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Characterization of the mechanical properties of CAD/CAM polymers for interim fixed restorations

Leila PEREA-LOWERY[1], Mona GIBREEL[1], Pekka K. VALLITTU[1,2] and Lippo LASSILA[1]

[1] Department of Biomaterials Science and Turku Clinical Biomaterials Center–TCBC, Institute of Dentistry, University of Turku, Finland
[2] City of Turku Welfare Division, Oral Health Care, Finland
Corresponding author, Leila PEREA-LOWERY; E-mail: leila.perea@utu.fi

This study investigated some mechanical properties of five CAD/CAM materials used for the fabrication of provisional restorations and tooth segments for digitally fabricated dentures. The CAD/CAM blocks were sectioned into bars for flexural strength and elastic modulus testing (n=80), and for surface microhardness (n=80). Half of the specimens were water-stored for 30 days while the other half was dry-stored. Additional specimens were prepared for bond strength (n=40). A 2-way analysis of variance (ANOVA) was conducted to detect the effect of material and water storage (α=0.05). Statistical software (IBM SPSS Statistics v21; IBM) was used for conducting all analyses. Material type and storage significantly affected the flexural strength, flexural modulus and microhardness (p<0.001). The type of material did not have a significant effect on bond strength (p>0.05). The tested materials showed variation in their flexural properties and surface microhardness whereas their bonding properties with resin luting cement were similar.

Keywords: CAD/CAM, PMMA, Digitally fabricated dentures, Flexural strength, Surface hardness

INTRODUCTION

The composition and quality of the materials used for the construction of temporary crowns and bridges are essential for the treatment outcomes of fixed prosthetic restorations[1)]. They are placed intraorally during the period between tooth preparation and cementation of the final restoration. In the case of implant-supported restorations, interim prostheses on immediately or conventionally loaded implants are crucial to restore function and esthetics during the osseointegration period, as well as to manage the healing of soft tissue around the implants[2-5)]. Accordingly, these types of restorations have to fulfill certain biological, esthetical, and mechanical requirements[6)].

The favorable outcomes of interim restorations depend on various factors, including their ability to withstand masticatory forces[7)]. Additionally, they protect the prepared tooth structure, the pulp, and the periodontal ligament in the case of natural teeth[6-8)], as well as to shape and preserve the coronal mucosa surrounding dental implants[9)] in the case of implant-supported restorations. They are placed intraorally immediately after their fabrication, being subjected to functional loading during mastication[10)]. Interim prostheses are commonly used for extended time when they are associated with implant rehabilitation or comprehensive occlusal reconstruction procedures[11,12)].

Commonly, over impression technique is used to fabricate interim restorations where templates of the desired morphology are filled with resin material and placed on the prepared tooth[13)]. These procedures are considered time-consuming and could also lead to voids incorporation within the restoration affecting its mechanical properties and marginal integrity[14)]. Laboratory processed restorations which are based on polymethyl methacylate (PMMA) are frequently used as interim prostheses, however, high incidence of fractures have been reported although the prostheses can be specifically reinforced[15-17)]. Furthermore, conventionally manufactured PMMA based temporary restorations have lower color stability and increased polymerization shrinkage which affects precision of fit. Some reports show their association with lower values of flexural strength immediately after their manufacturing process[1,16)].

Computer assisted design/computer-assisted manufacture (CAD/CAM) technologies have been introduced for the fabrication of interim restorations. Polymers which have various cross-linking densities are used for fabricating CAD/CAM provisional restorations[18,19)]. These cross-linked materials provide different mechanical properties depending on their chemical composition[20.21)]. The manufactures of these kinds of products as well as some researchers have reported their higher mechanical properties, better color stability, and more accurate marginal fit when compared with the conventionally polymerized resin[20,21-23)]. Additionally, they are considered to be time-saving[24)], and of easy machining without the need for reinforcement such as fibers or metal wires[17,25,26)].

