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DOCTORAL THESIS

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DOCTORAL THESIS

Supervisor:

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Stomatološki fakultet

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UTJECAJ TEHNOLOŠKOG PROCESA PROIZVODNJE NA MEHANIČKA SVOJSTVA I VEZNU ČVRSTOĆU GRADIVNIH MATERIJALA PROTEZE

DOKTORSKI RAD

Mentor:

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SUMMARY

Computer-aided design/computer-aided manufacturing (CAD/CAM) technology is one of the fastest-growing aspects of modern dentistry, effecting all disciplines of dentistry, especially the fields of prosthodontics and restorative dental medicine. New materials and techniques are being continuously improved. The data about digital technologies and mechanical properties of these materials are scarce.

The aim of the first part of the present study was to estimate and collate the mechanical properties of different materials and technologies for denture base manufacturing. The evaluated mechanical properties were flexural strength, surface hardness, and impact strength. The aim of the second part of the present study was to define the shear bond strengths of different denture base resins to different types of prefabricated teeth (acrylic, nanohybrid composite, and cross-linked) and denture teeth produced by CAD/CAM technology (milling).

Milled CAD/CAM materials for denture base fabrication showed enhanced mechanical properties when compared to heat-polymerized and 3D printed acrylics. 3D printed acrylics had mechanical properties lower than the majority of other tested denture base resins. Nevertheless, material's polymerization type cannot guarantee its optimal mechanical properties. When comparing shear bond strengths between denture base materials and prefabricated teeth, heat-polymerized and milled CAD/CAM denture base resins showed higher shear bond strength values compared to the cold-polymerized ones. Cold-polymerized resins should be averted for binding artificial teeth to denture base resins. Denture teeth glued to a milled CAD/CAM denture base resin by using a bonding agent show the same shear bond strength values as the denture teeth adhered to heat-polymerized denture base resins.

Keywords: acrylic resin, flexural strength, surface hardness, complete denture, digital denture, prefabricated teeth

PROŠIRENI SAŽETAK

Svrha rada

Napredak i razvoj dentalne medicine očituje se kroz dostupnost i uporabu novih materijala, jednako kao i njihovih novih tehnika obrade. Svrha ovog istraživanja bila je testirati mehanička svojstva materijala za izradu baza potpunih proteza izrađenih različitim tehnologijama, s naglaskom na digitalne tehnologije (*computer-aided design*/*computer-aided manufacturing* [CAD/CAM] što uključuje glodanje i trodimenzionalni ispis [3D *printing*]), te ispitati veznu čvrstoću između različitih vrsta zuba i baze proteze.

Materijali i postupci

Prvi dio istraživanja testirao je mehanička svojstva (savojna čvrstoća, tvrdoća i žilavost) materijala rabljenih za izradu baze potpunih proteza: tri različita toplopolimerizirajuća akrilata, tri CAD/CAM akrilata (glodanje), 3D printani akrilat te poliamid. Drugi dio istraživanja uspoređivao je veznu čvrstoću između različitih vrsta prefabriciranih zubi (akrilatni, nanohibridno kompozitni i umreženi) i umjetnih zubi dobivenih glodanjem s različitim materijalima za izradu baze potpunih proteza. Drugi dio istraživanja uključio je 10 grupa: hladnopolimerizirajući akrilat i akrilatne zube, hladnopolimerizirajući akrilat i zube od nanohibridnog kompozita, hladnopolimerizirajući akrilat i umrežene zube, toplopolimerizirajući akrilat i akrilatne zube, toplopolimerizirajući akrilat i zube od nanohibridnog kompozita, toplopolimerizirajući akrilat i umrežene zube, CAD/CAM (glodani) akrilat i akrilatne zube, CAD/CAM (glodani) akrilat i zube od nanohibridnog kompozita, CAD/CAM (glodani) akrilat i umrežene zube, CAD/CAM (glodani) akrilat i zube dobivene CAD/CAM obradom (glodanjem).

U prvom dijelu istraživanja blokovi toplopolimerizirajućeg akrilata pripremili su se kivetiranjem dok su se blokovi poliamida pripremili postupkom injekcijskog prešanja prema uputama proizvođača. 3D printani uzorci (blokovi) pripremili su se prema STL datoteci u odgovarajućoj jedinici za 3D printanje, uz naknadnu svjetlosnu polimerizaciju u odgovarajućem uređaju prema uputama proizvođača. Svi navedeni uzorci (toplopolimerizirajući, poliamidni, 3D printani i CAD/CAM blokovi za glodanje) obradili su se na stroju za rezanje s vodenim hlađenjem (IsoMet 1000). Sve su se plohe dodatno obrađivale uporabom standardnog metalografskog papira za brušenje (P500, P1000 i P1200) do zadanih dimenzija s glatkim plohama. Savojna čvrstoća mjerila se testom trotočkastog opterećenja uporabom univerzalnog stroja za testiranje (10 uzoraka za svaki testirani materijal dimenzija 64,0 x 10,0 x 3,3 ± 0,2 mm). Prije testiranja uzorci su bili uronjeni u vodenu kupku na 50 ± 1 sati, temperature 37 °C. Deset uzoraka dimenzija 64,0 x 10,0 x 3,3 ± 0,2 mm svakog materijala uporabila su se za testiranje tvrdoće. Sila od 358 N aplicirala se preko kuglice na vrijeme od 60 sekundi. Brinellova tvrdoća mjerila se na pet mjesta na svakom uzorku nakon čega je određena prosječna tvrdoća svakog uzorka. Ispitivanje udarne žilavosti provelo se na Charpyjevom batu. Ispitivanje žilavosti provelo se mjerenjem točnih dimenzija uzorka s odgovarajućim urezanim utorom nakon čega se uzorak stavljao u oslonac, te se na njega s određene visine spustio bat koji je slobodnim padom lomio uzorak. Svaka ispitna skupina imala je 10 uzoraka dimenzija 80,0 x 10,0 x 4,0 ± 0,2 mm.

Za drugi dio istraživanja uzorci su pripremljeni na sljedeći način: prefabricirani zubi uronjeni su u bezbojnu hladnopolimerizirajuću smolu u plastičnome kalupu. Nakon polimerizacije uzorci su izvađeni iz plastičnih kalupa. Cervikalna površina prefabriciranog zuba smještenog u bezbojnu smolu izložena je na rezalici s vodenim hlađenjem (IsoMet 1000). Metalni kalup uporabljen je za dobivanje silikonskog uzorka dimenzija 25,0 mm x 2,5 mm. U samom središtu silikonskog uzorka nalazio se kružni otvor dimenzija 5,0 mm x 2,5 mm koji je uporabljen za izradu cilindara. Navedeni silikonski uzorak zalijepio se na izloženu površinu zuba i hladnopolimerizirajuće smole uporabom univerzalnog ljepila. Cilindri toplopolimerizirajućeg akrilata izrađeni su postupkom kivetiranja dok su cilindri hladnopolimerizirajućeg akrilata izrađeni uporabom polimerizatora (Ivomat IP2) pri temperaturi od 40 °C i tlaku od 6 bara. Glodalica (Ceramill Mikro 5X) je rabljena za dobivanje CAD/CAM (glodanih) cilindara. CAD/CAM (glodani) cilindri zalijepljeni su adhezivnim sredstvom za CAD/CAM (glodani) materijal za izradu baze potpune proteze. Za testiranje uzoraka upotrijebljen je test smične čvrstoće (Nexygen). Uporabom stereomikroskopa (Olympus SZX10) prijelom je okarakteriziran kao adhezivan, kohezivan ili mješovit.

Rezultati

Glodani CAD/CAM materijal za izradu baze potpune proteze (IvoBase CAD) i poliamid pokazali su najveće vrijednosti savojne čvrstoće (ni jedan uzorak nije frakturirao tijekom testa trotočkastog opterećenja). U dvije skupine glodanih CAD/CAM materijala utvrđene su najviše vrijednosti tvrdoće (Interdent CC disc PMMA - 145,66 ± 2,22, te Polident CAD/CAM disc basic - 143,82 ± 2,22) dok je treća skupina pokazala najniže vrijednosti tvrdoće (Ivobase CAD - 95,54 ± 9,82), baš kao i poliamid (Vertex ThermoSens - 67,13 ± 10,64). 3D printani materijal (Nextdent Base) pokazao je najniže vrijednosti savojne čvrstoće (71,70 ± 7,38). Medijan test pokazao je značajne statističke razlike između testnih skupina za savojnu čvrstoću i tvrdoću ($p < 0{,}001$). Vrijednosti žilavosti iznosile su od 8,01 ± 3,52 kJ/m^2 do 44,68 ± 39,37 kJ/m^2. Medijan test pokazao je razlike između materijala prilikom testiranja žilavosti ($p < 0{,}001$).

Testom smične čvrstoće utvrđene su najniže vrijednosti između hladnopolimerizirajućeg akrilata i svih testiranih vrsta prefabriciranih zubi (akrilatni **-** 3,37 ± 2,14; nanohibridno kompozitni **-** 10,14 ± 3,30; umreženi **-** 10,06 ± 4,35). Između toplopolimerizirajućeg akrilata i CAD/CAM (glodanog) materijala za izradu baze potpune proteze, te testiranih umjetnih zubi nije pronađena značajna razlika ($p > 0{,}05$).

Zaključak

CAD/CAM materijali za izradu baze proteze (glodanje) općenito pokazuju bolja mehanička svojstva u odnosu na toplopolimerizirajuće akrilate i 3D printani akrilat. Unatoč tome, tip polimerizacije materijala ne jamči njegova optimalna mehanička svojstva. Postoje razlike između različitih kombinacija materijala za izradu baze potpune proteze i prefabriciranih zubi. Hladnopolimerizirajući akrilati trebali bi se izbjegavati prilikom pričvršćivanja prefabriciranih zubi u bazu proteze. CAD/CAM (glodanje) i toplopolimerizirajući akrilati za izradu bazu proteze u kombinaciji s prefabriciranim zubima imaju slične vrijednosti prilikom testiranja smične čvrstoće.

Ključne riječi: akrilat, savojna čvrstoća, tvrdoća, potpuna proteza, digitalna proteza, prefabricirani zubi

The list of abbreviations

PMMA	Polymethyl-methacrylate
CAD/CAM	Computer-aided design/computer-aided manufacturing
3D	Three dimensional
CP	Cold-polymerized
HP	Heat-polymerized
MMA	Methyl methacrylate
FS	Flexural strength
SH	Surface hardness
IS	Impact strength
TMD	Temporomandibular disorders
SLA	Stereolithography
DLP	Digital light processing
SBS	Shear bond strength
IPN	Interpenetrating polymer network
DCL	Double crosslinked
NHC	Nanohybrid composite
UDMA	Urethane dimethacrylate
ANOVA	Analysis of variance

CONTENT

1. INTRODUCTION

1.1 Polymethyl metacrylate

1.1.1 Structure

Polymethyl methacrylate (PMMA) has been used in dental medicine for more than 60 years and remains the main denture base material (1–3). It is not only a denture base material but can also be used for denture relining, rebasing, temporary crowns, maxillofacial prosthesis, splints for surgical and gnathological procedures, and orthodontic prosthesis (1). PMMA (poly[1-(methoxy carbonyl)-1-methyl ethylene]) is usually attainable as a powder-liquid form. The powder includes a clear polymer (PMMA). Pigments and nylon or acrylic synthetic fibers are assigned to modify mechanical properties and aesthetics (to simulate mucosa and teeth). The liquid component includes a monomer of methyl methacrylate, cross-linking agents, and inhibitors (4). Free radical addition and polymerization of methyl methacrylate ($C_5O_2H_8$) to poly methyl methacrylate $(C_5O_2H_8)_n$ occur during the polymerization reaction. A polymerization process can be activated by generating a free radical chemical substance or with energy (heat, light, microwaves). The process continues with the binding of monomers. A final phase of polymerization is termination, which happens with the shifting of free electrons to the chain end (4).

According to their polymerization type, acrylic resins can be divided into several groups; cold-polymerized acrylic resins, heat-polymerized acrylic resins, light-polymerized acrylic resins, acrylic resins polymerized with a microwave, and computer-aided design/computer aided manufacturing (CAD/CAM) PMMA resins (5, 6).

Cold-polymerized acrylic resins have a different mechanism of polymerization process compared to heat-polymerized acrylic resins because they do not request thermal energy. A tertiary amine initiator (dimethyl-p-toluidine) activates the benzyl peroxide, which initiates the polymerization process. Even though, the degree of polymerization of cold-polymerized acrylic resins is significantly lower when compared to heat-polymerized acrylic resins, leaving residual monomer in the polymerized material (4, 7).

Heat-polymerized acrylic resins are attainable in powder-liquid form and are being continually used for a denture base fabrication. The powder includes PMMA, initiator (benzoyl peroxide), opacifiers (titanium and zinc oxides), plasticizer (dibutyl phthalate), fibers, and pigments or dyes.

The liquid includes methyl methacrylate (MMA) monomer, a cross-linking agent (ethylene glycol dimethacrylate), and an inhibitor (hydroquinone) (4, 7). The polymerization begins when powder and liquid are mixed, and the process requests heat energy (e. g. water bath) to trigger off the initiator (4). As a result of a high degree polymerization, heat-polymerized acrylic resins contain favorable properties (4, 7).

Light-polymerized acrylic resins are polymerized when exposed to visible light. The resin is altered by replacing the initiator with a photo-sensitive agent (camphorquinone) (4). Due to its deficiencies (e. g. limited curing, price, and method sensitivity), they are not usually used as a denture base material. The mechanical properties of light-polymerized acrylic resins are inferior in comparison to conventional heat-polymerized acrylics, and for that reason their usage is limited to denture base reparations and relining (4, 5).

Acrylic resins polymerized in a microwave use microwave energy and a non-metallic denture flask to polymerize. These resins do not contain benzoyl peroxide initiator, so they cannot polymerize by using conventional water bath process. Properties of acrylic resins polymerized in a microwave are commensurable with conventional heat-polymerized acrylics. Lean shear bond strength with acrylic teeth represents a major limitation for these resins, and narrows its prosthodontic appliance (3, 4).

