements used as a luting agent for prosthetic restorations revealed that there is no doubt that conventional resin cement with proper surface modification provides better dentin bonding and sealing performance [22]. However, not only focusing on the stabilization of the restoration, but also effective and durable bonding to dentine is still a fundamental prerequisite for clinical success when using self-adhesive resin cements for the luting of ceramic restorations [21]. Some in vitro studies proved that bonding with self-adhesive, self-etch cements might be beneficial for the tooth tissue, as they may be less toxic than conventional agents [21]. On the other hand, this is in contrast to the results of Sawada et al., in which no significant differences were observed between the self-adhesive, self-etch bonding agents [13,27].

As the methods of research sample preparation and their bond strength evaluation presented in the literature differ significantly, it is impossible to conduct a reliable meta-analysis [27–31]. The methodology described in this study is standardized based on ISO guidelines, which provide an opportunity for their reproduction. However, the main limitation is connected with the simple method of artificial aging applied. Further research is required, taking other methods into account. Generally, it must be taken into account that resin-based luting cements, similarly as other materials used for the reconstruction of teeth hard tissues, must withstand a number of factors directly and indirectly affecting the strength and stability of the connection. It should be noted that the oral cavity environment is not only affected by temperature variations. Changes in humidity, pH, saliva enzymes effects or physical forces acting in three axes also significantly influence the maintenance of the reconstruction [19]. Shahin et al. used accelerated aging based on combined thermocycling and the dynamic loading of samples material, which seems to better reflect the oral cavity conditions [16]. Following this direction would be a valuable continuation for our current research.

4. Materials and Methods

The current study strictly followed a methodology applied in the previous research [17]. In this section, only a brief description is presented.

4.1. Samples Prepration

Detailed description of the materials used in this study is included in Table 4. Four resin cements were used: Panavia SA, RelyX U200, Maxcem Elite (self-etching, self-adhesive cements) and Panavia V5 (a conventional resin cement used as a control). Three types of ceramics were selected: IPS Empress CAD, IPS e.max CAD, IPS e.max ZirCAD. For each combination of cement and ceramics 12 samples were prepared. Following the PN-EN ISO 29022: 2013-10 standard, 144 ceramic cylinders with a diameter of 2.38 mm and a height of 5 mm were designed and milled in CAD/CAM technology using the Sirona Cerec inLAB SW 19.0 system (Sirona, New York, NY, USA). The cylinders were cemented into human dentin slices obtained from 67 freshly extracted, caries-free human molars (approved by Wroclaw Medical University Bioethical Committee, No. KB-37/2018). For this purpose, a PetroThin Thin Sectioning System with diamond disc and water cooling (Buehler, Lake Bluff, IL, USA) was used to cut the coronal dentin into 3 mm thick slices. Before cementing the ceramic cylinder, each prepared dentin specimen was grounded with a carborundum paper of P 400 granularity (Luna, Bern, Switzerland).

Table 4. Ceramics and resin cement manufacturers.

Name	Type	Manufacturer
Resin Cements		
RelyX U200 A1	Self-adhesive, self-etch	3M ESPE (Maplewood, MN, USA)
Maxcem Elite A1	Self-adhesive, self-etch	Kerr (Brea, CA, USA)
Panavia SA Cement Universal A1	Self-adhesive, self-etch	Kuraray Noritake (Tokyo, Japan)
Panavia V5 A1	Adhesive	Kuraray Noritake (Tokyo, Japan)
Ceramics		
IPS Empress CAD HT A1	Leucite glass	Ivoclar Vivadent (Schaan, Liechtenstein)
IPS e.max CAD HT A1	Lithium disilicate	Ivoclar Vivadent (Schaan, Liechtenstein)
IPS e.max ZirCAD	Zirconia	Ivoclar Vivadent (Schaan, Liechtenstein)

The cementation of ceramic cylinders onto human dentine was carried out in accordance with the manufacturer's guidelines. For self-etching, self-adhesive cements, only the modification of the ceramic surface was performed. The IPS Empress CAD and IPS e.max CAD ceramics were etched with 9% hydrofluoric acid (3M ESPE, Maplewood, MN, USA) for 1 min. The surface of the IPS e.max ZirCAD ceramic was pretreated using CoJet System (3M ESPE, Maplewood, MN, USA). Conventional Panavia V5 cement required additional modification of dentin surfaces with 37% orthophosphoric acid (3M ESPE, Maplewood, MN, USA). Cementation of ceramics onto dentin was carried out with a fixed compression force of 10 N under the control of the FB(C) dynamometer (Axis, Gdansk, Poland). Polymerization was performed using Elipar LED lamp (3M ESPE, Maplewood, MN, USA) for 20 s. Before performing simulated artificial aging, the prepared samples were stored in distilled water at 37 °C for 24 h.

4.2. Artificial Aging

The process of artificial aging of the samples was performed using THE-1100 thermocycler (SD Mechatronik, Munich, Germany). Each specimen was subjected to 2000 cycles in temperatures between 5 °C and 55 °C with a dwell time of 40 s and transfer time of 15 s. Immediately after completion of accelerated aging, the samples were sheared.

4.3. Evaluation of the Shear Bond Strength between Ceramic and Dentin

The shear bond strength tests were carried out using a universal testing machine (Thumler, Nurnberg, Germany), following PN-EN ISO 29022:2013-10 standard, with the crosshead speed of a 1-mm/min and a maximum force of 3000 N. Preparation of samples, thermocycling and shear bond strength test were presented in Figure 7.

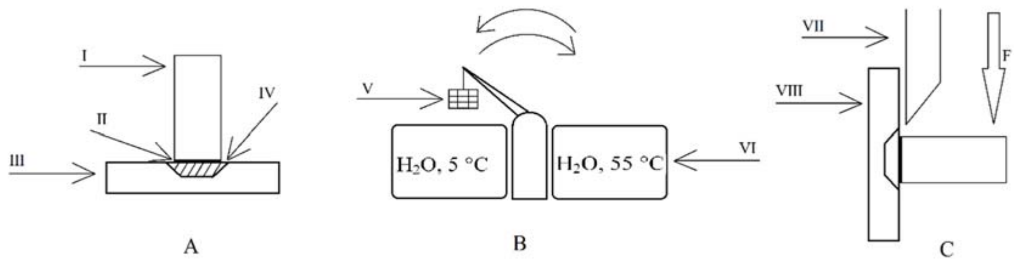

Figure 7. Schematic illustration of the sample preparation, thermocycling and shearing: (**A**) ceramic cylinder cemented onto human dentin embedded in an acrylic retainer; (**B**) thermocycling; (**C**) shear bond strength measurement; I-ceramic cylinder; II-resin luting cement. III and VIII-acrylic dentin slice retainer; IV-slice of human dentine; V-basket with thermocycled samples; VI-distilled water baths; VII-shear knife of the universal testing machine.