The mechanical properties of interim restorations are of particular significance as they might affect the integrity of temporary reconstructions when they are exposed to functional loads[27)]. Therefore, the aim of this study was to investigate some mechanical properties of five CAD/CAM materials used for the fabrication of provisional restorations and tooth segments for

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digitally fabricated dentures. The mechanical properties investigated were flexural strength, flexural modulus, surface microhardness and bond strength to an adhesive resin cement under two different storage conditions. Our research hypothesis was that the differences in the materials and the storage conditions would significantly affect the evaluated properties.

MATERIALS AND METHODS

In the present *in vitro* study, a variety of CAD/CAM materials were evaluated: Degos Dental L-Temp Multicolor (Degos Dental, Regenstauf, Germany), SR Vivodent CAD (Ivoclar Vivadent, Schaan, Liechtenstein), Zirkonzahn Temp basic (Zirkonzahn, South Tyrol, Italy), Zirkonzahn Multistratum flexible (Zirkonzahn), and Harvest ZCAD™ Temp Esthetic (Harvest Dental Products, Brea, CA, USA). The tested materials are used for fabricating provisional restorations except SR Vivodent CAD, which is a tooth material used for digitally fabricated dentures. The tested materials are listed in Table 1.

Flexural strength and flexural modulus testing

For each material, blocks were sectioned using a water-cooled diamond saw (Struers Secotom-50, Ballerup, Denmark) into equal bar-shaped (2×2×25 mm) specimens. For each material type half of the specimens were tested after 1 month of dry storage (n=8) while the other half was stored for 30 days in distilled water at 37°C before testing. A static 3-point bending test (Model LRX, Lloyds Instruments, Hampshire, UK) was performed in air to determine the flexural strength and flexural modulus. The testing machine was programmed to a constant displacement rate of 1 mm/min, a pre-load of 1.0 N, a pre-load speed of 10 mm/min and the distance between the supports of the test specimens was 20 mm. The test was considered finished when the current load was reduced to 50% of the maximum load or was less than 1.0 N.

Surface microhardness testing

Eighty specimens of (4×10×10 mm) were obtained (n=16/material). They were wet ground flat with 1200 grit (FEPA) silicon carbide grinding paper. The specimens were then cleansed in deionized water in an ultrasonic cleaning device (Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA) for 10 min. Half of the specimens were stored dry while the rest were stored in distilled water at 37°C for 30 days. Surface microhardness testing was performed on selected portions of the specimens with a Vickers hardness testing machine (Duramin-5, Struers). The force used was 245.2 mN for 15 s. One indentation was made on each specimen to obtain the surface microhardness value and the deformation of the indentation was measured after 3 s from the point of releasing the load.

Bond strength

An autopolymerizing acrylic resin (Palapress, Kultzer, Hanau, Germany) was used as a base material into which the CAD/CAM materials were embedded (20×10×2 mm). A total of forty specimens were prepared (n=8/material). The specimens were wet ground flat with 1200 grit (FEPA) silicon carbide grinding paper and afterwards cleansed in deionized water in an ultrasonic cleaning device (Quantrex 90, L&R Ultrasonics) for 10 min and allowed to dry under ambient laboratory conditions (23±1°C).

The bonding surfaces of the specimens were next treated with a universal adhesive (Scotchbond, 3M ESPE, St. Paul, MN, USA) using a fine microbrush following the manufacturer' recommendations. Thereafter, self-adhesive resin cement (Relyx Unicem, 3M ESPE) was applied in 1 mm increments to the substrate surface using a translucent polyethylene mold, with a diameter of 3.6 mm and height of 4 mm. Each specimen was then photopolymerized with a hand-held light-polymerizing unit (Elipar S10, 3M ESPE) for 40 s. Next, the specimens were stored in dry conditions for 24 h at room temperature (23±1°C). Bond strength testing was performed with a universal testing machine (Model LR 30K plus, Lloyds Instruments). Data were recorded with data analysis software (Nexygen, Lloyd instruments). The specimens were loaded at the interface of the substrate and the resin cement at a 1.0 mm/min crosshead speed until fracture occurred. Bond strengths were calculated in MPa. The fracture modes of the samples were analyzed visually and classified as adhesive or cohesive failures.