Recently, the advancement in digital technology enabled the production of a denture base in one piece, which could accelerate denture manufacturing. Fewer phases in the fabrication process could also decrease possibility of a failure (8, 9).

1.1.2 Mechanical properties

Heat-polymerized acrylic resins are continually being used as a denture base material (2, 10, 11) due to their advantages: biocompatibility, low cost, ease of use, high aesthetics, and color matching ability (11–15). Despite the given properties, they do not function perfectly (2). A poor fatigue failure, dimensional inaccuracy, high thermal expansion, and a fracture of a denture base are some of shortcomings of PMMA (12, 16–18). Allergic reaction to PMMA can also present an unexpected problem for patients and dentists as well (1, 19). Residual monomer in MMA can cause mucosal irritation and contact dermatitis. Besides, in some cases MMA products can cause asthma (1, 19).

Furthermore, the alternative to PMMA material has not been aggrandized yet (16). Consequently, cross-linking, copolymerization, and reinforcement were made to enhance mechanical properties of acrylic resins (1, 20). Glass fibers, metal wires and oxides have been suggested to reinforce acrylic resins. Admixture of metal fillers (aluminium, copper, silver) improved compressive and impact strengths, and fracture resistance of acrylic resin (18). Generally, the reinforcement of PMMA materials improved flexural strength, impact strength and amplified fatigue resistance (1).

Flexural strength (FS) is a material attribute defined as the stress in a material just before it yields in a flexure test. Whereas a denture base can break during its function, it is important that the resin for a denture base fabrication has high flexural strength values (8).

Surface hardness (SH) is a measure of the resistance to localized plastic deformation induced by either mechanical indentation or abrasion. Dentures with insufficient surface hardness values can be damaged by brushing. Plaque retention and pigmentations occur on a damaged denture (8).

Impact strength (IS) is defined as the energy required to fracture a material under an impact force. It can be interpreted as the ability of a material to resist shock loading. Impact fracture may occur when a denture is dropped, so high impact strength value of a denture base material is advisable (22, 23).

A three-point flexure test (Figures 1–3), a surface hardness testing (Figure 4), and an impact strength testing (Figure 5) are usually used for testing mechanical properties of denture base resins (8, 23). A universal testing machine for flexural strength testing increases its loading force from 0, using a steady shift of 5 ± 1 mm/min. Eventually, when a tested specimen cracks, formula $FS = 3FL/2bh^2$ is used for calculation. In the present equation FS stands for flexural strength (MPa), F stands for maximum force applied to a specimen (N), L stands for a distance between the specimen carrier (mm), b stands for specimen width (mm), and h stands for specimen height (mm) (Figures 1-3). Before testing, all specimens should be immersed in a water bath for 50 ± 1 hour at 37 °C (Figure 6). Brinell's method is used for determination of surface hardness. The force of 358 N is applied over a ball at five points on every specimen. The mean value of surface hardness of every specimen is determined after measuring. The formula $HB = F/\pi Dh_k$ is used for calculation. HB stands for Brinell hardness (MPa), F stands for force applied to the specimen (N), D stands for a ball diameter (mm), and h stands for depth of penetration (mm) (Figure 4). Charpy's bar is used for the impact strength testing (22, Figure 5). V notch needs to be cut on every specimen prior testing to target the stress on a certain part of a specimen. Pendulum hammer is used to bring the energy on a specimen. A formula used to calculate impact strength (kJ/m^2) is IS = (E/ab) x 1000, where E stands for absorbed energy (J), a stands for specimen height (mm), and b stands for specimen width (mm) (22).

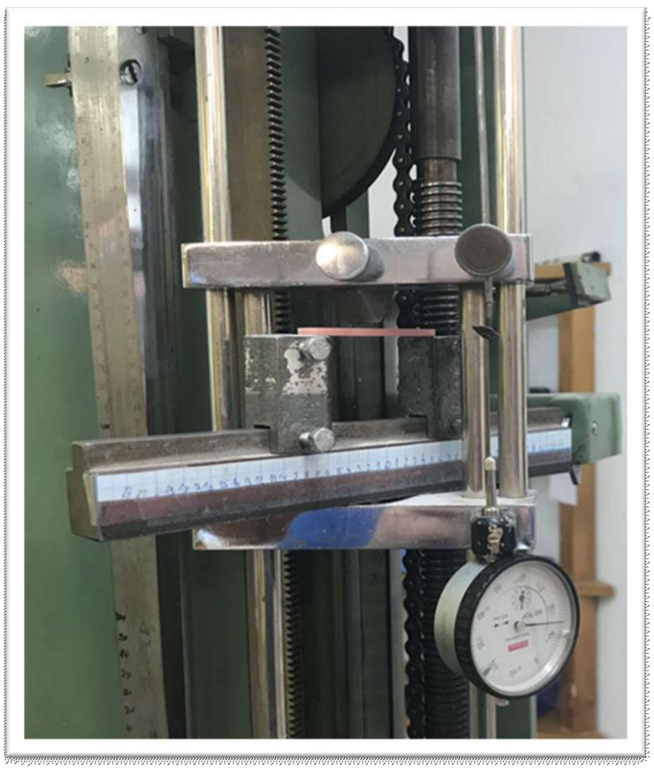

Figure 1. A "three-point flexure test" used for testing flexural strength

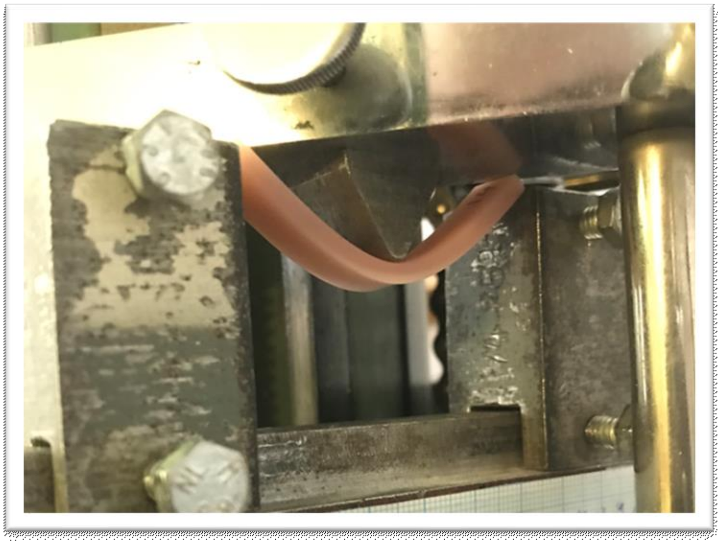

Figure 2. An undamaged CAD/CAM specimen (IvoBase CAD) with loading within the end limits of the possible movement of the penetrant

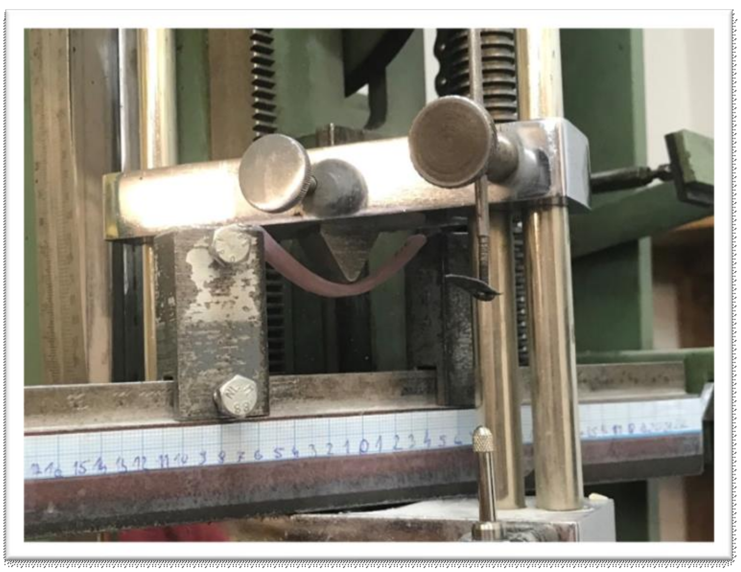

Figure 3. An undamaged polyamide specimen (Vertex ThersmoSens) with loading within the end limits of the possible movement of the penetrant

Figure 4. Zwick apparatus used for surface hardness testing

Figure 5. Charpy's bar used for an impact strength testing

Figure 6. Water bath at 37 °C used for storing specimens before testing

Recently, nylon polymer has attracted focus on itself as a material for a denture base fabrication. Condensation reactions between a diamine, NH_2-$(CH_2)_6$-NH_2, and a dibasic acid, CO_2-H-$(CH_2)_4$-COOH, occur during the process of production of polyamides (24–27). Some of the properties of polyamides such as elasticity, toxicological safety, and polymerization shrinkage are enhanced compared to heat-polymerized acrylic resins. However, polyamides show the following problems: water sorption, surface roughness, color deterioration, and difficulty in polishing. Given properties are inferior compared to heat-polymerized acrylic resins (24, 28).

1.1.3 Usage

Edentulism is defined as the loss of all permanent teeth and is considered a disability by the World Health Organization (WHO) (29, 30). The main cause of edentulism are caries, periodontal disease, pulpal pathology, trauma, and oral cancer (29). Dental caries are the main reason of edentulism among people who are 45 or younger, while periodontal disease is the leading cause of tooth loss in older age (31). According to Nagaraj et al. (29) more than one-third of people aged 65 and over are edentulous. Tooth loss has numerous negative consequences and the most visible is deterioration of orofacial tissues. Reduced chewing efficiency can lead to decreased food intake which can cause malnutrition. Tooth loss and edentulism are also associated with poor oral-health related quality of life (OHRQoL) (30–32). Albeit it is not deathly, edentulism has a great effect on facial appearance, eating ability, speaking, and socializing (33). In addition, some studies reported that edentulism can be related to systematic conditions like obesity, pneumonia, particular cancers, and cognitive decline (34, 35).

There have not been any reported differences of appearance of edentulism between males and females. Nevertheless, it seems that socioeconomic status is related to the percentage of edentulism. A higher percentage of edentulism is associated with a poverty level (36). Moreover, Wu et al. (37) reported the highest prevalence of edentulism in the Native Americans (23.98 %) while Hispanics had the lowest prevalence (14.18 %). Consequently, its prevalence varies between countries and between different areas of the same country (38).

Therapy options for edentulism include prosthodontic rehabilitation, either implant- or tissue-supported, with a fixed or removable solutions (30–32). Complete dentures have been used for a long time, but despite the high price of dental implants (39), they still represent a gold standard for treating edentulism. PMMA is a material of choice for fabricating a complete denture base (1–3), thus most dentures worldwide are still made of heat-polymerized PMMA (20).

The most widespread procedure for the fabrication of denture base resin is called compression molding technique. In the given method, after the wax removal, PMMA in the dough stage is packed into the gypsum mold (gypsum mold is placed in a dental flask). Dental flasks are positioned under pressure in a water bath. PMMA in the dental flask is heated in a water bath under defined time and temperature conditions (4, 40, 41). The temperature of the water bath should be enhanced gradually to provide adequate polymerization. On the other hand, injection molding technique can also be used to produce dentures (4). The given method requires a special dental flask with a sprue (4). PMMA can be used in both techniques (compression molding technique and injection molding technique) (42).

Polyamide is also used as a denture base material , and requires injection molding technique during the fabrication process (24). Polyamide denture base materials are thermoplastic materials which become fluid when heated in an electrical cartridge furnace. Those thermoplastic materials are accessible in the form of granules in cartridges of different sizes. Small ones are used for manufacturing small dimensioned flexible dentures, while big ones are used for large dimensioned removable flexible complete dentures (43).

Polymethyl methacrylate (PMMA) based polymers can also be used for occlusal device fabrication due to its mechanical properties and easy manipulation. Most frequently occlusal devices are fabricated from thermoforming foil and cold-polymerizing PMMA. However, digital technologies (CAD/CAM; milling and 3D printing) can also be used for the manufacturing process (44). Occlusal devices are commonly used for treating temporomandibular disorders (TMD), and to neutralize negative impact of bruxism on the masticatory system (44). The prevalence of bruxism has been reported to be 13% in adults between 18 and 29 years of age (44).

1.2 Digital technologies for denture base fabrication

Considering increased population lifetime, necessity for dental treatment has become greater, especially for edentulous patients. Although there are many possibilities for treating edentulous patients, conventional complete dentures remain a favored option due to anatomical, physiological, and financial limitations (45, 46).

Recent improvements in technology have enabled digital procedures for denture base fabrication – CAD/CAM (47). The manufacturing process consists of scanning the final impressions or casts and maxillomandibular relation, creating a denture base and adjusting the artificial teeth by using software, and manufacturing the denture by using subtractive (milling) or additive (3D-printing) method (48–52).

With a subtractive method, a denture base is milled from prepolymerized PMMA blocks (53). These PMMA blocks are made under high pressure and heat, whereby polymerization shrinkage is prevented. Less porosity, less residual monomer, and reduced retention of Candida albicans are the result of high condensation of a material (54). Different manufacturers have produced CAD/CAM PMMA blocks (Figures 7 and 8) as a variant to conventional denture base resins. Afterwards, prefabricated or milled denture teeth are bonded to the denture base. Ivoclar Digital Denture (Ivoclar Vivadent, Liechtenstein) is one of the systems with the mentioned workflow (53). Lately, several systems have developed a method in which a denture base and denture teeth can be milled out of a single block. Ivoclar Vivadent Ivotion (Ivoclar Vivadent, Liechtenstein) works that way (53). Major shortcomings of subtractive (milling) method include a waste of the material since greater part of blocks remain unused, as well as monochromatic and non-aesthetic teeth (53).