4.4. Microscopic Evaluation of a Failure Mode

Failure modes were observed under light microscope (Axio Lab. A1 MAT, Zeiss, Oberkochen, Germany) with ×5 magnification. For each sample, the type of fracture was specified as a failure of: adhesion between ceramic and cement, adhesion between dentin and cement, cohesion in cement, cohesion in ceramic, cohesion in dentin or mixed failure.

4.5. Statistical Analysis

Mixed-design analysis of variance (split-plot ANOVA) with ceramics (i.e., IPS e.max ZirCAD, IPS Empress CAD, IPS e.max CAD) as a within-group repeated measure and dental cement (i.e., Panavia SA, Panavia V5, RalyX U200, Maxcem) as a between-group factor was used for this study. We employed a post-hoc analysis with Tukey's honestly significant difference (HSD) test for equal sample sizes to investigate differences between all compared groups. Additionally, to directly test our predictions, we performed a planned contrast analysis.

In a second step, we used a paired sample *t*-test to compare the bond strength of four types of cement (Panavia SA, Panavia V5, RelyX U200, Maxcem) tested in two experimental conditions (with or without thermocycling). The analyses were conducted separately for three ceramics (IPS e.max ZirCAD, IPS Empress CAD, IPS e.max CAD). To estimate the effect size of the observed differences, we calculated Cohen's d_z for correlated samples [24]. A one-way ANOVA was conducted to test if the changes of the bond strength resulting from thermocycling (dependent variable) are statistically different in four types of cement (independent variable). To test this effect in more detail and identify which types of cement differ in the bond strength change from each other, we ran post-hoc pairwise comparisons using Tukey's HSD test. The complementary analyses were also conducted separately for each of the three ceramics.

A probability value of $p < 0.05$ indicated statistically significant results. All statistical analyses were conducted in Statistica (data analysis software system), version 10 (StatSoft Inc., Tulsa, OK, USA), and using Psychometrica online tools (Psychometrica—https://www.psychometrica.de/effect_size.html (accessed on 16 December 2021), Alexandra Lenhard, Dettelbach, Germany) for calculating the effect sizes for the planned contras analysis [24].

5. Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Conventional resin cement (Panavia V5) showed significantly higher bonding strengths compared to self-adhesive, self-etching cements after accelerated thermal aging.
2. Regardless of the tested cement, the lowest bond strength among the tested ceramics was obtained for IPS e.max ZirCAD.
3. The appropriate selection of cement for ceramics is crucial, since differences in bond strengths for the studied combinations were statistically significant.
4. Comparing samples not subjected to thermocycling to the ones that were artificially aged, the greatest decreases in bond strength were observed for self-etching self-adhesive cements.

Author Contributions: Conceptualization: J.W. and M.W.; methodology: A.M. and W.G.; validation: J.W. and M.W.; investigation: A.M. and W.G.; data curation: A.M. and J.W.; formal analysis: D.P.D.; writing—original draft preparation: A.M., J.W. and D.P.D.; writing—review and editing: J.W. and M.W.; supervision: J.W. and M.W.; funding acquisition: M.W. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement: The study was approved by the Ethics Committee of the Wroclaw Medical University (approval No. KB-37/2018).

Informed Consent Statement: Not applicable.

Data Availability Statement: The data are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

Appendix A.

Appendix A.1. Validation of the Assumptions for Split-Plot ANOVA

The Mauchly's test showed no deviations from sphericity (W = 0.99, χ^2 = 0.46, p = 0.80). The Leven's test indicated equal variances for IPS e.max CAD (F(3, 44) = 0.41, p = 0.75) and variance heterogeneity for IPS e.max ZirCAD (F(3, 44) = 6.13, p = 0.001) and IPS e.max CAD (F(3, 44) = 4.89, p = 0.005). Since in balanced designs (as in the current study) the F statistic is reasonably robust to variance heterogeneity we considered observed heterogeneity acceptable.

Appendix A.2. Descriptive Statistics for the Examined Samples

Table A1. Descriptive statistics for the examined samples; N–number of samples, SD–standard deviation.

Cement	Ceramics	Shear Bond Strength [MPa]		N
		Mean	SD	
Panavia SA	IPS e.max ZirCAD	2.55	0.558	12
	IPS Empress CAD	2.98	0.667	12
	IPS e.max CAD	3.21	0.588	12
Panavia V5	IPS e.max ZirCAD	7.60	0.765	12
	IPS Empress CAD	16.84	0.844	12
	IPS e.max CAD	19.45	0.564	12
RelyX U200	IPS e.max ZirCAD	1.95	0.520	12
	IPS Empress CAD	3.36	0.260	12
	IPS e.max CAD	3.14	0.595	12
Maxcem	IPS e.max ZirCAD	2.32	0.182	12
	IPS Empress CAD	3.37	0.229	12
	IPS e.max CAD	2.06	0.445	12

Appendix A.3. Results of the Post-Hoc Tukey's HSD Test

Table A2. Tabularized representation of the results from the post-hoc Tukey's HSD test for differences between means of the shear bond strength in particular samples defined by ceramics and cement type. The samples are sorted according to the means in ascending order. The means that are not significantly different from each other form a homogenous group and are marked by three black dots ("●●●") in the same column. All means that do not share dots in the same column are significantly different from each other.

Cement	Ceramics	Shear Bond Strength [MPa]		Homogenous Groups						
		Mean	SE	1	2	3	4	5	6	7
RelyX U200	IPS e.max ZirCAD	1.95	0.158		●●●					
Maxcem	IPS e.max CAD	2.06	0.159		●●●					
Maxcem	IPS e.max ZirCAD	2.32	0.158		●●●		●●●			
Panavia SA	IPS e.max ZirCAD	2.55	0.158		●●●	●●●	●●●			
Panavia SA	IPS Empress CAD	2.98	0.163	●●●		●●●	●●●			
RelyX U200	IPS e.max CAD	3.14	0.159	●●●		●●●				
Panavia SA	IPS e.max CAD	3.21	0.159	●●●		●●●				
RelyX U200	IPS Empress CAD	3.36	0.163	●●●						
Maxcem	IPS Empress CAD	3.37	0.163	●●●						

Table A2. Cont.

Cement	Ceramics	Shear Bond Strength [MPa]		Homogenous Groups						
		Mean	SE	1	2	3	4	5	6	7
Panavia V5	IPS e.max ZirCAD	7.60	0.158					●●●		
Panavia V5	IPS Empress CAD	16.84	0.163						●●●	
Panavia V5	IPS e.max CAD	19.45	0.159							●●●

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molecules

Review

Effect of Different Surface Treatment Methods on Bond Strength of Dental Ceramics to Dental Hard Tissues: A Systematic Review

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Abstract: For long-term successful use of ceramic materials in dental procedures, it is necessary to ensure reliable bonding of restorations to dental substrates. This can be achieved by the application of a proper luting cement and through additional surface conditioning. The present systematic review summarizes the most up-to-date evidence on the use of different surface modification methods to enhance the bond strength of dental ceramics to the hard tissues of the teeth. The authors of the review searched the Web of Science, Scopus, and MEDLINE databases to identify relevant articles published between 1 January 2010 and 1 January 2020. A total of 4892 records were identified, and after screening, the full text of 159 articles was evaluated, which finally resulted in the inclusion of 19 studies. The available reports were found to be heterogeneous in terms of materials and methodology, and therefore, only within-studies comparison was performed instead of comparison between studies. A statistically significant difference in the bond strength between the samples treated with different methods of surface conditioning, or between conditioned and nonconditioned samples, was revealed by most of the studies. Predominantly, the studies showed that a combination of mechanical and chemical methods was the most effective way of enhancing bond strength. Artificial aging and luting cement were also identified as the factors significantly influencing bond strength.