A specimen of each material was placed in Tetrahydrofuran (THF; Sigma-Aldrich, St. Louis, MN, USA) for 10 s and allowed to dry under ambient laboratory conditions for 60 min. This was made in order to identify differences in the materials' cross-linking densities. An

Table 1 Materials used

Material	Manufacturer	Indication
L-Temp Multicolor	Degos Dental	Long-term temporary restorations
SR Vivodent CAD	Ivoclar Vivadent	Tooth segments in removable denture prosthetics (digital denture)
Temp basic	Zirkonzahn	Short-term temporary restorations
Multistratum flexible	Zirkonzahn	Long-term provisional restorations
ZCAD™ Temp Esthetic	Harvestdental	Long-term provisional restorations

evaluation of the gold sputtered surfaces was performed with a scanning electron microscope (SEM; JSM 5500, Jeol, Tokyo, Japan) to visually analyze the polymer structure of the different CAD/CAM materials.

All data for flexural strength, flexural modulus, surface microhardness, and bond strength were collected and statistically analyzed. A 2-way analysis of variance (ANOVA) was conducted to detect the effect of material and water storage as the independent variables on the evaluated properties (α=0.05). Statistical software (IBM SPSS Statistics v21, IBM, Redmond, WA, USA) was used for conducting all analyses.

RESULTS

The statistical analysis by 2-way ANOVA showed that material type and water storage significantly affected the flexural strength, flexural modulus and surface microhardness (p<0.001). Additionally, the interaction between material and water storage was significant (p<0.001). Material type did not seem to have a significant effect on bond strength (p>0.05) (Fig. 1). The mean values for flexural strength, flexural modulus, and surface microhardness of the tested groups are presented in Table 2.

For dry specimens, the mean flexural strength and flexural modulus values of Zirkonzahn Temp basic specimens were significantly lower than the other 4 materials (p<0.001). Figure 2 shows a load-deflection graph as a graphic representation of the behavior of the tested materials. A non-significant difference was found for flexural strength among Zirkonzahn Multistratum flexible, SR Vivodent CAD, L-Temp multicolor, and ZCAD™ Temp (p=0.207).

For flexural modulus, SR Vivodent CAD specimens were significantly higher than Zirkonzahn Temp basic and Zirkonzahn Multistratum flexible (p<0.001) for water and dry-stored specimens, and not significantly different from ZCAD™ Temp Esthetic and Degos Dental L-Temp Multicolor (p>0.05). For surface microhardness under dry and wet conditions, Zirkonzahn Temp basic and Zirkonzahn Multistratum flexible specimens recorded the lowest values, which were significantly different from the other 3 materials (p<0.001). SR Vivodent microhardness values were not significantly different from L-Temp Multicolour and ZCAD™ Temp Esthetic with the two storage conditions (p>0.05).

Visual examination revealed only adhesive failure types for all tested materials after bond strength testing. Additionally, only specimens fabricated from Zirkonzahn Multistratum flexible bended without fracturing when subjected to flexural strength testing.

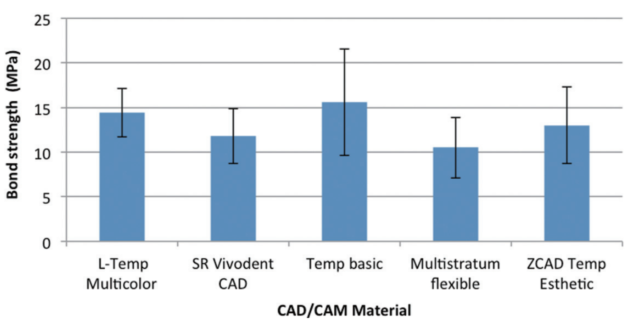

Fig. 1 Bond strength of tested materials.