Figure 7. Material for a milled denture base – IvoBase CAD

Figure 8. Material for a milled denture base – Interdent CC disc PMMA

Additive (3D-printing) methods include techniques that manufacture objects layer by layer (53). American Society for Testing and Materials (ASTM) defines it as “the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies” (55). Several categories have been named by now: stereolithography (SLA), material extrusion, material jetting, powder bed fusion, binder jetting, sheet lamination, and direct energy deposition (56). SLA was invented by Chuck W. Hull and is the oldest and most frequently used technique (among 3D-printing techniques) in dentistry (56, 57). SLA works in a following way: the building platform is placed in a fluid resin, which is polymerized by an ultraviolet (UV) laser. Cross-section of the planned structure is drawn by the laser to establish each layer. The building platform steps down when the layer is polymerized, which enables unpolymerized resin to cover the prior layer. This procedure is repeated for as long as the printed structure is built (56).

Apart from dental medicine, the aforementioned method can also be used in other fields, including engineering and medicine (53, 58). CAD/CAM systems have already started a new era in prosthodontics (59).

1.2.1 Mechanical properties

The first study of usage of CAD/CAM systems was published in 1994 (60), and additive technology was used for fabrication of these first CAD/CAM denture bases (45). Since CAD/CAM systems are highly sophisticated, it took approximately 20 years for their first commercial appearance (45). The main goal of new technologies and new materials is to overcome negative sides of a conventional manufacturing process, and to improve mechanical properties of materials used in the process (61).

Some drawbacks of conventional complete denture fabrication process are fractures, inappropriate aesthetics, and weak retention (61–64). Beside that, poor mechanical properties of a denture base resin could affect clinical performance of complete dentures (61, 65, 66). Multiple methods have been used to improve given properties, including microstructure modification with additives, advancements in manufacturing process, and adjustment of the powder-liquid ratio (61, 67-69).

CAD/CAM systems represent a new concept for designing and manufacturing complete dentures (9, 61, 70). The above-mentioned systems could change and shorten clinical protocols, archive digital information, and automatize a whole manufacturing process (45).

A milled manufacturing process requires polymerized PMMA blocks from which denture bases are milled (61). PMMA blocks are polymerized by injecting, under high pressure and temperature leading to a higher degree of monomer conversion and low values of residual monomer (70, 71). In other words, polymerization shrinkage is obstructed in milled denture bases (9, 45). The attendance of residual monomer in denture base resin is undesirable due to its effects on mechanical properties. However, the amount of residual monomer is inevitable because of the monomer-polymer balance, and concluding zero residual monomer cannot be accomplished (72). In vitro studies showed superior mechanical properties in milled dentures (8, 45). One of the shortcomings of the above-mentioned technology is inability to form complicated details (e. g. undercuts) (73). Since present findings related to mechanical properties of milled CAD/CAM denture base resins have determined some variations, (71, 74–76), further in vitro studies are necessary to affirm or oppose these findings.

An additive manufacturing process requires a light-polymerized resin for denture base fabrication (74). PMMA materials show high amount of shrinkage throughout the light polymerization process and poor mechanical properties (73, 77). Currently, additive manufacturing seems to be a promising technology which needs to be refined (78).

1.3 Shear bond strength between denture base and artificial teeth

Numerous studies have shown that dentures can be fabricated by using different materials and different processing techniques (79). Digital technologies enabled fabrication of denture base using milling or 3D printing. Afterwards, artificial teeth and CAD/CAM (milled) teeth can be bonded by using appropriate adhesive or by using a cold or heat polymerization process (80). Although they do not possess the best mechanical properties compared to other materials for denture base fabrication, acrylic resins remain the most popular choice, mainly because of its simple processing method and relatively low cost of manufacturing process (79, 81).

Denture base materials and artificial teeth have different structures and are manufactured apart. Due to separated manufacturing process, debonding of artificial tooth can occur, especially in the anterior area of a denture (80). As some studies have already reported, 30 % of all denture repairs are result of debonding of artificial teeth from a denture base (82–84). Debonding can be caused by the presence of wax on the ridge lap surface of an artificial tooth, a neglectful application of the separating agent, and a polymerization technique used for manufacturing denture base resins (85, 86).

A shear bond strength (SBS) testing (87) is usually used to investigate the bond strength between denture base resins and artificial teeth (85, 88–92). Universal testing machine (in the present study model LRX, Lloyd Instruments, Fareham, Great Britain) uses a 1 mm/min crosshead speed. The procedure is as follows: the final specimens should be put in a fixture, while crosshead implements a force parallel to the bonding area between an artificial tooth and denture base material (93). After the fracture, the load is registered and displayed by the software (in the present study Nexygen, Lloyd Instruments, Fareham, Great Britain). Figures 9–11 show phases of the specimen preparation.

Figure 9. An artificial tooth immersed in a colorless cold-polymerized acrylics

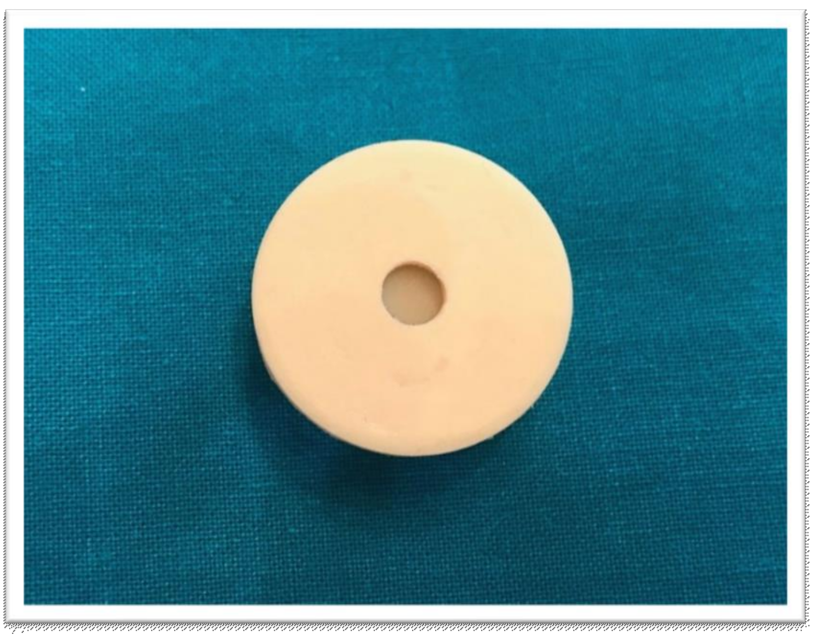

Figure 10. The same specimen from Figure 9 with a silicone sample measuring 25.0 mm diameter × 2.5 mm height, and an empty roller in the middle (5.0 mm in diameter) to obtain space for denture base resin cylinder

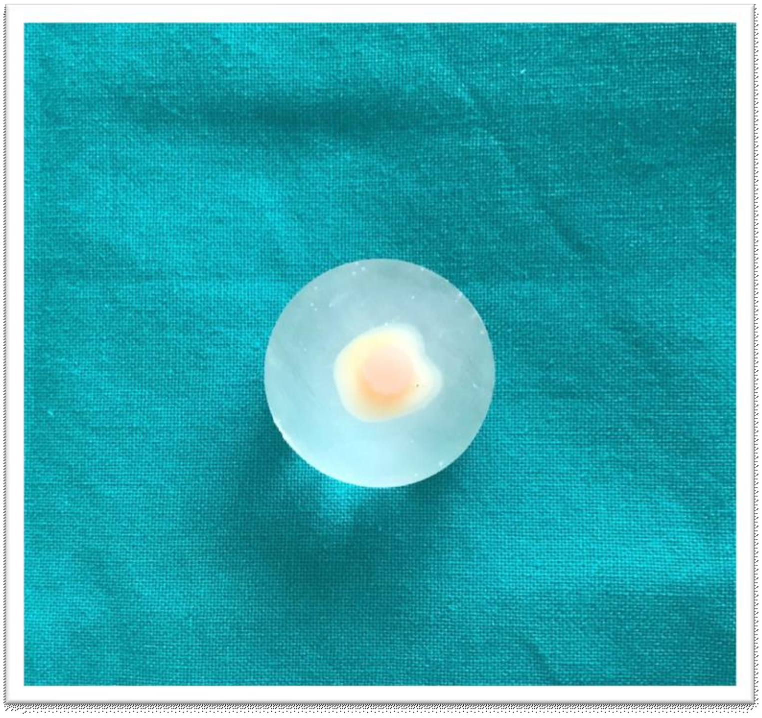

Figure 11. The final specimen looks like a denture tooth incorporated in an acrylic resin, and bonded to denture base resin cylinder with a 5.0 mm diameter × 2.5 mm height

1.3.1 Prefabricated teeth

Prefabricated teeth are an extremely significant part of conventionally fabricated complete dentures, and a final outcome of complete dentures depends on aesthetics and function of prefabricated teeth (94, 95).

Artificial teeth should imitate anatomic and aesthetic features of natural teeth. Moreover, prefabricated teeth should be non-reactive with oral tissues, non-toxic, and cost effective (95). One of significant mechanical properties is wear resistance. A deficiency of adequate wear resistance can result in an inordinate reduction in structure which can cause:

- lapse of vertical dimension of occlusion
- lapse of posterior tooth support
- lapse of masticatory efficacy
- fatigue of masticatory muscles
- changes in the functional path of masticatory movement
- inaccurate tooth relation
- lapse of esthetics (95).

When choosing a type of prefabricated denture teeth in complete dentures manufacturing process, physical properties of denture teeth play an important role (95). Presently, there are several types of prefabricated teeth attainable for usage:

- acrylic teeth
- cross-linked teeth
- nanohybrid composite teeth

Conventional acrylic denture teeth are particularly comprised of polymethylmethacrylate (PMMA). Its favorable properties include natural texture, high bond strength to denture base material, appropriate strength, high resiliency, and facility of occlusal adjustment (94). However, acrylic denture teeth show low endurance to abrasion, increased susceptibility to color change, biofilm formation which can cause occlusal disbalance, and aesthetic problems (96).

Microfilled composite denture teeth and highly cross-linked acrylic denture teeth have been developed to enhance mechanical properties of denture teeth (94). Crosslinking agents were added to PMMA material which leaded to a highly dense structure. Two types of denture teeth strengthened by a crosslinking agent are interpenetrating polymer network (IPN) and double crosslinked (DCL) denture teeth (97, 98).

Composite denture teeth are comprised of nanohybrid composite (NHC) matter on a urethane dimethacrylate (UDMA) matrix. UDMA matrix contains different types and sizes of fillers, and PMMA clusters. Some of the fillers include highly densified inorganic microfillers, highly cross-linked inorganic macrofillers, and silanized nanoscale fillers based on silicon dioxide (94). The present material shows better mechanical properties, whereas the fillers amplified rigidity and the hardness of the material (97). On the other hand, NHC denture teeth show low shear bond strength values when bonded to denture base resin. Brittleness, porosity, and staining are also some of the unwanted properties NHC denture teeth possess (97).

Artificial denture teeth were preliminary sectionalized into two layers, dentin and enamel layer (94, 95). Since some composite teeth can have intermediate layer(s), the above-mentioned two layers-classification did not fit well. Every layer of artificial teeth contains different properties like monomer diffusion, roughness, and hardness (94, 95). Frequently, the enamel layer is eliminated due to wear during the mastication process and occlusal adaptation (94).

1.3.2 CAD/CAM (milled) denture teeth

In recent years, technology enabled production of complete dentures in a digital way which include the use of subtractive or additive technologies (3D printing). Prefabricated, CAD/CAM (milled) or 3D printed denture teeth are attached to a denture base (99–101). Artificial teeth can be attached with an appropriate adhesive or by using a heat or cold polymerization (a conventional approach).

CAD/CAM (milled) denture teeth can be produced in two ways:

- in a single body (denture base resin and teeth in one piece)
- in separate pieces (one disc for denture base, the other for denture teeth) (72, 102, 103) (Figure 12).

Currently, the second method which enables production of denture base with sockets made for milled denture teeth from a prepolymerized block is more popular (47, 72, 104–107). Positive sides of the given method have been reported and include:

- a better fitting and retention due to the reduction of polymerization shrinkage compared to conventionally heat-polymerized dentures (105, 108)
- allowance of using various denture teeth with fine mechanical properties and aesthetic features (109-111).

Figure 12. CAD/CAM disc used for milling of a denture teeth

2. GENERAL AND SPECIFIC AIMS

The aim of the present study was to estimate and collate the mechanical properties (FS, SH, and IS) of various materials and technologies for denture base manufacturing, and to define shear bond strengths (SBS) of various denture base resins to various types of prefabricated teeth (acrylic, nanohybrid composite, and cross-linked) and denture teeth produced by CAD/CAM technology. The study emphasized digital technologies (milling and 3D printing) and materials which are intended to be used with these technologies.

The individual aims of the present study were to determine and compare mechanical properties as follows:

- FS, SH, and IS of PMMA heat-polymerized denture base resins
- FS, SH, and IS of CAD/CAM (milled) PMMA denture base resins
- FS, SH, and IS of 3D printed denture base resins
- FS, SH, and IS of polyamide denture base resin
- SBS of cold-polymerized denture base resin to acrylic teeth
- SBS of cold-polymerized denture base resin to nanohybrid composite teeth
- SBS of cold-polymerized denture base resin to cross-linked teeth
- SBS of heat-polymerized denture base resin to acrylic teeth
- SBS of heat-polymerized denture base resin to nanohybrid composite teeth
- SBS of heat-polymerized denture base resin to cross-linked teeth
- SBS of CAD/CAM (milled) denture base resin to acrylic teeth
- SBS of CAD/CAM (milled) denture base resin to nanohybrid composite teeth
- SBS of CAD/CAM (milled) denture base resin to cross-linked teeth
- SBS of CAD/CAM (milled) denture base resin to CAD/CAM (milled) denture teeth

3. A STUDY OF THE FLEXURAL STRENGTH AND SURFACE HARDNESS OF DIFFERENT MATERIALS AND TECHNOLOGIES FOR OCCLUSAL DEVICE FABRICATION

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3.1 Abstract

Statement of problem: With the emergence of digital technologies, new materials have become available for occlusal devices. However, data are scarce about these different materials and technologies and their mechanical properties.