Keywords: dental ceramic restoration; resin cement; luting agent; teeth; dentin; enamel; surface conditioning; surface modification; artificial aging; adhesion

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1. Introduction

Due to growing esthetic demand and the development of computer-aided design/computer-aided manufacturing (CAD/CAM) systems in recent years, ceramics have become a very popular material for the manufacturing of fixed dental prosthetics, ranging from veneers, inlays, and onlays to full-crown restorations and bridges. This popularity is mainly attributed to their properties, such as biocompatibility, excellent esthetic effect, and chemical and volumetric stability [1–4]. However, the clinical success of a ceramic restoration also depends on good marginal adaptation as well as strong and reliable adhesion of the ceramic surface to the tooth tissues. Adhesive bond strength, calculated by dividing the failing load by the bond area, could be determined using various tests (shear, tensile, microtensile or pull-out test). Reliable adhesion could be achieved by using a proper luting cement providing attachment of dental restoration to the prepared teeth (including conventional cements, such as zinc phosphate or glass-ionomer, and contemporary cements, such as resin and resin-modified glass-ionomer) and through additional surface conditioning. This will not only increase the retention of the restoration but also minimize microleakage, improve marginal adaptation, and increase the fracture resistance, thereby ensuring durability and long life of the prosthetic reconstruction [5–7].

The successful bonding of ceramic restorations is strongly associated with proper chemical and mechanical interactions of the ceramic surface with luting cement and the hard tissues of the teeth [5,8–10]. For this purpose, various methods of surface treatment are applied to increase the adhesion of the ceramic material to the luting cement and the dental substrates [11–13]. Micromechanical retention, which results in increased surface roughness, could be facilitated by methods such as acid etching, airborne particle abrasion (APA), tribochemical silica coating, and laser irradiation [12]. On the other hand, chemical conditioning can be performed using bifunctional silane agents that enhance the wettability of the ceramic surface and improve the penetration of the resin cement into microscopic porosities created in the conditioned surface [13,14]. A frequently studied alternative is the universal adhesive system which is based on phosphate monomers (10-methacryloyloxydecyl dihydrogen phosphate, MDP) [10–13]. The 10-MDP, incorporated into dental adhesive systems as a functional monomer, promotes chemical interaction with dental substrates, enhancing adhesion forces. Through the formation of MDP-calcium salts it promotes also the protection of collagen fibers [15].

The proper choice of surface conditioning method is of huge importance for the clinical utility of ceramic restorations. The selection of this method is dependent on the chemical and physical properties of the material. Silica-based ceramics, such as leucite, lithium disilicate, or feldspathic porcelain, are easier to work with because their glassy phase can be more easily chemically treated than high-strength zircon dioxide [3–5]. On the other hand, zirconia has favorable mechanical properties such as high flexural strength, relatively low elastic modulus, and high fracture toughness [16]. This material is also characterized by good chemical and dimensional stability [5,6]. However, the adhesion of zirconium to the dental substrates is unstable and poor which attracts the attention of many research groups attempting to achieve optimum bond strength without altering the strength of the ceramic itself [5–9]. Due to their crystalline structure, zirconium materials are acid-resistant. Therefore, the first modification of their surface mainly involves a mechanical process and the creation of a layer containing a glassy phase that can be modified chemically in a much simpler way. For this purpose, APA, silica coating, or porcelain glazing was performed [3,5,9,10].

Although numerous studies have been carried out on ceramic surface conditioning, there is still no consensus on the optimal protocol that would enable the best bonding between a ceramic restoration and the dental tissue to be obtained. The aim of this systematic review was to summarize the most up-to-date available evidence on the use of different surface conditioning methods to enhance the bond strength of dental ceramics to the hard tissues of the teeth. The authors of the review focused on the critical revision of the technical details concerning the materials and techniques applied in the most recent experimental research, which could allow the identification of the strengths and weaknesses of the available reports. Additionally, the review is intended to determine the significance of the other factors influencing the bond strength values, such as artificial aging and luting cement, in order to identify the most effective surface conditioning methods that would contribute to increasing the clinical utility of modern dental materials.

2. Results

2.1. Study Selection

Three authors (A.M., S.O. and W.F.) were involved in the literature identification and record screening procedure. The selection process is detailed in the PRISMA flow diagram in Figure 1. A total of 4892 records were found in the databases: 2035 in Web of Science, 1724 in Scopus, and 1133 in MEDLINE. In addition, two records were added after screening the reference lists of the qualified studies. After removing the duplicates from the studies identified from the different databases, a total of 4070 records remained. Then, three authors screened the titles and abstracts of these remaining records based on the inclusion and exclusion criteria, after which 3911 articles were excluded. Afterward, two authors (A.M. and W.F.) independently assessed the full text of 159 selected articles for the final evaluation of eligibility. Their assessment was critically revised by another author (J.W.). Finally, 19 articles were included in this systematic review.