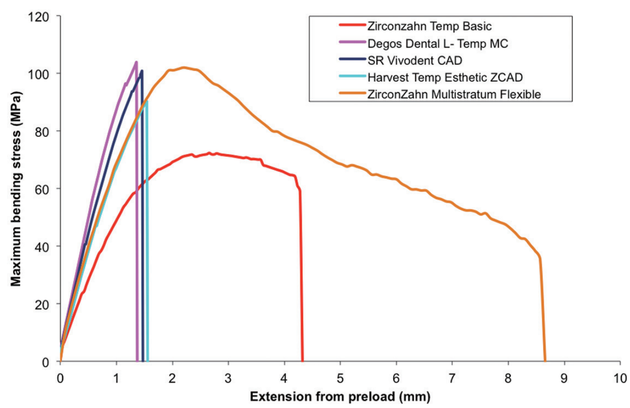

Fig. 2 Load-deflection graph representing the maximum bending stress (MPa) of the tested materials.

Table 2 Mean flexural strength (FS), flexural modulus (FM), and surface microhardness values of the materials investigated

Material	FS (MPa)		FM (GPa)		Surface hardness (VHN)	
	Dry	Water	Dry	Water	Dry	Water
Degos Dental L-Temp MC	102 (8) [a]	108 (12) [a]	3.0 (0.3) [a]	3.1 (0.8) [ac]	22 (0.7) [a]	21 (0.3) [a]
SR Vivodent CAD	105 (11) [a]	117 (11) [ac]	3.0 (0.2) [a]	3.7 (0.4) [ad]	22 (0.8) [ac]	20 (0.5) [a]
Zirkonzahn Temp Basic	74 (12) [b]	64 (12) [b]	1.6 (0.2) [b]	1.1 (0.1) [b]	16 (0.5) [b]	12 (0.6) [b]
Zirkonzahn Multistratum Flexible	109 (12) [a]	124 (8) [c]	2.2 (0.3) [c]	2.7 (0.4) [c]	17 (0.9) [b]	15 (0.6) [c]
Harvest Temp Esthetic ZCAD	96 (17) [a]	131 (11) [c]	2.8 (0.3) [a]	4.0 (0.2) [d]	21 (0.8) [c]	20 (0.4) [a]

(SD) standard deviation.
Materials labeled with similar letters in each column are not statistically different.

Table 3 Advantages and disadvantages identified in the materials investigated

Material	Advantage	Disadvantage
Degos Dental L-Temp MC	Natural aesthetic High flexural strength, flexural modulus and surface hardness Flexural strength and flexural modulus increase after water storage	Decreased surface hardness after water storage
Zirkonzahn Temp Basic	Pleasant cosmetic-esthetic values	Lowest mechanical properties (flexural strength, flexural modulus, and surface hardness) among the investigated materials Decreased mechanical properties after water storage
Zirkonzahn Multistratum Flexible	Multicolor shading High flexibility (low risk of fracture) High flexural strength Flexural strength and flexural modulus increase after water storage	Decreased surface hardness after water storage
SR Vivodent CAD	High esthetic appearance High flexural strength, flexural modulus and surface hardness Flexural strength and flexural modulus increase after water storage	Decreased surface hardness after water storage
Harvest ZCAD™ Temp Esthetic	Good anterior esthetics High flexural strength, flexural modulus and surface hardness Flexural strength and modulus increased significantly after water storage Surface hardness is not significantly affected by water storage	—

Table 3 summarizes the advantages and disadvantages identified in the materials investigated. Figure 3 shows the differences in the polymer structure of the materials after being treated with solvent THF. This figure shows that SR Vivodent CAD had some phases which behaved differently by the solvent treatment whereas the others had homogeneous looking surface after being solvent treated.

DISCUSSION

The aim of this study was to investigate the mechanical properties of five CAD/CAM materials used for the fabrication of provisional restorations or digital denture teeth in terms of flexural strength, flexural modulus, surface microhardness, and bond strength under two different storage conditions. The results of the study supported the research hypothesis, which stated that, the differences in material type and storage conditions would significantly affect the evaluated mechanical properties. However, the materials' differences did not seem to affect the bond strength of the materials investigated.

Interim restorations fabricated from industrially polymerized resins for CAD/CAM manufactured prostheses might be suitable to be used as long-term reconstructions. The reasoning lies on their superior mechanical properties and better marginal fit when compared with those fabricated manually[16,22,28]. The use of these kinds of CAD/CAM materials for interim restorations also offers new treatment options as it is the case in complex treatments and immediate loading protocols[18,25]. Long-term interim prostheses are commonly used for implant-supported treatments, as well as for periodontal therapy that requires extended follow-up and maxillofacial rehabilitations where the prostheses could be exposed to functional loading[3,29]. Clinical studies have investigated the effect that the type of material has on the prosthetic complications' rate[4]. Some authors have reported this prosthetic complication rate for temporary restorations as 17.1% and less than 1% for definitive prostheses[30].

