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

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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.

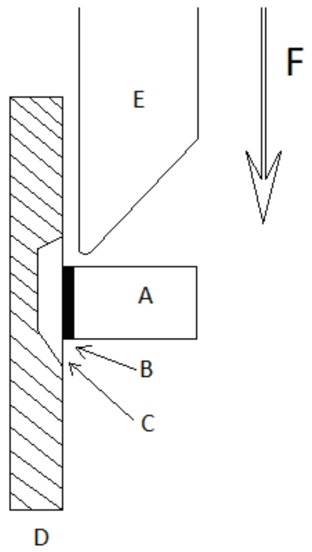

Fig. 1. Schematic illustration of a sample subjected to shear force: *A* – ceramic cylinder, *B* – resin cement, *C* – slice of human tooth, *D* – acrylic used for mounting the prepared tooth, *E* – shear blade of the testing machine, and *F* – direction of the applied force

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

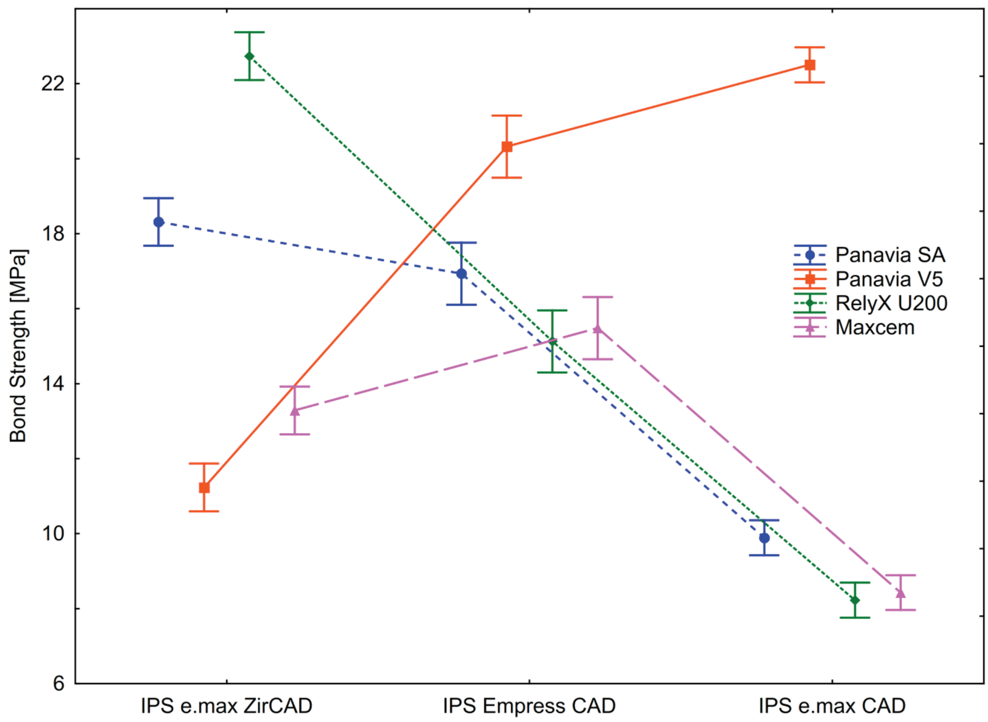

Fig. 2. Shear bond strength of the examined samples. Vertical bars denote 0.95 confidence intervals

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.