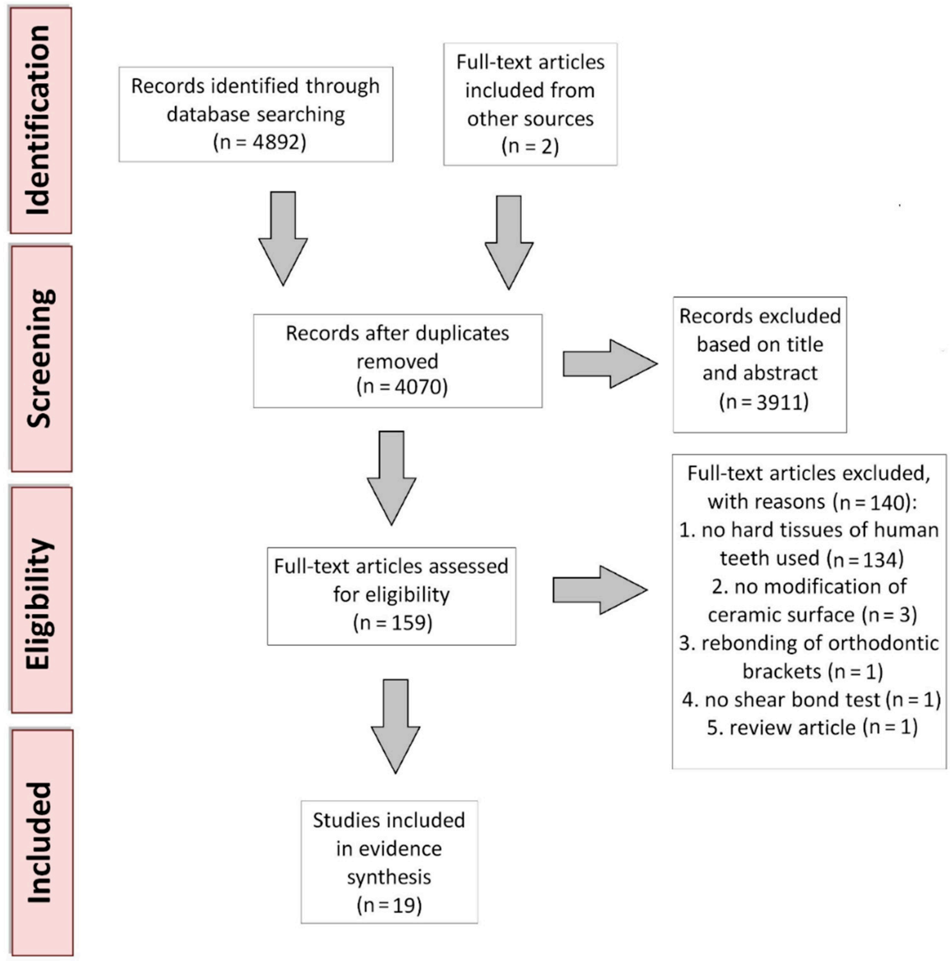

Figure 1. PRISMA flow diagram of the systematic review protocol.

2.2. *Material Characterization and Specimen Preparation*

All the qualified papers investigated the bond strength of dental ceramics to dental hard tissues. Fifteen of these studies described ceramics luted to human dentin [7,16–29], two described ceramics luted to human enamel [30,31] and one described ceramics luted to the dentin of bovine teeth [32]. Saker et al. performed a comparative study on two human dental tissues: dentin and enamel [33].

The types of ceramic materials and dental cements used in the included studies for specimen preparation are summarized in Table 1. Most of the selected studies focused on yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) [16,18–22,24,26,31–33]. Furthermore, lithium disilicate glass-ceramic was investigated by Madina et al. [17], feldspathic ceramic by Jetti et al. [25], and monolithic zirconia by Reddy et al. [20], Feng et al. [23], Butler et al. [29], and Zandparsa et al. [30], while Park et al. evaluated resin nanoceramics [7]. Different types of dental ceramics were compared by Kara et al. [27] (feldspathic ceramic, leucite-reinforced hot-pressed ceramic, hot-pressed lithium disilicate ceramic, and zirconia) and Gamal et al. [28] (lithium disilicate and zirconia).

Table 1. Characteristics of the materials used in the studies included in the systematic review, presented in chronological order.

Author and Year	Ceramics (Commercial Name, Manufacturer)	Cement (Commercial Name, Manufacturer)
Madina 2010 [17]	**IPS e.max PRESS** (Ivoclar Vivadent)	**Panavia F 2.0** (Kuraray)
Qeblawi 2010 [16]	**IPS e.max ZirCAD** (Ivoclar Vivadent)	**Multilink Automix** (Ivoclar Vivadent)
Shahin 2010 [18]	**In-Ceram YZ for inLAB** (Vita)	(1) **Hoffmann quick setting** (Hoffmann Dental) (2) **Ketac Cem Maxi Cap** (3M ESPE) (3) **Panavia 21 TC** (Kuraray)
Chai 2011 [19]	(1) **In-Ceram Zirconia** (Vita) (2) **YZ Zirconia** (Vita)	**Panavia F 2.0** (Kuraray)
Reddy 2012 [20]	**Incoris ZI** (Sirona)	**Multilink Speed** (Ivoclar Vivadent)
De Castro 2012 [21]	**In-Ceram YZ** (Vita)	(1) **RelyX ARC** (3M ESPE) (2) **RelyX U100** (3M ESPE) (3) **Panavia F** (Kuraray)
Saker 2013 [33]	**In-Ceram Zirconia** (Vita)	**Panavia F 2.0** (Kuraray)
Zandparsa 2013 [30]	**Zirconia** (3M ESPE)	**Panavia F 2.0** (Kuraray)
Bottino 2014 [22]	**In Ceram YZ 2000** (Vita)	(1) **Panavia F** (Kuraray) (2) **Clearfil SA Cement** (Kuraray)
Feng 2014 [23]	**Sintered zirconia blocks** (3M ESPE)	(1) **Panavia F** (Kuraray) (2) **RelyX Unicem** (3M ESPE)
Menani 2014 [32]	**Lava Frame Y-TZP** (3M ESPE)	**Panavia F** (Kuraray)
Alves 2015 [24]	**InCeram YZ** (Vita)	(1) **RelyX ARC** (3M ESPE) (2) **RelyX U200** (3M ESPE)
Jetti 2015 [25]	**CEREC** (Sirona)	**Variolink II** (Ivoclar Vivadent)
Lv 2015 [31]	**Yttria-stabilized zirconia powder** (Tosho)	(1) **Superbond C and B** (Sun Medical) (2) **Panavia F 2.0** (Kuraray)
Unal 2015 [26]	**ZirkonZahn** (Steger)	(1) **Panavia F 2.0** (Kuraray) (2) **Variolink N** (Ivoclar Vivadent)
Park 2016 [7]	**Lava Ultimate** (3M ESPE)	**RelyX** (3M ESPE)
Kara 2017 [27]	(1) **Finesse** (Ceramco) (2) **IPS Empress Esthetics** (Ivoclar Vivadent) (3) **IPS Empress e.Max** (Ivoclar Vivadent) (4) **Zirkonzahn Prettau** (Zirkonzahn GmBh)	**Clearfil Esthetic Cement** (Kuraray)
Gamal 2018 [28]	(1) **IPS e.max CAD** (Ivoclar Vivadent) (2) **IPS e.max ZirCAD** (Ivoclar Vivadent)	**RelyX Ultimate** (3M ESPE)
Butler 2018 [29]	**NexxZr** (Sagemax Bioceramic)	**Duo-link** (Bisco)

The included papers also differed in terms of the dental cement used to lute the ceramic to the tooth tissue. In many studies, Panavia F2.0, a self-etching, MDP-containing dual-polymerizing resin cement, was either used separately [17,19,30,33] or compared with adhesive self-curing resin cement (Superbond C and B [31]) or dual-polymerizing resin cement (Variolink N [26]). Other self-adhesive materials evaluated were Multilink Speed, a self-curing composite resin cement, which can be light-cured if desired [20], and Clearfil Esthetic Cement [27]. Some studies also investigated the dual-polymerizing adhesive cements, including Multilink Automix [16], Variolink II [25], RelyX Ultimate [28], and Duo-link [29]. Shahin et al. compared the various groups of cements, namely zinc phosphate cement (Hoffmann quick setting), glass-ionomer cement (Ketac Cem Maxi Cap), and adhesive resin cement (Panavia 21) [18]. Alves et al. compared an adhesive resin cement (RelyX ARC) with a self-adhesive resin cement (RelyX U200) [24], while De Castro et al. compared an adhesive resin cement (RelyX ARC) with a self-adhesive (RelyX U100) and a dual-polymerizing resin cement (Panavia F) [21]. Menani et al. [32] also separately studied Panavia F as well as comparing this cement with self-adhesive dual-polymerizing resin cements (Clearfil SA Cement [22], RelyX Unicem [23]). One study focused on a cement material described as "RelyX," but it is not very informative [7].