Purpose: The purpose of this in vitro study was to investigate the flexural strength and surface hardness of different materials using different technologies for occlusal device fabrication, with an emphasis on the digital technologies of computer-aided design and computer-aided manufacturing (CAD-CAM) and 3D printing.

Material and methods: A total of 140 rectangular specimens were fabricated from two 3D-printed (VarseoWax Splint and Ortho Rigid), 2 CAD-CAM-produced (Ceramill Splintec and CopraDur), and 3 conventional autopolymerizing occlusal device materials (ProBase Cold, Resilit S, and Orthocryl) according to ISO 20795-1:2013. Flexural strength and surface hardness were determined for 10 specimens of each tested material using the 3-point bend test and the Brinell method. The data were analyzed using descriptive statistics and 1-way ANOVA with Bonferroni corrections (α=.05).

Results: Surface hardness values ranged from 28.5 ±2.5 MPa to 116.2 ±1.6 MPa. During flexural testing, neither the CopraDur nor the VarseoWax Splint specimens fractured during loading within the end limits of the penetrant's possible movement. Flexural strength values for other groups ranged from 75.0 ±12.0 MPa to 104.9 ±6.2 MPa. Statistical analysis determined significant differences among the tested materials for flexural strength and surface hardness.

Conclusions: Mechanical properties among different occlusal device materials were significantly different. Acrylic resins were less flexible than polyamide and nonacrylic occlusal device materials for 3D printing but had higher and more consistent values of surface hardness. Clinicians should

consider the different mechanical properties of the available materials when choosing occlusal device materials.

3.2 Introduction

Temporomandibular disorder (TMD) is a collective term that involves several clinical problems affecting the masticatory muscles, temporomandibular joints, and associated structures (1). Bruxism is defined as a diurnal or nocturnal parafunctional activity that includes unconsciously clenching, grinding, or bracing the teeth (2). The incidence of TMD is over 10% in the general population, (3) whereas some studies confirm an overall 8% incidence of bruxism, although this differs with age (4, 5). Occlusal devices are often used to manage TMD symptoms and prevent the negative effects of bruxism on the stomatognathic system.

Occlusal devices are usually made of poly(methyl methacrylate) (PMMA)-based polymers, whose mechanical properties and ease of use represent the gold standard for occlusal device material. Recently, although the most common technique of occlusal device fabrication remains vacuumthermoforming foil and autopolymerizing PMMA, (6) occlusal devices can be made by using computer-aided design and computer-aided manufacturing (CAD-CAM) or by additive manufacturing (6-8). Occlusal device production using the CAD-CAM technology has 3 main requirements: data acquired directly through intraoral scanners or indirectly through a dental stone cast, software to design a virtual restoration, and a computerized milling device (9). Stereolithography is the type of additive technology most frequently used for 3D printing occlusal devices. Stereolithography photopolymers are polymerized from liquid to solid under ultraviolet light. The lightpolymerizing resin is polymerized layer by layer until the final size of the object (occlusal device) according to the standard tessellation language file is achieved. New technologies imply the adaptation of existing materials or the development of new ones, depending on the technology and the purpose of the material (10). The materials used for occlusal device fabrication with CAD-CAM or 3D printing are acrylics, polyamides, or other resins. These materials must have appropriate biomechanical properties (11).

Unlike denture materials, which have been frequently investigated, (12-23) published data are scarce on the different materials and technologies used for occlusal device fabrication and their resulting mechanical properties. The purpose of this in vitro study was to investigate the flexural strength and surface hardness of different materials using different technologies for occlusal device fabrication, with an emphasis on the digital technologies of CAD-CAM and 3D printing. The null hypothesis was that different materials would have similar flexural strength and surface hardness.

3.3 Material and methods

Two types of CAD-CAM materials, 2 types of 3D printing materials, and 3 conventional materials (autopolymerizing acrylic resins) for occlusal device fabrication were selected. A list of the materials, manufacturers, types, and occlusal device fabrication techniques is shown in Table 1. Most of the materials used were acrylic resins (ProBase Cold [PRC], Orthocryl [ORT], Resilit S [RES], Ceramill Splintec [CSP], and Ortho Rigid [ORR]); 1 was a polyamide resin (CopraDur [COP]), and VarseoWax Splint (VWS) was a poly(oxy-1,2-ethandiyl), alpha, alpha'-[(1-methylethyliden)di-4 1-phenylene]bis [omega-[(2-methyl-1-oxo-2-propenyl)oxy]-based material. Power analysis to estimate the appropriate sample size was based on the results of the study by Ayaz et al, (15) who found mean flexural strengths of 89.1 ±7.5 MPa and 69.6 ±4.1 MPa for PMMA and polyamide denture base materials, respectively. The effect size was hypothesized to be 1.4. Accordingly, with a=.05 and b=.95, the projected sample size needed was n=10 (GPower 3.1), as in similar studies (6, 20, 22, 23).

Table 1. Materials, types, manufacturers, and occlusal device fabrication techniques

Material name	Abbreviation	Type	Manufacturer	Occlusal device fabrication technique
ProBase Cold	PRC	PMMA	Ivoclar Vivadent AG	Conventional; autopolymerizing
Orthocryl	ORT	PMMA	Dentaurum KG	Conventional; autopolymerizing
Resilit S	RES	PMMA	Erkodent Erich Kopp	Conventional; autopolymerizing
Ceramill Splintec	CSP	PMMA	Amann Girbach AG	CAD-CAM
Copradur	COP	Crosslinked polyamide	Whitepeaks Dental Solutions KG	CAD-CAM
VarseoWax Splint	VWS	Non-acrylic light-polymerizing resin	Bego KG	3D printing
Ortho Rigid	ORR	Acrylic light-polymerizing resin	Next Dent B.V.	3D printing

CAD-CAM, computer-aided design and computer-aided manufacturing; PMMA, poly(methyl methacrylate).

A total of 140 rectangular specimens (64.0×10.0×3.3 ±0.2 mm) were fabricated for flexural strength and surface hardness testing. All the testing procedures were performed according to ISO standards (ISO 20795-1:2013 [24]; ISO 2039-1:2001 [25]). A specimen plate (block) each of ORT, RES, and PRC (conventional autopolymerizing resins) was prepared from a silicone mold (Elite HD; Zhermack SpA) according to the manufacturer's instructions. After the monomer and polymer were mixed in an appropriate ratio and the silicone mold was filled, the mold was placed into a pressure-polymerizing unit (Ivomat IP3; Ivoclar Vivadent AG) at 0.22 MPa for 15 minutes. A VWS and an ORR specimen plate were prepared according to the standard tessellation language (STL) file in an appropriate light-polymerizing unit (VWS: Varseo 3DPrinter [Bego KG]; ORR: DentalFab [Microlay]) following postpolymerization (VWS: Bego Otoflash [Bego KG]; ORR: LC-3DPrint Box [NextDent B.V.]). Polymerization and postpolymerization were conducted according to the manufacturer's instructions.

Slightly oversized specimens were cut of conventional (ORT, RES, and PRC) and 3D-printed specimen plates (VWS and ORR) and CAD-CAM blocks (CSP, and COP) using a water-cooled milling machine (IsoMet 1000; Buehler). All sides of the specimens were wet-ground using standard metallographic grinding papers (P500, P1000, and P1200) to the required width, height, and surface smoothness. Flexural strength was measured according to ISO 20795-1:2013 (24) in a 3-point bend test using a universal testing machine (VEB Werkstoffprüfmaschinen).

Ten specimens of each material were tested. Specimens were immersed in a water bath for 50 ±1 hours at 37°C before testing. After random removal from the water (randomization chart was made using Excel; Microsoft), each specimen was immediately laid symmetrically with the flat surface on the supports of the flexural test rig. The force on the loading plunger was increased uniformly from zero by using a constant displacement rate of 5 ±1 mm/min until the specimen fractured.

The flexural strength of each specimen was calculated in megapascals (MPa) according to the following formula: $FS = 3FL/2bh^2$, where FS is flexural strength, F is the maximum load exerted on the specimen in newtons (N), L is the distance between the supports (mm), b is the width of the specimen (mm), and h is the height of the specimen (mm).

Ten specimens of each material were measured for surface hardness (Brinell method, ISO 2039-1:200125). Brinell hardness was calculated from the equation: $HB = F/\pi Dh_k$, where HB is Brinell hardness (MPa), F is load applied to the specimen (N), D is diameter of the indenter (mm), and hk is penetration depth (mm). Brinell hardness was determined for each specimen using a Zwick apparatus (Zwick Roell Group). A 358 N load was applied through the indenter with a dwell time of 60 seconds (for 1 tested material, VWS, a 132 N load was used because of that material's lower hardness). Brinell hardness was measured at 5 locations on each specimen, and mean hardness was determined for each specimen.

Descriptive statistics were calculated for all study groups. One-way ANOVA and Bonferroni post hoc tests were performed using a statistical software program (SPSS Statistics 17.0; SPSS Inc) to compare the tested groups of materials (a = .05).

3.4. Results

Descriptive statistics for the obtained values of surface hardness and flexural strength are presented in Figures 1 and 2. All specimens in the COP and VWS groups were loaded to the end limits of the possible movement of the penetrant. Because neither specimen fractured, flexural strength for the COP and VWS groups could not be measured. To include these 2 groups in statistical analyses, an arbitrarily high value was assigned (maximum flexural strength value obtained in other groups plus 1).

One-way ANOVA showed statistically significant differences among the tested groups for surface hardness ($F = 879.9$, $P < .05$) and flexural strength ($F = 36.2$, $P < .05$). Results of the Bonferroni post hoc test for flexural strength and surface hardness are presented in Figures 1 and 2. One-way ANOVA showed statistically significant differences among different fabrication techniques in flexural strength ($F = 5.1$, $P < .05$) and surface hardness values ($F = 17.7$, $P < .05$). The Bonferroni post hoc test determined higher flexural strength values in the CAD-CAM materials than in the autopolymerized materials ($P = .042$) and 3D-printed materials ($P = .011$), and lower surface hardness values were found for the 3D printing materials than for the autopolymerizing ($P < .001$) and CAD-CAM materials ($P = .004$).

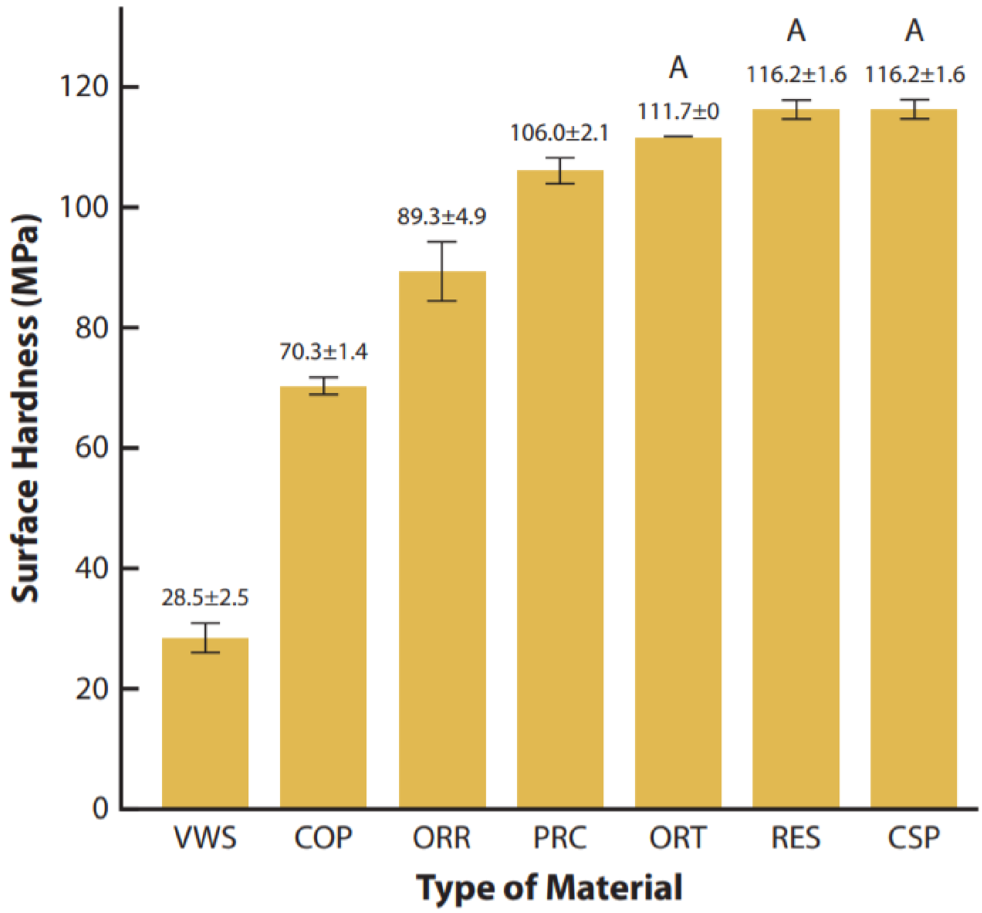

Figure 1. Means and standard deviations of surface hardness for groups. Similar uppercase letters denote no significant differences among groups (Bonferroni post hoc test, $P > .05$). COP, CopraDur; CSP, Ceramill Splintec; ORR, Ortho Rigid; ORT, Orthocryl; PRC, ProBase Cold; RES, Resilit S; VWS, VarseoWax Splint.