It was found in the current study that Zirkonzahn Temp Basic showed the lowest values for flexural strength, flexural modulus, and surface microhardness when tested dry or after 30 days water storage. This was in agreement with the findings reported by some authors[31] when they evaluated the load-bearing capacities of resin-based fixed dental prostheses. They found that restorations fabricated from Zirkonzahn Temp

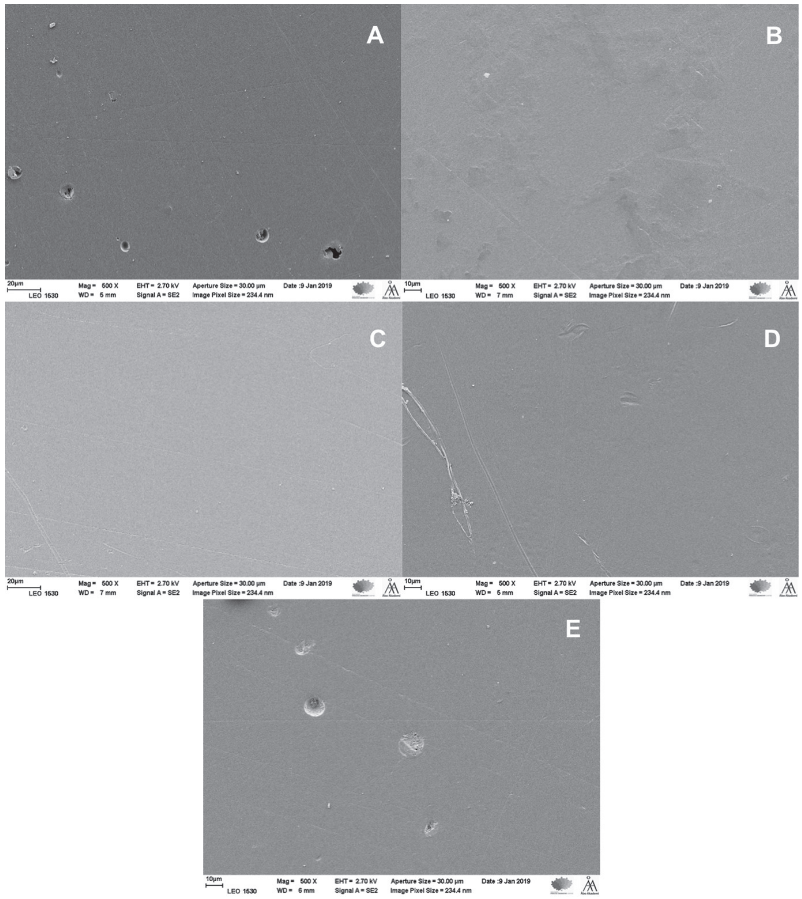

Fig. 3 SEM images (500×) showing the polymer structure of the materials investigated after the surface has been treated with solvent THF. A: L-Temp MC, B: SR Vivodent CAD, C: Zirkonzahn Temp Basic, D: Zirkonzahn Multistratum Flexible, E: ZCAD Temp Esthetic.

basic recorded the lowest values (280±87.3 N) and were not significantly different from those fabricated with conventional techniques. Therefore, this material might be suitable for short-term provisional restorations of up to six months as it is indicated by the manufacturer.

In the present *in vitro* study, SR Vivodent CAD recorded high values for flexural strength, flexural modulus, and surface hardness, which were not statistically significantly different from the other three materials (Zirkonzahn Multistratum flexible, Degos Dental L-Temp multicolor, and ZCAD™ Temp Esthetic) under dry conditions. This difference is most likely related to the different chemical composition of the materials investigated. This composition in the case of SR Vivodent CAD is a PMMA-based double cross-linked material, which means that the polymer filler and matrix are homogeneously cross-linked. The result is a thoroughly cross-linked material system, offering substantial advantages in terms of bond to denture base materials and improved mechanical properties[32].