2.3. Methodology of the Selected Studies: Surface Treatment, Artificial Aging, and Bond Strength Evaluation

The methods used for surface conditioning and artificial aging in the included studies are presented in Table 2.

Table 2. Characteristics of the surface treatment and artificial aging methods, and primary and secondary outcomes of the studies included in the systematic review, presented in chronological order. HF acid = hydrofluoric acid; APA = airborne particle abrasion; SBS = shear bond strength.

Author and Year	Surface Treatment	Artificial Aging	Primary Outcome: Impact of Different Surface Treatment Methods on the Bond Strength	Secondary Outcome: Impact of the Other Studied Factors on the Bond Strength
Madina 2010 [17]	(1) HF acid 5% + silane (2) APA + tribochemical silica coating + silane	None	No statistically significant difference was found between the surface conditioning methods.	-
Qeblawi 2010 [16]	16 groups: 4 different mechanical treatments: (1) No mechanical treatment (2) APA (3) Tribochemical silica coating (4) Wet hand grinding Combined with 4 different chemical treatments: (1) No chemical treatment (2) Acid etching + silane (3) Silane (4) Zirconia primer	(1) None (2) 90 days at 100% humidity and 37 °C; 2000 thermal cycles (5–55 °C, 10 s dwell time) every 30 days for a total of 6000 cycles	The highest SBS values were achieved for silica coating + silane.	(1) Statistically significant difference was observed between the groups (immediate/aged). (2) Artificial aging resulted in significantly lower SBS for silica coating + silane and for no mechanical treatment + zirconia primer.
Shahin 2010 [18]	(1) No treatment (2) APA	(1) 3 days in distilled water at 37 °C (2) 150 days in distilled water at 37 °C; 37,500 thermal cycles (5–55 °C, 30 s dwell time); after thermocycling, masticatory simulation (300,000 cycles, load of 50 N)	APA significantly increased crown retention.	(1) Artificial aging significantly reduced retention. (2) Adhesive resin cement (Panavia 21) allowed significantly higher retention than the conventional cements.
Chai 2011 [19]	(1) No treatment (2) Chairside tribochemical silica coating + silane (CoJet, 3M ESPE) + resin-bonding agent (Visio Bond, 3M ESPE) (3) Laboratory tribochemical silica coating + silane (Rocatec, 3M ESPE)	None	In-Ceram Zirconia treated with CoJet had a significantly higher SBS than those untreated or treated with Rocatec.	The bond strength between the two ceramic types was not significantly different.
Reddy 2012 [20]	(1) No treatment (2) APA (3) HF acid 4.5% (4) HF acid 4.5% + silane (5) Zirconia primer	None	(1) The highest values were obtained for zirconia primer, the second highest for APA, and the third for HF acid with silane. (2) There were no significant differences between HF acid and nontreated control.	-
De Castro 2012 [21]	(1) APA (2) Tribochemical silica coating	(1) No additional storage (2) 60 days in distilled water at 37 °C (3) 10,000 thermal cycles (5–55 °C, 30 s dwell time)	Statistically significant difference was found between the groups treated with different surface conditioning methods.	(1) Resin cement and artificial aging significantly affected the mean bond strength values. (2) The highest bond strength was achieved for Panavia F with APA after thermal cycling.
Saker 2013 [33]	(1) No Treatment (2) APA (3) Tribochemical silica coating + silane (4) Tribochemical silica coating + metal primer-containing MDP (5) Glaze ceramic + HF acid 9.6% + silane	5000 thermal cycles (5–55 °C, 20 s dwell time)	(1) All the surface treatment protocols significantly increased the tensile bond strength compared to control. (2) The lowest increase was achieved for APA, and the highest for glaze + HF acid (for enamel) or tribochemical silica coating + metal primer (for dentin).	Substrate type (enamel vs. dentin) had a significant influence on the bond strength.
Zandparsa 2013 [30]	(1) APA (2) APA + Z-PRIME Plus (3) APA + alloy primer (4) Piranha solution 7:1 (5) Piranha solution 7:1+ Z-PRIME (6) Piranha solution 7:1 + alloy primer (7) Tribochemical silica coating + silane	500 thermal cycles (5–55 °C, 15 s dwell time)	APA + Z-PRIME Plus showed significant improvement in SBS compared to all other groups.	-