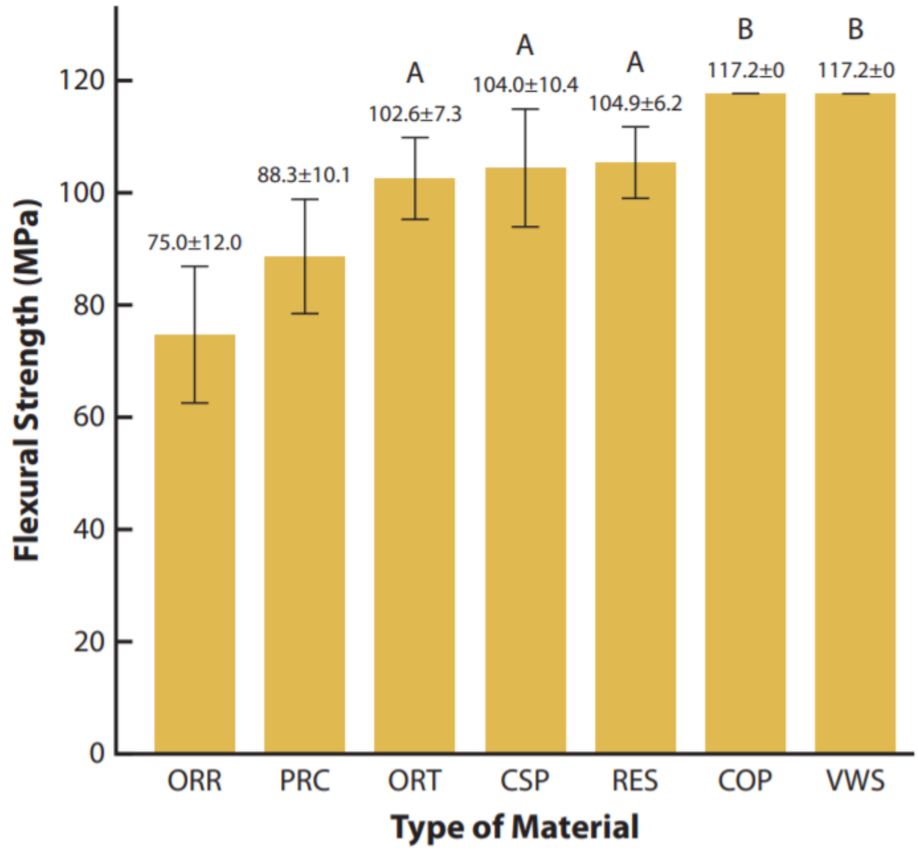

Figure 2. Means and standard deviations of flexural strength for groups. Similar uppercase letters denote no significant differences among groups (Bonferroni post hoc test, $P > .05$). For groups COP and VWS, neither specimen fractured during loading within end limits of possible movement of penetrant. For inclusion of these 2 groups in statistical analysis, an arbitrarily high number was assigned (the maximum flexural strength value obtained in other groups + 1). COP, CopraDur; CSP, Ceramill Splintec; ORR, Ortho Rigid; ORT, Orthocryl; PRC, ProBase Cold; RES, Resilit S; VWS, VarseoWax Splint.

3.5 Discussion

The flexural strength and surface hardness of different occlusal device materials produced with different techniques, especially digital technologies, were investigated. Because statistical analysis demonstrated betweenmaterial differences in flexural strength and surface hardness, the null hypothesis was rejected.

Surface hardness describes the density of a material and its resistance to wear and/or scratching, and it affects prosthetic restorations during function and cleaning (12). Because occlusal loads in functional or parafunctional activity, especially bruxism, can be higher than 785 N, (26) the wear resistance of occlusal device materials is important. In the present study, PMMA (3 conventional autopolymerizing materials and 1 CAD-CAM occlusal device material) had the most consistent results for surface hardness, followed by the acrylic resin for additive manufacturing (Fig. 1). Despite the difference between the PMMA and the acrylic resin for additive manufacturing, the polyamide material (CAD-CAM) and the nonacrylic light-polymerizing resin for additive manufacturing showed the lowest surface hardness values.

Nguyen et al (13) tested 2 polyamide materials that were used as denture bases and reported statistically significantly lower values of surface hardness than PMMA. Hamanaka et al (14) and Ayaz et al (15) reported similar results. Although other authors (13-15) investigated denture base materials and not occlusal device materials, their results were comparable with those of the present study (Fig. 1). It is safe to conclude that polyamide materials used for occlusal devices have lower surface hardness than PMMA.

To the authors' knowledge, the only comparable study of a light-polymerizing resin for additive manufacturing of occlusal devices was carried out by Huettig et al (6). The authors examined the polishability and wear resistance of different occlusal device materials for oral appliances. A nonacrylic light-polymerizing resin for additive manufacturing showed lower wear values than a conventional acrylic resin, which seems contrary to the results of the present study (Fig. 1). Opposite results can most easily be explained by the different properties investigated (wear resistance compared with surface hardness). Although surface hardness describes resistance to wear (12) and several studies have found a correlation between hardness and wear resistance, (16-18) some authors disagree with this correlation (14). For a more thorough comparison of surface hardness and intraoral wear resistance of different digitally processed occlusal device materials, further investigations are necessary, especially clinical studies.

In the study by Ayman, (19) conventional heatpolymerized PMMA had lower surface hardness values than CAD-CAM PMMA. In the present study, PMMA processed conventionally and with CAD-CAM had similar surface hardness values, whereas 2 lightpolymerized resins (acrylic and nonacrylic) processed with additive manufacturing and 2 resins (polyamide and PMMA) processed with CAD-CAM differed significantly (Fig. 1). Between-material differences were mainly due to their chemical compositions rather than the technology, but differences due to different technologies are not excluded (19).

As in the study by Ucar et al, (20) neither a specimen of a polyamide-based material nor one of a nonacrylic lightpolymerizing resin for additive manufacturing fractured during flexural testing. Several studies have investigated polyamide materials for removable partial and complete denture bases, (13-15, 20-23) although not for occlusal device fabrication. Three of these studies established that polyamide materials are more flexible than acrylic resins, (21-23) similar to the results of the present study (Fig. 2). Although flexibility is important for absorbing energy when a patient drops an appliance, (20) nonflexible occlusal devices are considered a better option for a patient with bruxism (27-29). The clinical implications of the flexibility of occlusal device materials produced with digital technologies are not yet clear. For a better comparison with acrylic resin occlusal devices, clinical studies with newly developed materials having different mechanical properties and influence on the stomatognathic system are necessary.

Unlike the polyamide material and the nonacrylic light-polymerizing resin for additive manufacturing, all specimens of the different acrylic resin materials fractured. Two conventional occlusal device materials and one CAD-CAM PMMA material showed similar values for flexural strength (Fig. 2), contrary to the results of Alp et al.30 Despite the statistically significant differences found among the tested materials (Fig. 2), all the tested materials met the ISO requirements for flexural strength 65 MPa (24).

3.6 Conclusions

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The mechanical properties of occlusal devices depend more on the material than on the particular technology.

2. Acrylic resin has the most consistent values of surface hardness regardless of the technology.

3. Polyamide resins and nonacrylic light-polymerizing resins for additive manufacturing have lower surface hardness, but their flexural strength is higher than that of acrylic resin.

4. No specimen of either polyamide or nonacrylic light-polymerizing resin for additive manufacturing fractured during flexural strength measurement.

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4. COMPARISON OF MECHANICAL PROPERTIES OF 3D-PRINTED, CAD/CAM, AND CONVENTIONAL DENTURE BASE MATERIALS

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4.1 Abstract

Purpose: To evaluate and compare the mechanical properties (flexural strength and surface hardness) of different materials and technologies for denture base fabrication. The study emphasized the digital technologies of computer-aided design/computeraided manufacturing (CAD/CAM) and three-dimensional (3D) printing.

Materials and Methods: A total of 160 rectangular specimens were fabricated from three conventional heat-polymerized (ProBase Hot, Paladon 65, and Interacryl Hot), three CAD/CAM produced (IvoBase CAD, Interdent CC disc PMMA, and Polident CAD/CAM disc), one 3D-printed (NextDent Base), and one polyamide material (Vertex ThermoSens) for denture base fabrication. The flexural strength test was the three-point flexure test, while hardness testing was conducted using the Brinell method. The data were analyzed using descriptive and analytical statistics ($\alpha = 0.05$).

Results: During flexural testing, the IvoBase CAD and Vertex ThermoSens specimens did not fracture during loading. The flexural strength values of the other groups ranged from 71.7 ± 7.4 MPa to 111.9 ± 4.3 MPa. The surface hardness values ranged from 67.13 ± 10.64 MPa to 145.66 ± 2.22 MPa. There were significant differences between the tested materials for both flexural strength and surface hardness. There were also differences between some materials with the same polymerization type. CAD/CAM and polyamide materials had the highest flexural strength values. Two groups of CAD/CAM materials had the highest surface hardness values, while a third, along with the polyamide material, had the lowest. The 3D-printed materials had the lowest flexural strength values.

Conclusions: Generally, CAD/CAM materials show better mechanical properties than heat-polymerized and 3D-printed acrylics do. Nevertheless, a material's polymerization type is no guarantee of its optimal mechanical properties.

4.2 Introduction

Complete dentures have been used for many years, and they are the gold standard for treating edentulism (1). Recent improvements in science and technology have provided digital methods for denture base production, including computer-aided design/computer-aided manufacturing (CAD/CAM) and threedimensional (3D) printing (2-5). Digital methods allow the production of a denture base in one block and provide the ability to attach prefabricated teeth with an appropriate adhesive. The advantages of digital methods are faster denture fabrication and fewer phases in the work process, (6) which can reduce the possibility of mistakes. With the further development of digital technology, there are now new 3D-printed materials from various dental manufacturers and more CAD/CAM materials for denture base fabrication. While the mechanical properties of conventionally polymerized denture base acrylics (7-11) and polyamide materials have been investigated and reported (12-16)— along with new data on mechanical properties of CAD/CAM dentures, even if scarce (17-19)—to our knowledge, no studies have been published on the mechanical properties of denture bases produced from 3D-printed materials.

Surface hardness testing and the three-point flexure test are regularly used for analyzing the mechanical properties of denture base materials (2, 9, 15). The aim of this study was to employ such testing for examining the mechanical properties (flexural strength and surface hardness) of different materials for denture base fabrication, with an emphasis on digital technologies (CAD/CAM and 3D printing), and compare them with heat-polymerized acrylics and thermoplastic material for the production of complete denture bases. The null hypothesis was that different materials would have similar flexural strength and surface hardness values.

4.3 Materials and methods

The following materials were selected for denture base fabrication: three heat-polymerized acrylics, three types of CAD/CAM materials, one type of resin for 3D printing, and one polyamide material. A list of the materials, manufacturers, types, and denture base fabrication techniques is given in Table 1.

Table 1 Materials, types, manufacturers and denture base fabrication techniques

Material	Abbreviation	Type	Manufacturer	Denture base fabrication technique
ProBase Hot	PBH	PMMA	Ivoclar Vivadent AG	Conventional; heat-polymerized
Paladon 65	PAL	PMMA	Kulzer GmbH	Conventional; heat-polymerized
Interacryl Hot	IAH	PMMA	Interdent d.o.o.	Conventional; heat-polymerized
IvoBase CAD	IBC	PMMA	Ivoclar Vivadent AG	CAD/CAM
Interdent CC disc PMMA	IDP	PMMA	Interdent d.o.o.	CAD/CAM
Polident CAD/CAM disc basic	PDD	PMMA	Polident d.o.o.	CAD/CAM
NextDent Base	NDB	Monomer based on acrylic esters	Nextdent B.V.	3D printing
Vertex ThersmoSens	VTS	Polyamide	Vertex-Dental B.V.	Injection pressing

A total of 160 specimens were tested for flexural strength and surface hardness. The heat-polymerized acrylic blocks were prepared using the compression molding technique. A rectangular template made of wax was invested with gypsum. A first layer of gypsum was poured in the lower half of the flask, and a wax template was placed inside. After induration, the first gypsum layer was coated with separating medium (Separating Fluid, Ivoclar Vivadent, Schaan, Liechtenstein). The second layer of gypsum was poured, and the flask was completely closed. After gypsum setting, the flask was opened, the wax was completely removed, and the mold was coated with separating medium. The packing stage involved placement and adaptation of denture base resin within the mold cavity. Next, the flasks were placed in a hydraulic press for 5 minutes under a 1250 kgf load and put in the appropriate polymerization unit (EWL Typ 5509, Kavo, Biberach, Germany) with the flask carrier to maintain pressure. All three heat-polymerized acrylics were prepared according to the manufacturers' instructions (ratio of polymer and monomer and polymerization method). After polymerization, the flasks were left to cool at room temperature. Finally, the rectangular acrylic blocks were carefully deflasked.

Rectangular polyamide blocks were prepared in a similar way (injection pressing) following the manufacturer's instructions.

A rectangular block was designed (Netfabb Premium 2019, Autodesk, San Rafael, CA) and saved as a standard tessellation language (STL) file; the 3D-printed samples were prepared according to the obtained STL file. Using the STL file, the 3D printing was conducted using an appropriate 3D unit (DentalFab, Microlay Dental 3D Printers, Madrid, Spain), with subsequent light polymerization done in a suitable device (LC-3DPrint Box, NextDent, Soesterberg, the Netherlands) following the manufacturers' instructions.

CAD/CAM specimens were prepared from CAD/CAM discs. First, the CAD/CAM discs were cut with a diamond disc to obtain rectangular blocks. After rectangular heatpolymerized, polyamide, 3D-printed, and CAD/CAM blocks were prepared, specimens to be used for flexural strength and surface hardness testing were cut from the blocks on a water-cutting machine (IsoMet 1000, Buehler, Lake Bluff, IL). All surfaces were ground using standard metallic grinding paper (P500, P1000, and P1200) to smooth surfaces with the default dimensions.