L-Temp multicolor and ZCAD™ Temp Esthetic materials consist of a cross-linked PMMA which is known as interpenetrating polymer network (IPN) material which is produced by polymers of different chemical and physical natures that penetrate each other and become interlaced with the help of swelling processes[33,34]. The glass transition temperature of regular non-cross linked PMMA is 125°C. Cross-linking increases the glass-transition temperature and hence the mechanical strength of the product[35]. Additionally, it improves the material fracture resistance[36]. This is in agreement with a study reported in the literature where higher flexural strength values were found for fixed partial dentures fabricated from a highly cross-linked PMMA when compared with those fabricated from a non-cross-linked material[20].

Surface hardness can predict the wear resistance of a material and its ability to abrade the opposing structure[35]. Although Zirkonzahn Multistratum flexible recorded high values for flexural strength, their flexural modulus and surface hardness values were significantly lower than the other tested materials except for Zirkonzahn Temp basic. This might be attributed to its flexibility since Zirkonzahn Multistratum flexible

consists of a thermoplastic resin, which is a linear polymer without cross-linking agent as stated by the manufacturer[37,38]. Interestingly, the THF solvent treatment test did not reveal dissolving of surface which may suggests that the polymer structure although it was linear (not cross-linked) was syndiotactic or isotactic providing better capability to resist effects by solvents[38-41].

Previous studies concluded that thermoplastic resins with low modulus of elasticity and nanohardness were more liable to wear when compared with PMMA[42]. In a previous study[43], significantly greater wear resistance was found on denture teeth fabricated from DCL material or highly cross-linked PMMA when compared to those made of conventional acrylic. The statistical analysis in this study showed that the storage condition had a significant effect on the tested parameters. Water storage significantly increased the flexural strength and flexural modulus of SR Vivodent CAD, Multistratum Flexible, and ZCAD™ Temp Esthetic. However, it significantly decreased their surface microhardness except for ZCAD™ Temp Esthetic. This was in agreement with the results of a study that compared the fracture strength of temporary fixed partial dentures fabricated from CAD/CAM versus directly fabricated restorations after water storage at 37°C for 3 months[20]. They attributed that to the fact that CAD/CAM blocks are polymerized under optimal conditions, without the interference of water. During the water storage period of these blocks, post polymerization processes as well as relaxation phenomena may have occurred causing an improvement of the physical properties.

This study also found a non-significant difference in terms of bond strength between the groups investigated. A previous study did not find any connection between chemical composition (cross-linking agents or conventional PMMA) and bond strength of artificial teeth[44]. Similar bonding characteristics of the tested materials is supported also by the findings of the THF solvent treatment test: The materials had similar behavior, which suggests that the bonding based on the surface dissolution (so-called secondary IPN bonding) do not considerably differ between the studied materials[45].

For implant-supported dental prostheses, firm primary implant stability, immediate splinting, and controlled occlusion are essential parameters for achieving successful clinical outcomes. Implant splinting with a rigid implant-supported bridge is thought to minimize transferring occlusal loads to the implants[46,47]. Additionally, it can be beneficial in minimizing lateral forces on implants if more than 2 implants are involved[48]. Therefore, based on the results of this study, SR Vivodent CAD, Harvest ZCAD™ Temp Esthetic, and Degos Dental L-Temp MC might be considered for constructing implant-retained provisional bridges as a first choice instead of Zirkonzahn Temp Basic and Zirkonzahn Multistratum Flexible.

The results of this study convey advice to clinicians in terms of selecting carefully CAD/CAM disks when planning interim fixed restorations because disks from different brands vary in strength. As static loads were applied in this study and results may be different when cyclic loads are used, the results of the current study should be elucidated accordingly. The results presented here should also allow clinicians to make comparisons among different systems concerning the performance of the tested materials under standardized conditions.

CONCLUSION

The tested materials showed variation in their flexural properties and surface microhardness whereas their bonding properties with resin luting cement were similar.

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