Table 2. *Cont.*

Author and Year	Surface Treatment	Artificial Aging	Primary Outcome: Impact of Different Surface Treatment Methods on the Bond Strength	Secondary Outcome: Impact of the Other Studied Factors on the Bond Strength
Bottino 2014 [22]	(1) Low-fusing porcelain glaze + HF acid 10% + silane (2) Tribochemical silica coating	5000 thermal cycles (5–55 °C, 30 s dwell time)	No statistically significant difference was found between the groups treated with different surface conditioning methods.	Resin cement (Panavia > Clearfil) and storage conditions (nonaging > aging) significantly influenced the SBS.
Feng 2014 [23]	(1) No treatment (2) APA+ silane (3) APA+ tribochemical silica coating + silane	None	The bond strength of APA + silica coating + silane group was the highest, while the bond strength in the control group was the lowest.	Specimens bonded with Panavia F exhibited significantly higher bond strength than those with RelyX Unicem regardless of the surface treatments.
Menani 2014 [32]	(1) No treatment (2) Alloy primer (3) HF acid 40% (4) HF acid 40% + alloy primer	None	(1) The extrusion shear strength of the group etched with 40% HF acid was significantly higher than that of other groups. (2) Differences for the other groups were not statistically significant.	-
Alves 2015 [24]	(1) No treatment (2) Chairside tribochemical silica coating + silane (CoJet, 3M ESPE) (3) Laboratory tribochemical silica coating + silane (Rocatec + 3M ESPE) (4) Universal primer	30 days in distilled water at 37°C	(1) Universal primer application provided the highest SBS compared to other methods. (2) Nontreated control group presented the lowest SBS.	Cement type did not significantly affect the SBS.
Jetti 2015 [25]	(1) HF acid <5% + Prime and Bond NT (2) HF acid <5% + silane + Prime and Bond NT (3) HF acid <5% + Xeno III (4) HF acid <5% + silane +Xeno III	None	(1) The application of silane significantly increased the SBS in both groups bonded with Prime and Bond NT and with Xeno III. (2) There were no significant differences in SBS between the groups bonded with Prime and Bond NT and with Xeno III. (3) The highest SBS was achieved using <5% HF acid + silane and Prime and Bond NT.	-
Lv 2015 [31]	(1) No treatment (2) APA (3) Hot-etching treatment (800 mL of methanol, 200 mL of 37% HCl and 2 g of $FeCl_3$) for 1 h at 100 °C	5000 thermal cycles (5–55 °C, 30 s dwell time)	The hot-etching group had significantly higher SBS than the control and APA groups.	SBS was significantly lower after thermal cycling in all groups except for the hot-etching group that was cemented with Panavia F2.0.
Unal 2015 [26]	(1) No treatment (2) APA (3) Tribochemical silica coating (4) YbPL laser	5000 thermal cycles (5–55 °C, 15 s dwell time)	Laser-irradiated groups had significantly higher SBS than the other groups.	Cement type significantly affected the SBS values (Panavia F 2.0 > Variolink N).
Park 2016 [7]	(1) APA (2) APA + Singlebond Universal Adhesive (3) HF acid 4% + Singlebond Universal Adhesive (4) HF acid 4% + silane + Singlebond Universal Adhesive (5) Tribochemical silica coating (6) Tribochemical silica coating + Singlebond Universal Adhesive	None	(1) APA + universal adhesive resulted in the highest bond strength followed by tribochemical silica coating + universal adhesive. (2) The lowest bond strength was achieved for 4% HF acid etching + universal adhesive. (3) Universal adhesive increased the bond strength, while silane had no significant effect.	-
Kara 2017 [27]	(1) No treatment (2) APA (3) HF acid 9% (4) Hot acidic solution containing HCl and $FeCl_3$ (100 °C) applied for 30 min (5) Nd:YAG laser (6) Nd:YAG laser + APA (7) Nd:YAG laser + HF acid 9% (8) Nd:YAG laser + hot acidic solution	5000 thermal cycles (5–55 °C, 30 s dwell time)	(1) No significant differences in bond strength were seen in Finesse ceramic groups treated with different methods. (2) HF acid etching increased the bond strength of IPS Empress Esthetics. (3) APA and HF acid etching increased the bond strength of IPS Empress e-Max. (4) APA and Nd:YAG + APA increased the bond strength of Zirkonzahn Prettau.	-

Table 2. *Cont.*

Author and Year	Surface Treatment	Artificial Aging	Primary Outcome: Impact of Different Surface Treatment Methods on the Bond Strength	Secondary Outcome: Impact of the Other Studied Factors on the Bond Strength
Gamal 2018 [28]	(1) CO_2 laser + HF acid 9% + silane (2) HF acid 9% + silane (3) CO_2 laser + APA + silane (4) APA + silane	None	(1) Laser irradiation increased the SBS between zirconia and dentin compared with nonirradiated ceramic surfaces. (2) Laser irradiation combined with HF acid and silane did not seem to be an alternative method for improving the dentin-to-ceramic surface (lithium disilicate) bonding.	
Butler 2018 [29]	(1) No treatment (2) APA (3) Primer (4) APA + primer (5) APA + All-Bond Universal (6) APA + ScotchBond Universal Adhesive	None	(1) SBS was significantly influenced by the use of APA, primer, or adhesive. (2) The use of Z-Prime Plus and All-Bond Universal resulted in significantly higher bond strength.	-

The included studies investigated the techniques of both micromechanical and chemical bonding of ceramics to dental hard tissues. Among the methods applied to achieve micromechanical bonding, there were different kinds of mechanical treatments such as APA [7,16–18,20,21,23,26–31,33], tribochemical silica coating [7,16,17,19,21–24,26,30,33], laser irradiation [26–28], and wet hand grinding [16]. The second approach utilized for micromechanical bonding was a chemical-based one which involved the use of various acid solutions to etch the conditioned surface [7,16,17,20,22,25,27,28,30–33]. On the other hand, different methods applied to achieve chemical bonding were also evaluated. These included the use of porcelain glaze [22,33] and coupling agents such as primers and silanes [7,16,17,19,20,22–25,28–30,32,33]. A nontreated control was used in 12 of the 19 included studies [16,18–20,23,24,26,27,29,31–33]. In the rest of the studies, different methods of surface conditioning were compared with each other [7,17,21,22,25,28,30].

Additionally, in 10 of the 19 selected studies, artificial aging was performed [16,18,21,22,24,26,27,30,31,33]. The parameters of aging differ significantly. In the studies, the specimens were subjected to prolonged storage in distilled water at 37 °C for different periods of time [18,21,24] or subjected to different numbers of thermal cycles between 5 °C and 55 °C with different dwell times [21,22,26,27,30,31,33]. Both prolonged water storage and thermal cycles were performed in the study conducted by Qeblawi et al., [16]. In a study carried out by Shahin et al., water storage and thermocycling were followed by masticatory simulation [18].

To investigate the bond strength between dental ceramics and dental hard tissues, most of the researchers used shear bond strength test with a shear crosshead speed of 1.0 mm/min [16,20,24–26,28,31] or 0.5 mm/min [19,22,29,30]. The other methods used for evaluating bond strength were the pull-out test of retentive strength [17,18], extrusion shear test [32], tensile test [33], and microtensile strength test [7,21,23,27].

2.4. Outcomes

The primary and secondary outcomes of the selected studies are described in Table 2.

As a primary outcome, a statistically significant difference in bond strength between the samples treated with different surface conditioning methods, or between the conditioned and nonconditioned samples, was revealed in most of the studies [7,16,18–21,23–33]. Only two studies showed no statistically significant difference between the compared experimental groups [17,22]. However, in these studies, there were no nontreated control groups, but different surface conditioning methods were compared to each other (hydrofluoric (HF) acid + silane vs. APA + tribochemical silica coating + silane [17] or low-fusing porcelain glaze + HF acid + silane vs. tribochemical silica coating [22]). Kara et al. found no significant differences in bond strength in one out of four evaluated ceramic groups that were treated with different methods [27]. All the studies conducted using a nontreated control group concluded that the bond strength of the nontreated specimens was significantly lower than that of the specimens subjected to surface modification [16,18–20,23,24,26,27,29,31–33]. Many studies suggested that a combination of mechanical and chemical methods, such as silica coating + silane [16], silica coating + primer or HF acid + glaze [33], APA + primer [30], APA + silica coating + silane [23], and APA + universal adhesive [7], was the most effective way of enhancing bond strength.

The impact of the other studied factors on bond strength between ceramics and teeth was investigated as a secondary outcome in 10 of the 19 selected papers. Artificial aging [16,18,21,22,31] and luting cement [18,21,23,26] were identified as the factors significantly influencing the obtained values of bond strength. Furthermore, Saker et al. demonstrated that substrate type (enamel vs. dentin) also had a significant influence on bond strength [33].