The flexural strength was tested using a three-point flexure test on a universal testing machine (10 specimens for each tested material, 64 × 10 × 3.3 ± 0.2 mm, ISO 20795- 1:2013 [20]). Before testing, the specimens were immersed in a water bath for 50 ±1 hour at 37°C. Immediately following this, the specimens were removed from the water and placed symmetrically on the base of a universal testing machine (VEB Werkstoffprüfmaschinen, Leipzig, Germany). The load force was increased evenly from 0, using a steady shift of 5 ± 1 mm/min until the specimen cracked. The flexural strength of each specimen was measured according to the following formula: $FS = 3FL/2bh^2$, where FS is flexural strength (MPa), F is the maximum force applied to a specimen (N), L is the distance between the specimen carrier (mm), b is the specimen width (mm), and h is the specimen height (mm). Ten specimens of each material with the dimensions 64.0 × 10.0 × 3.3 ± 0.2 mm were used for surface hardness testing (Brinell's method, ISO 2039-1:2001 [21]).

The surface hardness was determined using Brinell's method according to the following formula: $HB = F/\pi Dh_k$, where HB is the Brinell hardness (MPa), F is the force applied to the specimen (N), D is the ball diameter (mm), and h is the depth of penetration (mm). The 358 N force was applied via a ball for 60 seconds (for one material, VTS, a 196 N load was used because of the material's lower hardness). Brinell's hardness was measured at five points on each specimen, after which, the average hardness for each sample was determined. Testing was carried out on a Zwick apparatus (Zwick Härteprüfgerät Modell 3106 No. 29542/1965, Zwick Roell Group, Kennesaw, GA).

Descriptive and analytical methods were used. The normality of distribution was tested with the Shapiro-Wilk test. Analysis of variance test and Tukey's multiple comparison test were used for comparing the obtained values between different types of material (normal distribution). If the data tested were not normally distributed, the median test was used and a post hoc multiple comparison was made using Holm-Bonferroni correction. The analysis was made using a statistical software package (SAS 8.2, SAS, Cary, NC) on the Windows platform. The significance level was set at $p < 0.05$.

4.4 Results

Descriptive statistics for the flexural strength and surface hardness values are presented in Figures 1 and 2. During flexure testing, no specimens in the IBC and VTS groups fractured during loading within the end limits of the penetrant's possible movement; thus, the values of flexural strength for these two groups could not be measured. The maximal and minimal flexural strength values (MPa) of the rest of the tested groups were 119.1 and 103.7 for IDP, 116.4 and 97.0 for PDD, 110.6 and 84.1 for IAH, 100.7 and 71.8 for PBH, 88.5 and 62.2 for PAL, and 84.5 and 60.0 for NDB. The maximal and minimal surface hardness values (MPa) of the tested groups were 147.04 and 142.44 for IDP, 147.04 and 142.44 for PDD, 138.13 and 130.23 for PBH, 134.06 and 126.62 for IAH, 123.19 and 113.95 for PAL,123.19 and 106.0 for NDB, 103.60 and 69.06 for IBC, and 80.50 and 51.99 for VTS. For the inclusion of IBC and VTS in statistical analyses, an arbitrarily high value was assigned (the maximum flexural strength value determined in other groups plus 1). The median test determined the statistically significant ($p < 0.001$) differences in flexural strength values between the tested groups. Figure 1 shows the results of the Holm-Bonferroni post hoc analysis. The median test determined the statistically significant between-group differences in surface hardness values ($p < 0.001$). Figure 2 shows the differences in surface hardness between the tested groups (Holm-Bonferroni correction).

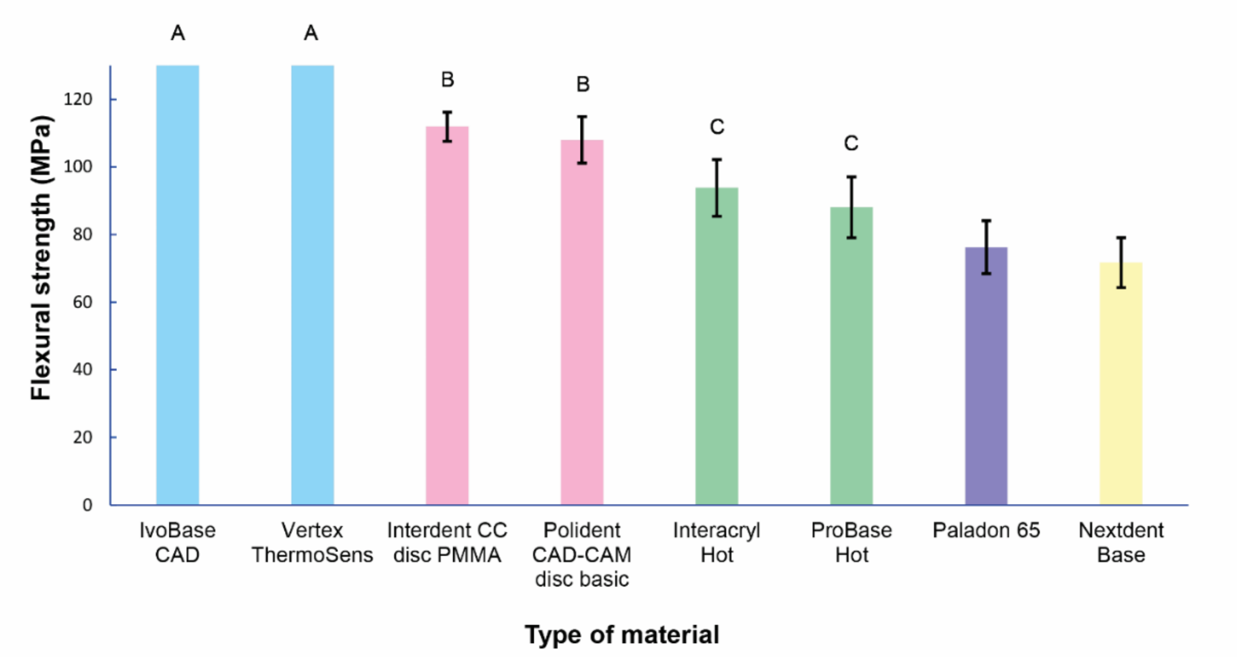

Figure 1 Means and standard deviations of flexural strength for the groups. Matching uppercase letters denote no significant differences between groups (Holm–Bonferroni post hoc test, $p < 0.05$). For better visualization of results, groups without significant differences are marked with the same color. For the IBC and VTS groups, neither specimen fractured during loading within the end limits of possible movement of the penetrant. For inclusion of these two groups in statistical analysis, an arbitrarily high number was assigned (maximum flexural strength value obtained in other groups plus 1).

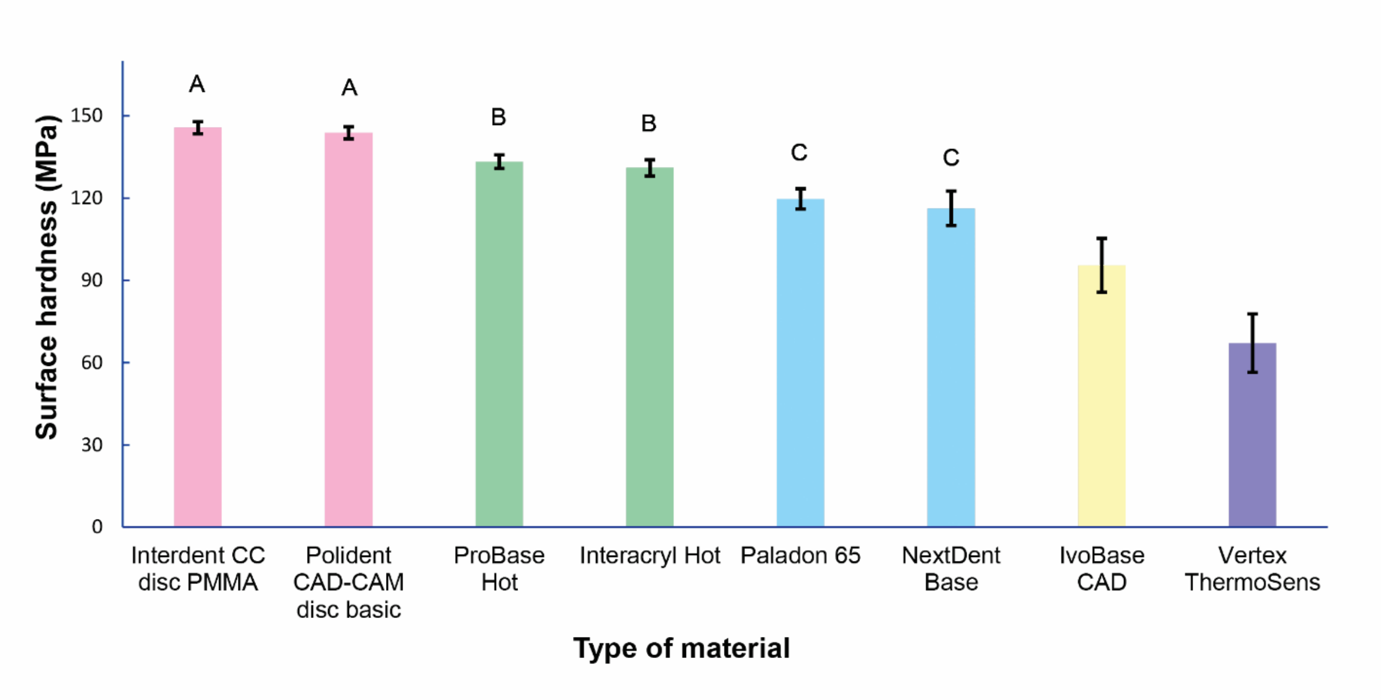

Figure 2 Means and standard deviations of surface hardness for the groups. Matching uppercase letters denote no significant differences between groups (Holm–Bonferroni post hoc test, $p < 0.05$). For better visualization of results, groups without significant differences are marked with the same color.

4.5 Discussion

This in vitro study investigated the mechanical properties of denture base materials made with different technologies, with an emphasis on digital technologies (CAD/CAM and 3D printing). The null hypotheses were rejected because statistical analysis showed differences between study groups for both flexural strength and surface hardness.

Flexural strength, also known as modulus of rupture, bend strength, or transverse rupture strength, is a material property defined as the stress in a material just before it yields in a flexure test. Since a denture base may fracture in real life for various reasons, it is important that its material has high flexural strength. Findings related to the flexural strength of CAD/CAM materials for denture base vary (5, 17-19). A study by Steinmassl et al (5) obtained mixed results where different CAD/CAM denture base resins showed similar, lower, or higher flexural strength values than the control heat-polymerized group did. Ayman (18) and Pacquet et al (17) determined higher values of flexural strength in heat-polymerized PMMA than in CAD/CAM denture base material. In contrast to the studies by Steinmassl et al, (8) Ayman, (18) and Pacquet et al, (17) the present study results (Fig 1) agree with those of Aguirre et al, (19) where CAD/CAM materials showed higher flexural strength values than compressionmolded denture base materials did. Since CAD/CAM PMMA blocks are made under high heat and pressure conditions with condensed acrylic resin and minimal shrinkage, porosity, or free monomers, (6) the higher flexural strength values of CAD/CAM materials, confirmed by both the present study results (Fig 1) and Aguirre et al, (19) are expected. Still, it should be noted that differences among the flexural strength values of CAD/CAM and heat-polymerized denture base materials (5, 17-19) (Fig 1) may be due to the use of different materials (manufacturers) in different studies. The only material that had higher flexural strength values than two of the CAD/CAM materials (IDP and PDD) in the present study was polyamide (Fig 1). Previous studies confirmed that polyamide materials for denture bases have higher flexural strength than heat-polymerized PMMA does, (12-14) which agrees with the present study results (Fig 1). The 3D-printed material (NDB) had the lowest flexural strength compared with the other study groups (Fig 1). Although the 3D-printed material (monomer based on acrylic esters) had the lowest values (Fig 1), it met ISO requirements for flexural strength (65 MPa) (20). It is safe to conclude that 3D-printed materials for denture bases are a new option for denture production, but for now, they have lower flexural strength values than most other denture base materials.

Hardness is a measure of the resistance to localized plastic deformation induced by either mechanical indentation or abrasion. Dentures made of a material with low surface hardness can be damaged by mechanical brushing, causing plaque retention and pigmentations, which can decrease the life of dentures. In the present study, two groups of CAD/CAM materials (IDP and PDD, Fig 2) were determined to have the highest surface hardness among the study groups. This finding is comparable to that of Ayman, (18) who reported higher hardness values for CAD/CAM materials than for heat-polymerized ones (Fig 2). However, the third group of tested CAD/CAM materials (IBC) in the present study (Fig 2) had lower hardness values than the other CAD/CAM materials, heat-polymerized PMMA, and 3D-printed materials. Given that significant between-group differences were observed for groups using the identical polymerization process as that reported in similar studies, (5) it can be concluded that denture base materials cannot be studied solely via the polymerization processing; in other words, the differences in results cannot be attributed only to different polymerization technologies. Except for one CAD/CAM group (IBC), 3D-printed material had lower surface hardness values than the other tested acrylics did (Fig 2). As in other studies, (7, 15) polyamide was found to be the material with the lowest hardness (Fig 2). Comparing flexural strength and surface hardness between the investigated groups (Figs 1 and 2), it can be concluded that the mechanical properties of most CAD/CAM and heat-polymerized acrylics are superior to those of 3D-printed materials. With no studies of the mechanical properties of 3D-printed denture base material for comparison, further research is necessary to confirm or dispute these findings.

The obtained surface hardness and flexural strength results can be explained according to the materials' inner structures. Polyamide material has a lower amount of cross-linking agents, which can influence the surface hardness (22). Acrylic resins for 3D printing of removable dentures have relatively low double-bond conversion compared with traditional acrylic resins, which can also affect mechanical properties (23). In contrast, with CAD/CAM resin, the high pressure influences the formation of longer polymer chains and can lead to a higher degree of monomer conversion (24, 25). In addition, inorganic fillers and high temperature during the polymerization process of the CAD/CAM resins also improve some mechanical properties, including flexural strength and surface hardness (18, 26). The differences in mechanical properties' values between CAD/CAM brands (Fig 2) can be explained in terms of the different density of each material (27).