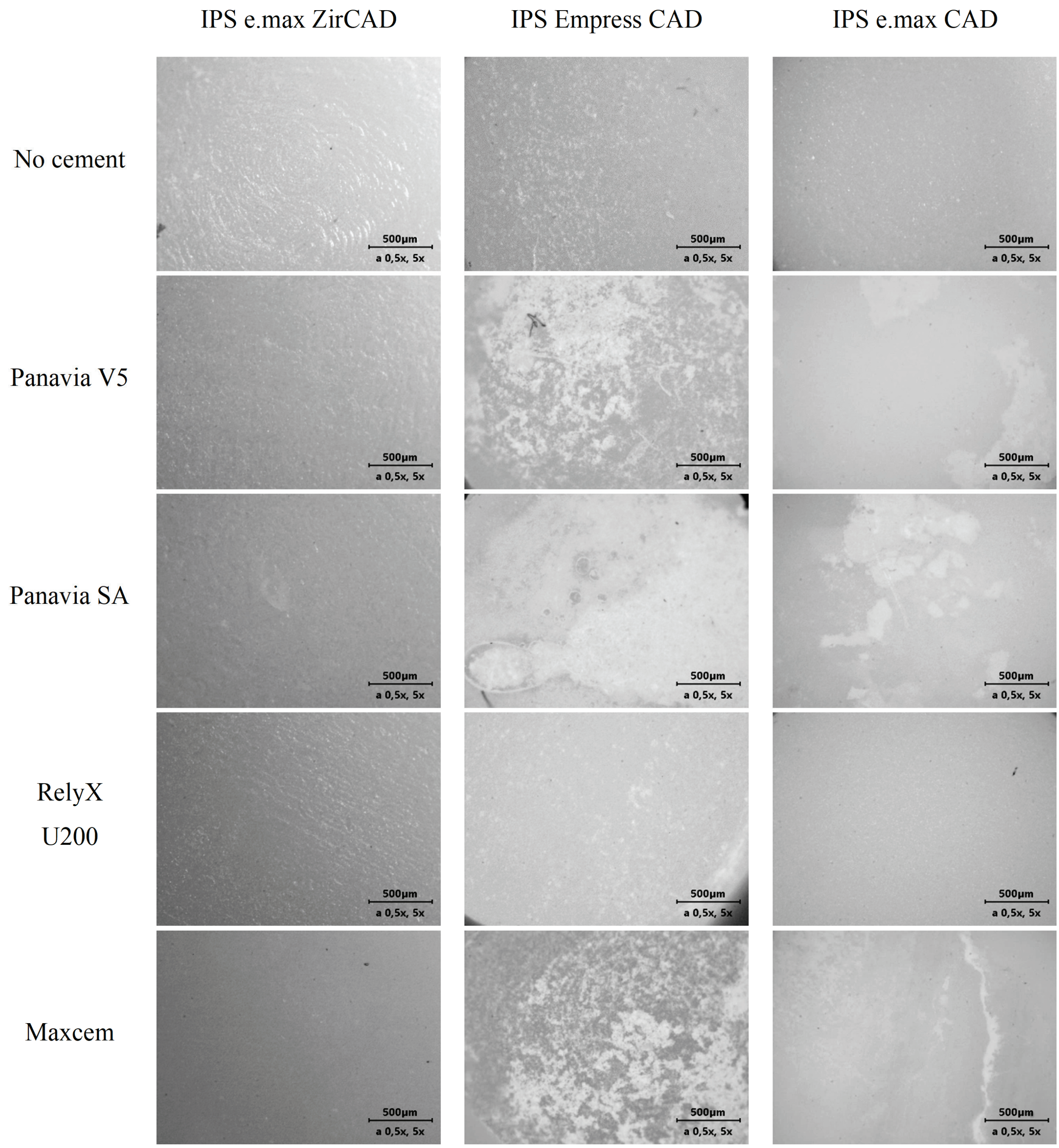

Fig. 3. Light microscope photographs of different failure modes observed on ceramic surfaces

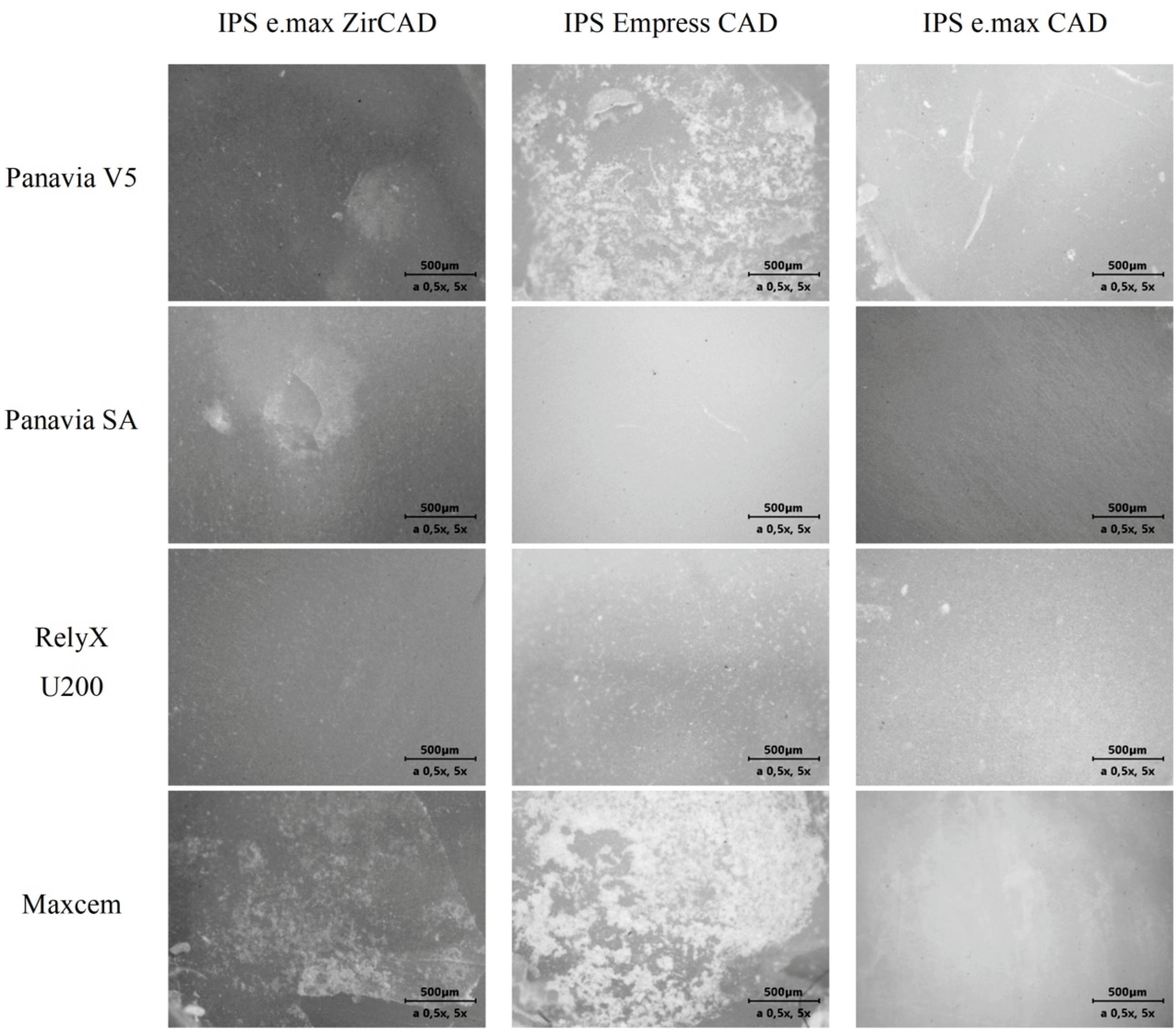

Fig. 4. Light microscope photographs of different failure modes observed on dentin surfaces

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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