2.5. Evidence Synthesis

The quality of the evidence presented in the studies, with overall GRADE (Grading of Recommendations Assessment, Development and Evaluation) scores for primary and secondary outcomes, is shown in Table 3. The number of samples in each experimental group used in the included studies ranged from 3 [21] to 30 [7]. Most of the included studies (17 out of 19) revealed the significant influence of the surface conditioning methods on the bond strength of dental ceramics to dental hard tissues. A significant effect of the other studied factors (e.g., luting cement and artificial aging) was demonstrated in 8 out of 10 studies.

Table 3. Summary findings for the primary and secondary outcomes.

Outcome	Outcome Significance	Author and Year	No. of Specimens per Group	Quality of the Evidence (GRADE)
Primary outcome	Significant correlation	Qeblawi 2010 [16]	12	++++ high
		Shahin 2010 [18]	8	+++− moderate due to indirectness
		Chai 2011 [19]	12	++−− low due to imprecision and risk of bias
		Reddy 2012 [20]	4	+++− moderate due to imprecision
		De Castro 2012 [21]	3	++−− low due to imprecision and risk of bias
		Saker 2013 [33]	10	++++ high
		Zandparsa 2013 [30]	10	+++− moderate due to risk of bias
		Feng 2014 [23]	10	+++− moderate due to imprecision
		Menani 2014 [32]	7	++− low due to imprecision and indirectness
		Alves 2015 [24]	10	++++ high
		Jetti 2015 [25]	10	++−− low due to imprecision and risk of bias
		Lv 2015 [31]	10	++++ high
		Unal 2015 [26]	15	+++− moderate due to imprecision
		Park 2016 [7]	30	++−− low due to imprecision and risk of bias
		Kara 2017 [27]	12	++++ high
		Gamal 2018 [28]	6	++−− low due to imprecision and risk of bias
		Butler 2018 [29]	10	++++ high
	No significant correlation	Madina 2010 [17]	8	++−− low due to indirectness and risk of bias
		Bottino 2014 [22]	10	+++− moderate due to risk of bias
Secondary outcome	Significant correlation	Qeblawi 2010 [16]	12	+++− moderate due to risk of bias
		Shahin 2010 [18]	8	+++− moderate due to indirectness and risk of bias
		De Castro 2012 [21]	3	++−− low due to imprecision
		Saker 2013 [33]	10	+++− moderate due to indirectness
		Bottino 2014 [22]	10	++++ high
		Feng 2014 [23]	10	+++− moderate due to imprecision
		Lv 2015 [31]	10	++++ high
		Unal 2015 [26]	15	+++− moderate due to imprecision
	No significant correlation	Chai 2011 [19]	12	++−− low due to imprecision and risk of bias
		Alves 2015 [24]	10	++++ high

The quality of the evidence presented in most of the included studies was scored as +++− (moderate), ++++ (high), or ++− (low). The common causes of score reduction included imprecision and risk of bias.

3. Discussion

Due to their huge clinical importance, the methods that promote reliable bonding of ceramic restorations to the dental hard tissues are of interest to many research groups. Several interesting reviews of the research concerning surface conditioning methods applied to increase the bond strength between ceramics and teeth have been published in recent years. The conducted analyses drew the conclusion that a combination of mechanical and chemical treatments is essential for good adhesion. However, they revealed that currently there is a lack of evidence to support a universal adhesion protocol [34–36].

This systematic review focused primarily on the influence of surface modification methods on the bond strength between ceramics and dental substrates. The vast majority of the selected articles performed the modification of zirconia to achieve long-term, durable bonding of this material. In one study, a lithium disilicate glass-ceramic [17] and feldspathic ceramic [25] were investigated. One research paper was based on resin nanoceramic [7], which is a relatively new material, used mainly for minor restorations. Different types of dental ceramics were compared in the studies by Kara et al. (feldspathic ceramic, leucite-reinforced hot-pressed ceramic, hot-pressed lithium disilicate ceramic, and zirconia) [27] and Gamal et al. (lithium disilicate and zirconia) [28]. These studies demonstrated that different types of ceramics required different methods of surface conditioning for strong bonding to dental substrates [27,28].

The present systematic review revealed that different mechanical treatments (APA, tribochemical silica coating, laser irradiation, and wet hand grinding) and chemical treatments (acid etching) were investigated to achieve micromechanical bonding. Other methods of chemical bonding such as the use of porcelain glaze and coupling agents (primers and silanes) were also evaluated. A statistically significant difference in bond strength between the samples treated with different surface conditioning methods, or between conditioned and nonconditioned samples, was revealed in most of the studies. Predominantly, the studies showed that a combination of mechanical and chemical methods, such as silica coating + silane [16], silica coating + primer or HF acid + glaze [33], APA + primer [30], APA + silica coating + silane [23], and APA + universal adhesive [7], was the most effective way of enhancing bond strength. Three studies investigated the effectiveness of laser irradiation as an alternative technique for treating ceramic surfaces prior to bonding resin cements. They revealed increased shear bond strength between zirconia and dentin after irradiation with YbPL laser [26], Nd:YAG laser [27], and CO_2 laser [28] compared with nonirradiated ceramic surfaces.

Apart from evaluating the effectiveness of surface conditioning methods in the present review, attention was also paid to the significance of the effects of artificial aging performance and the selection of luting agent on bond strength. Artificial aging was performed in 10 out of 19 selected studies [16,18,21,22,24,26,27,30,31,33]. The parameters of aging differ significantly—in the selected studies, the specimens were stored in distilled water at 37 °C for different periods of time or were subjected to different numbers of thermal cycles between 5 °C and 55 °C. One study combined prolonged water storage and thermocycling [16], while another study additionally performed masticatory simulation after water storage and thermocycling [18]. Only 5 out of 10 studies that used artificial aging compared the results for aged and nonaged samples. All of them reported a statistically significant decrease in the bond strength of specimens after artificial aging [16,18,21,22,31].

In total, 6 out of 19 studies compared the bond strength values achieved using different luting agents. Shahin et al. compared various groups of cements—zinc phosphate cement (Hoffmann quick setting), glass-ionomer cement (Ketac Cem Maxi Cap), and adhesive resin cement (Panavia 21), and demonstrated that the adhesive resin cement (Panavia 21) provided significantly higher retention than the conventional cements [18]. Most of the other studies also revealed a statistically significant difference in bond strength between the groups luted with different cements [21–23,26]. Application of the adhesive resin cement Panavia F resulted in a significantly higher bond strength compared to several self-adhesive cements (RelyX U100 [21], Clearfil SA [22], RelyX Unicem [23]). Unal et al. showed a higher bond strength after cementation with adhesive MDP-containing Panavia F 2.0 compared to Bis-GMA-containing Variolink N cement [26]. Only one study did not show any statistically significant difference between the compared adhesive resin cement (RelyX ARC) and self-adhesive resin cement (RelyX U200), and thus did not confirm the influence of the type of cement on bond strength [24].