According to the results of the present study, clinicians should consider that, with the emergence of digitally produced dentures, new denture base materials with different mechanical properties are available. Although it seems logical to compare materials based on manufacture type (e.g., 3D printing, CAD/CAM, or heat polymerization), the mechanical properties of the selected denture base material depend solely on the material itself, and not how it was made. However, 3D-printed materials for denture base fabrication do have lower mechanical property values than most CAD/CAM and heat-polymerized acrylics do.

The study had two major limitations. First, oral conditions were absent in the present research, and second, different testing conditions (dry vs. wet) and different testing media (air or water) were not included. Both may have affected the results (16). To obtain more comprehensive knowledge on new denture base materials, future studies considering flexural modulus, bonding to synthetic polymer teeth, and residual monomer testing are necessary.

4.6 Conclusions

Based on the findings of this in vitro study, polyamide and CAD/CAM materials exhibited higher flexural strength than heat-polymerized and 3D-printed acrylics. Materials with the same polymerization type can have different mechanical properties and 3D-printed acrylics have lower mechanical properties than most other denture base materials.

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5. COMPARISON OF SHEAR BOND STRENGTHS OF DIFFERENT TYPES OF DENTURE TEETH TO DIFFERENT DENTURE BASE RESINS

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5.1 Abstract

Purpose: To determine the shear bond strengths of different denture base resins to different types of prefabricated teeth (acrylic, nanohybrid composite, and cross-linked) and denture teeth produced by computer-aided design/computer-aided manufacturing (CAD/CAM) technology.

Materials and methods: Prefabricated teeth and CAD/CAM (milled) denture teeth were divided into 10 groups and bonded to different denture base materials. Groups 1–3 comprised of different types of prefabricated teeth and cold-polymerized denture base resin; groups 4–6 comprised of different types of prefabricated teeth and heat-polymerized denture base resin; groups 7–9 comprised of different types of prefabricated teeth and CAD/CAM (milled) denture base resin; and group 10 comprised of milled denture teeth produced by CAD/CAM technology and CAD/CAM (milled) denture base resin. A universal testing machine was used to evaluate the shear bond strength for all specimens. One-way ANOVA and Tukey post-hoc test were used for analyzing the data ($\alpha = .05$).

Results: The shear bond strengths of different groups ranged from 3.37 ± 2.14 MPa to 18.10 ± 2.68 MPa. Statistical analysis showed significant differences among the tested groups ($P < .0001$). Among different polymerization methods, the lowest values were determined in cold-polymerized resin.There was no significant difference between the shear bond strength values of heat-polymerized and CAD/CAM (milled) denture base resins.

Conclusion: Different combinations of materials for removable denture base and denture teeth can affect their bond strength. Cold-polymerized resin should be avoided for attaching prefabricated teeth to a denture base. CAD/CAM (milled) and heat-polymerized denture base resins bonded to different types of prefabricated teeth show similar shear bond strength values.

5.2 Introduction

Debonding of a tooth from a denture base of a complete or partial removable denture is the most common clinical situation requiring subsequent repair (1). According to studies, 30% of all denture repairs are caused by debonding of prefabricated teeth (2-4). The problem is even greater with implantsupported overdentures because their higher chewing capacity increases the risk of tooth detachment from the overdenture base (5).

Denture base resin and prefabricated teeth differ in a structure and are fabricated separately. Separated fabrication processes are considered to be one of the main factors that can lead to tooth failure, especially in the anterior portion of a removable denture (6, 7). The denture base resin that has been used most frequently in dental medicine is polymethylmethacrylate (PMMA) (7). Findings related to the bond strength between denture base materials and artificial teeth vary (8-16). As some studies have already reported, different types of prefabricated teeth also showed differences in the shear bond strength (4, 17).

Advancements in technology have ensured digital methods for denture base fabrication (computer-aided design/computer-aided manufacturing [CAD/CAM]), including subtractive (milling) and additive technologies (three-dimensional [3D] printing) (18-21). Digital techniques allow fabrication of a denture base in one block, and they have the ability to attach prefabricated teeth or CAD/CAM (milled) teeth with an adequate adhesive or to bond them using cold or heat polymerization (conventional approach) (19). Most manufacturers suggest use of adhesive as a better option (22, 23). The literature findings about the bond strength between digitally produced denture base resins and different types of artificial teeth (including teeth produced with CAD/CAM technology) are scarce. To the authors' knowledge, there are only two studies which have included digitally produced denture bases and different types of denture teeth (9, 24). However, these studies did not include CAD/CAM (milled) denture teeth.

Shear bond strength testing is the most widely used type of testing (25) for analyzing the bond strength between denture base resins and artificial teeth (13-17, 26). The aim of this study was to examine the shear bond strength of different types of prefabricated teeth (acrylic, nanohybrid composite, and cross-linked teeth) and CAD/CAM (milled) denture teeth to CAD/CAM (milled), cold-polymerized, and heat-polymerized denture base resins. The null hypothesis was that different materials would have similar shear bond strength values.

5.3 Materials and methods

Eighty specimens were prepared from three different types of prefabricated teeth (acrylic [SR Orthotyp S PE, Ivoclar Vivadent, Schaan, Liechtenstein], nanohybrid composite [Phonares II Typ, Ivoclar Vivadent, Schaan, Liechtenstein], cross-linked [SR Orthotyp DCL, Ivoclar Vivadent, Schaan, Liechtenstein]), and one type of milled CAD/CAM denture teeth (SR Vivodent CAD, Ivoclar Vivadent, Schaan, Liechtenstein). They were combined with three types of denture base resins-cold-polymerized acrylics (ProBase Cold, Ivoclar Vivadent, Schaan, Liechtenstein), heat-polymerized acrylics (ProBase Hot, Ivoclar Vivadent, Schaan, Liechtenstein), and CAD/CAM (milled) denture base resin (IvoBase CAD, Ivoclar Vivadent, Schaan, Liechtenstein). A list of groups, name of materials, and the manufacturer is shown in Table 1. The specimens were divided into 10 groups, and each group had eight specimens. Fig. 1 and Fig. 2 show diagrams of specimens.

Table 1 Groups, name of materials and manufacturer

Group	Name of materials	Manufacturer
Cold-polymerized denture base resin and acrylic teeth	Probase cold and SR Orthotyp S PE	Ivoclar Vivadent, Schaan, Liechtenstein
Cold-polymerized denture base resin and nanohybrid composite teeth	Probase cold and Phonares II Typ	Ivoclar Vivadent, Schaan, Liechtenstein
Cold-polymerized denture base resin and cross-linked teeth	Probase cold and SR Orthotyp DCL	Ivoclar Vivadent, Schaan, Liechtenstein
Heat-polymerized denture base resin and acrylic teeth	Probase hot and SR Orthotyp S PE	Ivoclar Vivadent, Schaan, Liechtenstein
Heat-polymerized denture base resin and nanohybrid composite teeth	Probase hot and Phonares II Typ	Ivoclar Vivadent, Schaan, Liechtenstein
Heat-polymerized denture base resin and cross-linked teeth	Probase hot and SR Orthotyp DCL	Ivoclar Vivadent, Schaan, Liechtenstein
CAD/CAM (milled) denture base resin and acrylic teeth	IvoBase CAD and SR Orthotyp S PE	Ivoclar Vivadent, Schaan, Liechtenstein
CAD/CAM (milled) denture base resin and nanohybrid composite teeth	IvoBase CAD and Phonares II Typ	Ivoclar Vivadent, Schaan, Liechtenstein
CAD/CAM (milled) denture base resin and cross-linked teeth	IvoBase CAD and SR Orthotyp DCL	Ivoclar Vivadent, Schaan, Liechtenstein
CAD/CAM (milled) denture base resin and CAD/CAM (milled) denture teeth	IvoBase CAD and SR Vivodent CAD	Ivoclar Vivadent, Schaan, Liechtenstein

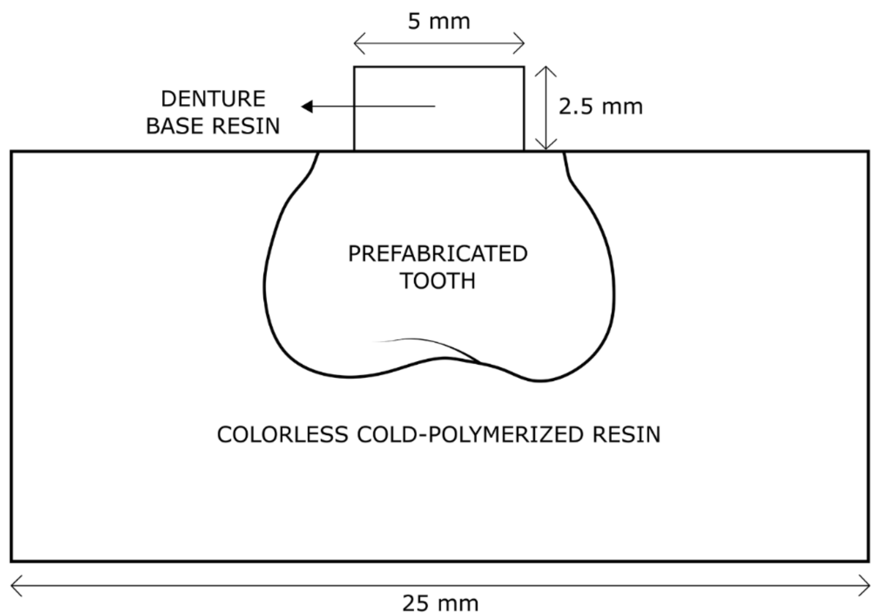

Figure 1. Diagram of the specimens for groups 1-9.

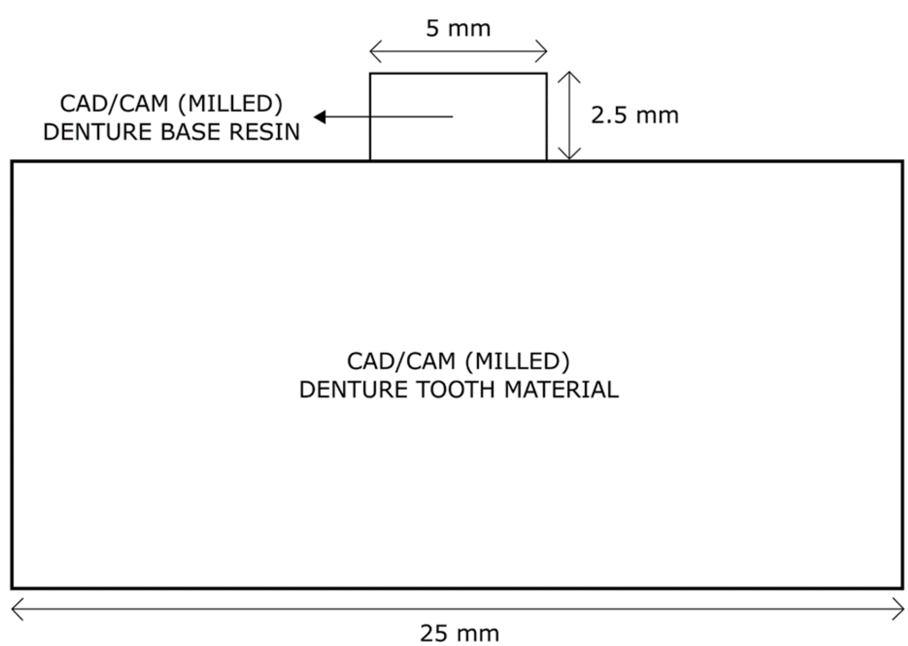

Figure 2. Diagram of the specimens for group 10.

Specimen preparation was similar to that in a previous research (1, 26, 27). Each prefabricated tooth was immersed in a colorless cold-polymerized acrylate (Clarocit Kit, Struers Co., Ballerup, Denmark) in a plastic mold (FlexiForm round, Struers Co., Ballerup, Denmark) according to the manufacturer's instructions. After cooling, specimens (acrylic cylinder with immersed tooth) were carefully removed from the plastic molds. The ridge lap surface of each tooth was exposed with a water cutting machine (IsoMet 1000, Buehler, Lake Bluff, IL, USA). No other treatment of the ridge lap surface of teeth was used. A metallic mold was employed to create silicone samples (Optosil Comfort Putty, Kulzer, Hanau, Germany) with a 25.0 mm diameter × 2.5 mm height. Each sample had a circular opening in the center, with a 5.0 mm diameter × 2.5 mm height, for preparation of the denture base resin cylinders. Silicon samples were fixed with an instant adhesive (Loctite, Henkel, Düsseldorf, Germany) on the exposed surface of the acrylic cylinders, which comprised of the embedded prefabricated tooth. The circular opening in silicone was filled with wax before the flasking procedure to secure space for heat-polymerized acrylics. Specimens of prefabricated teeth and heat-polymerized cylinders were obtained via the flasking procedure. The lower part of the flask was filled with gypsum, and the specimen was immersed. The gypsum was coated with separating medium (Separating Fluid, Ivoclar Vivadent, Schaan, Liechtenstein), the upper part of the flask was positioned, and the second layer of gypsum was placed, followed by complete closure of the flask. After gypsum induration, the flask was opened, and the wax was removed. The packing stage followed, and polymerization was carried out according to the manufacturer's instructions in an appropriate polymerization unit (EWL Typ 5509, Kavo, Biberach, Germany). Each flask was left for cooling at room temperature. Then, the specimens were carefully deflasked and cleaned.

Cold-polymerized cylinders were obtained in a similar manner by inserting cold-polymerized acrylics into a circular opening of the silicon mold. Following the manufacturer's instructions, polymerization was carried out in a pressure device (Ivomat IP2, Ivoclar Vivadent, Schaan, Liechtenstein) at 40°C and at 6 bar pressure for 15 minutes.