An additional huge advantage of this systematic review is the selection of papers describing research conducted on samples luted to dental hard tissues (dentin or enamel) of humans. This criterion for the method of specimen preparation significantly reduced the number of studies that could be qualified for the review, but it enabled a more precise analysis in terms of the clinical utility of the results obtained. In one study, bovine teeth were used as a substitute for human tissue [32], but the validity of such an approach was confirmed in previous reports [37–40].

The main limitation of this review is the lack of a meta-analysis, which could not be performed due to the heterogeneity of the available reports on dental ceramic surface modifications, both in terms of materials and protocols. Therefore, the results were compared only within studies but not between studies. The identified risk of bias can be attributed mainly to the lack of information regarding the number of operators performing the experiments and a low sample size which was observed in several studies. Furthermore, some of the reports did not precisely define the full names of the materials used.

One of the sources of heterogeneity was the application of different bond strength tests (shear, pull-out, extrusion, tensile, and microtensile strength tests). Most of the included studies performed a shear bond strength test, which is easy to use, but is characterized by less uniform stress distribution compared to a tensile bond strength test [7,41]. In addition, some previous analyses revealed that microbond tests are more reliable than macrobond tests [42].

Another interesting issue that should be investigated in the future is the limited usefulness of bond strength testing, including shear loading. As they do not fully mimic the real clinical situation with a complex pattern of stress distribution during failure, additional methods should be applied to better predict the clinical behavior of ceramic restorations. Thus, the performance of fatigue tests under cyclic loading, as a way of masticatory simulation, should be considered [43]. The application of degradation protocols (e.g., water or saliva storage and thermal cycling) should also be included to simulate the chemical and thermal conditions that restorations may be subjected to. Due to their low costs, water storage and thermocycling in water are the most common methods of artificial aging. However, many different models could be proposed to evaluate the effect of the oral environment (different pH levels, thermal fluctuations, enzymatic activity, masticatory forces, etc.,) on the degradation of dental materials. Consideration of these factors is strongly recommended for future laboratory research in order to simulate the clinical situation more accurately. Finally, apart from the recommendation for using more comparable methodologies in laboratory studies evaluating the different aspects of bond strength, further clinical trials are needed to provide relevant evidence of successful bonding [34,35].

4. Materials and Methods

This systematic review was accomplished in accordance with the PRISMA (Preferred Reporting Items for Systematic Reviews and Meta-Analyses) guidelines used to collect and report data [44,45]. It was conducted in an attempt to answer the following questions: (1) Does surface conditioning significantly influence the bond strength of dental ceramics to dental tissue? (2) Which surface conditioning method can most effectively improve the bond strength of dental ceramics to dental tissue? (3) What are the other factors (e.g., artificial aging, luting cement) that significantly influence the bond strength of dental ceramics to dental tissue?

4.1. Search Strategy

4.1.1. Data Sources and Searches

The authors searched the Web of Science, Scopus, and MEDLINE databases to identify the relevant articles published in English between 1 January 2010 and 1 January 2020. The literature search was performed combining each of the following keywords: (1) dental ceramic, (2) dental resin cement, (3) dental luting cement, and (4) teeth; with each of the following keywords: (A) surface modification, (B) surface treatment, and (C) surface conditioning; and with each of the following additional keywords: (a) bond strength, (b) durability, and (c) adhesion. The database search was supplemented with a hand search of the bibliographic references of the retrieved articles aimed at the identification of potentially relevant papers [44,45].

Three authors (A.M., S.O. and W.F.) were involved in the literature identification and record-screening procedure. After removing the duplicates from the records identified in different databases, the three authors screened the titles and abstracts of the remaining records based on the inclusion and exclusion criteria. For a final evaluation of eligibility, two authors (A.M. and W.F.) performed an independent assessment of the full text of the selected articles, which was critically revised by another author (J.W.). None of the review authors was blind to the title of the articles, author names, and affiliations.

4.1.2. Eligibility Criteria for Initial Study Selection

During the database search, the authors aimed to select studies that quantitatively investigated the effects of different surface treatment methods on the bond strength of dental ceramics luted with resin cements to the hard tissues of the tooth.

The authors added filters to identify only English language and full-text articles published between 1 January 2010 and 1 January 2020. Inclusion and exclusion criteria were defined according to the PICOS (Population, Intervention, Comparison, Outcomes and Study Design) approach and are listed in Table 4.

Table 4. Inclusion and exclusion criteria.

PICOS	Inclusion Criteria	Exclusion Criteria
Population	Ceramic samples luted to hard tissues of tooth (enamel or dentin)	Samples that are not made of ceramic Ceramic–cement combination without tooth tissue Ceramic luted to another material (composite, metal), without tooth tissue
Intervention	Any method of surface modification	No surface modification applied
Comparator	Nontreated control or any other method of surface modification	None
Outcome	Shear or tensile bond strength or retentive strength of the ceramics luted to the tooth tissue	Any other methods used for the evaluation of the quality of the bond between the ceramic and the tooth
Study	Only English language and full-text articles published between 1 January 2010 and 1 January 2020	Review papers Articles not in English Articles published before 1 January 2010

4.2. Data Extraction

After the inclusion of final studies, two reviewers (A.M. and W.F.) carried out data extraction independently. Then, the third author (J.W.) checked the validity of the extracted data. The data extraction process included retrieval of information regarding the type of specimen, type and name of ceramics, type and name of the resin cement, number of samples, methods of surface treatment, methods of artificial aging, methods of bond strength evaluation, and primary and secondary outcomes.

The primary outcome of interest was the impact of surface treatment methods on the bond strength of dental ceramics to the tooth structures, while the secondary outcome was the impact of the other studied factors on the mentioned parameter.

4.3. Data Synthesis and Analysis and Quality Assessment

The studies included in this systematic review were very heterogeneous; therefore, it was not possible to perform a meta-analysis, and instead, a narrative and qualitative summary was prepared.

The GRADE approach was used to assess the quality of evidence for the primary and secondary outcomes. For each outcome, the quality of evidence was assigned to one of the following categories: very low, low, moderate, or high [46].

5. Conclusions

Different methods of surface treatment can be applied to achieve strong, durable bonding of different types of ceramics to dental substrates. The present review of laboratory studies revealed a statistically significant difference in bond strength between the samples treated with different surface conditioning methods, or between conditioned and nonconditioned samples. Based on the results analyzed, a combination of mechanical and chemical methods is proposed as the most effective way of enhancing bond strength.

In addition, this review of the available literature highlights the need for standardizing the methodology of surface modification for future investigations. Due to the use of different materials, protocols, and tests by researchers, data comparison is quite difficult. Moreover, standardized protocols should attempt to reproduce clinical conditions by applying different methods of testing, including fatigue tests, as well as through artificial aging of samples. Such an approach will allow better prediction of the real clinical behaviors of the evaluated ceramic materials.

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