CAD/CAM cylinders (5.0 mm diameter × 2.5 mm height) were constructed (Netfabb Premium 2019, Autodesk, San Rafael, CA, USA) and saved as a standard tessellation language (STL) file. A milling machine (Ceramill Mikro 5X, Amann Girrbach, Koblach, Austria) was used to obtain CAD/CAM cylinders (IvoBase CAD) according to the attained STL file. The cylinders were glued with a PMMA-based bonding material (IvoBase CAD Bond Kit 10, Ivoclar Vivadent, Schaan, Liechtenstein) to the exposed surface of the teeth (SR Orthotyp S PE; Phonares II Typ; SR Orthotyp DCL; and SR Vivodent CAD). CAD/CAM tooth cylinders of 25.0 mm diameter × 12.0 mm height were milled from CAD/CAM discs (SR Vivodent CAD, Ivoclar Vivadent, Schaan, Liechtenstein) using the same software (Netfabb Premium 2019) and the milling machine (Ceramill Mikro 5X). In the center of each CAD/CAM (milled) tooth cylinder, a CAD/CAM (milled) denture base resin cylinder (5.0 mm diameter × 2.5 mm height) was glued with a bonding agent (IvoBase CAD Bond Kit 10). The specimens appeared like denture teeth embedded in acrylic (first mandibular molars) bonded to denture base resin cylinders with 5.0 mm diameter × 2.5 mm height (1). All specimens were stored in distilled water at 37°C for 48 hours before testing.

The shear bond strength between denture base resins and prefabricated teeth and teeth produced by CAD/CAM (milled) was determined using a universal shear bond strength testing machine (model LRX, Lloyd Instruments, Fareham, Great Britain) at a 1 mm/min crosshead speed. The load at fracture was recorded and presented by the software of the testing machine (Nexygen, Lloyd Instruments, Fareham, Great Britain). Failure was classified by using a stereomicroscope (Olympus SZX10, Olympus, Tokyo, Japan) at a magnification of 120× as adhesive, cohesive, or mixed. Adhesive failure (Fig. 3) refers to complete detachment between the denture base resin and a prefabricated tooth; cohesive failure (Fig. 4) refers to a complete fracture in the denture base resin or tooth; mixed failure (Fig. 5) refers to both occurring simultaneously (7). Normality of distribution was tested with the Shapiro–Wilk test. Analysis of variance (one-way ANOVA) and Tukey multiple comparison test were used to compare the obtained values among different groups of material (normal distribution). The analysis was carried out using a statistical software package (SPSS Statistics 17.0, IBM, Armonk, NY, USA) on the Windows platform. The significance level was set at 5%.

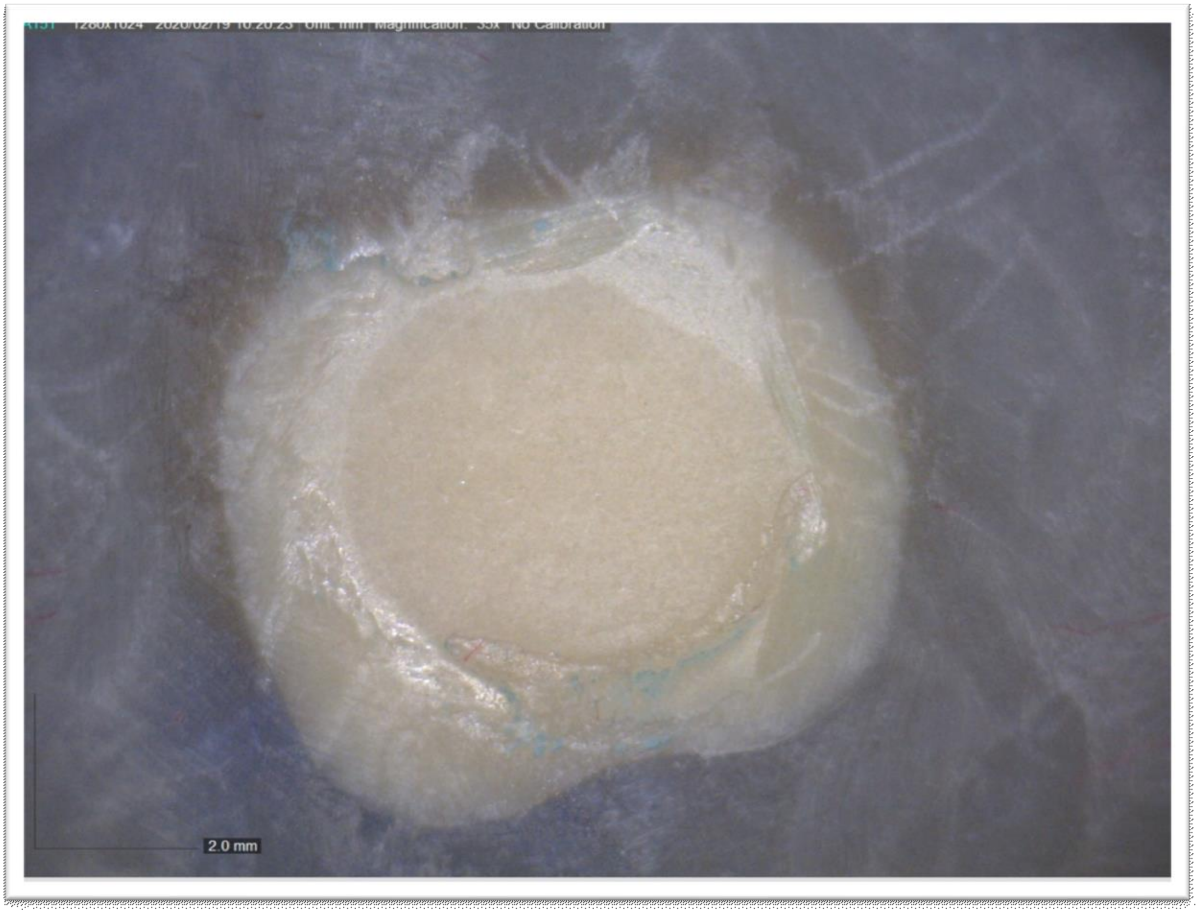

Figure 3. Adhesive failure.

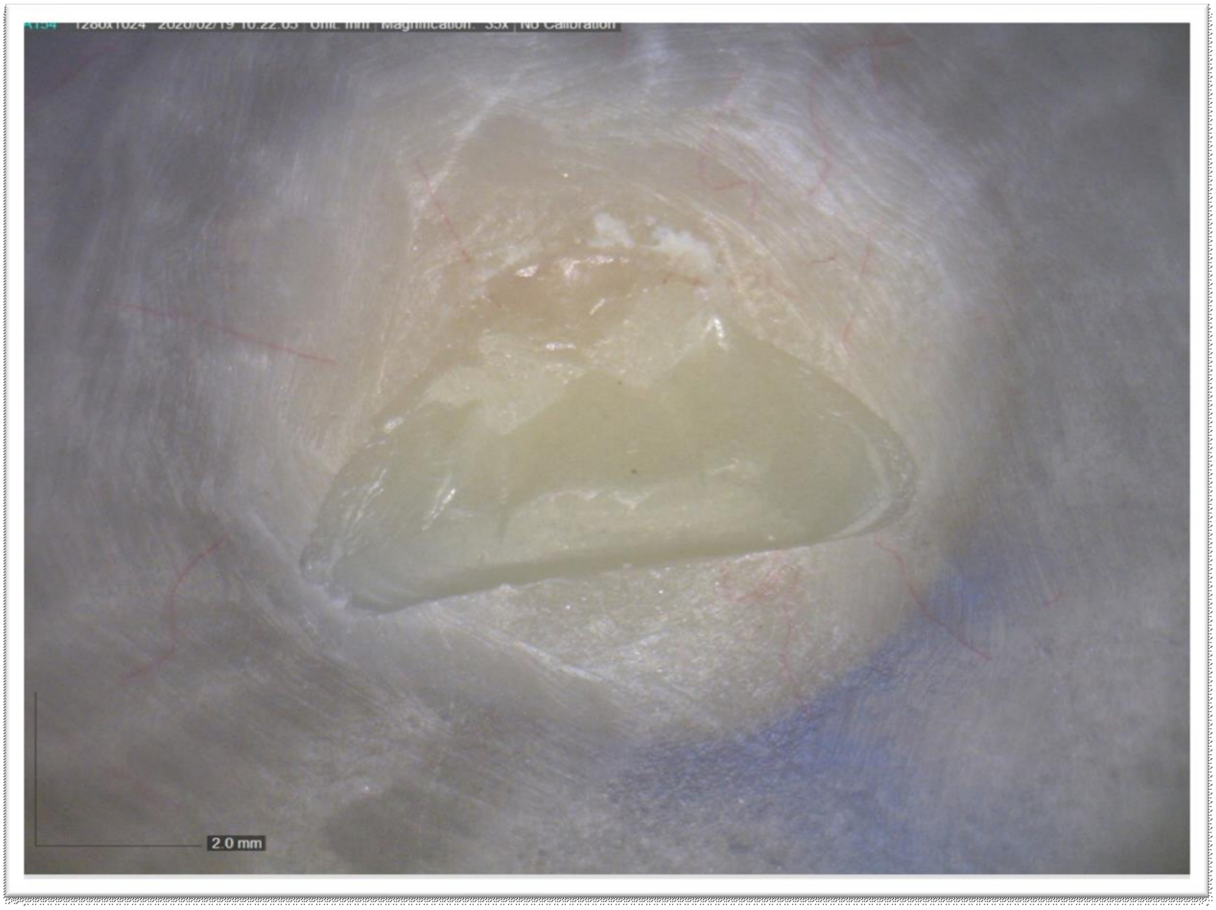

Figure 4. Cohesive failure.

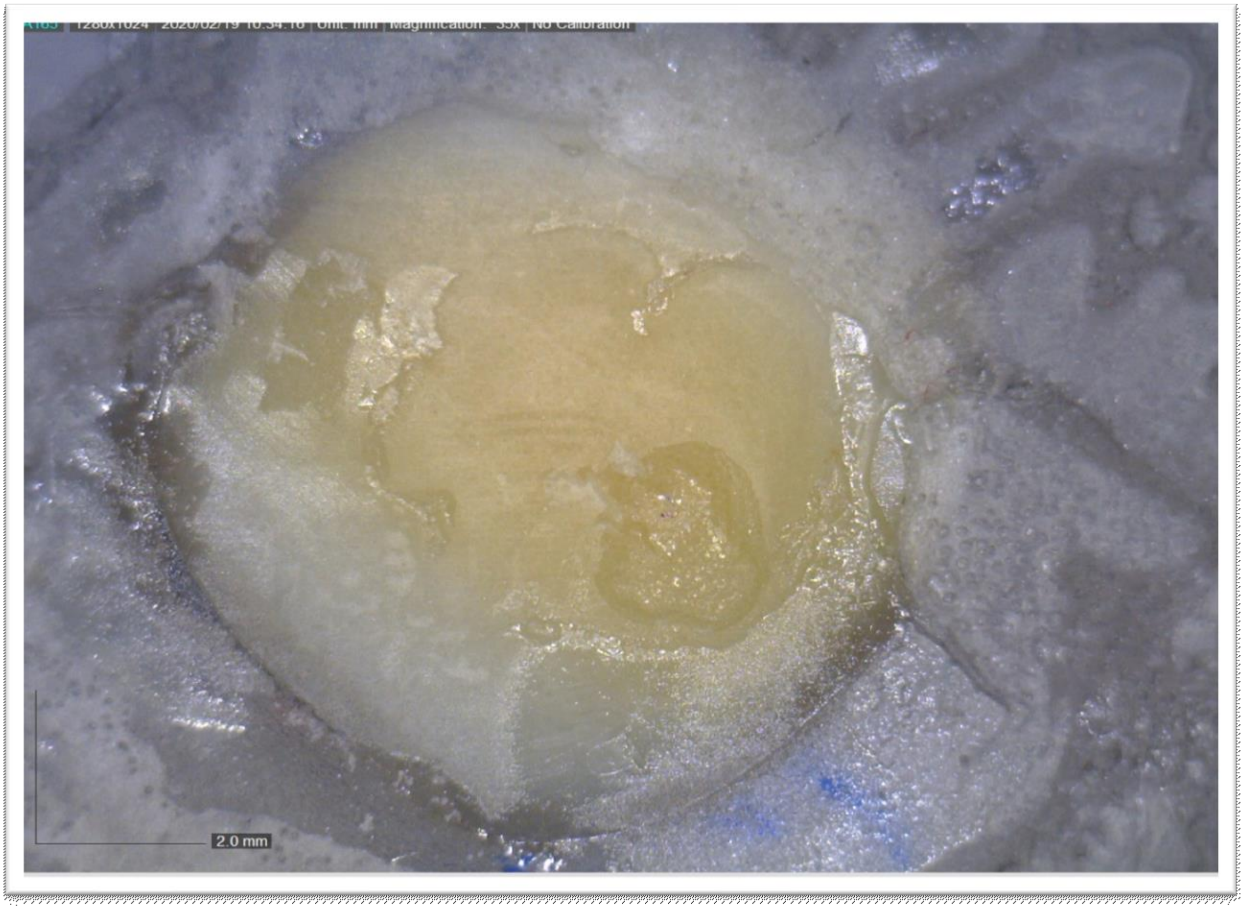

Figure 5. Mixed failure.

5.4. Results

Descriptive statistics for shear bond strength values of different groups are presented in Fig. 6. The minimal and maximal shear bond strength values (in MPa) of the tested groups were 0.90 and 6.49 for group 1, 5.58 and 13.18 for group 2, 5.73 and 18.27 for group 3, 13.02 and 20.45 for group 4, 5.53 and 19.07 for group 5, 7.20 and 19.89 for group 6, 8.54 and 16.03 for group 7, 12.66 and 17.71 for group 8, 9.26 and 18.92 for group 9, and 8.28 and 22.54 for group 10. One-way ANOVA showed significant differences in shear bond strength values among the tested groups ($P < .0001$). The results of the Tukey post-hoc test are shown in Fig. 6. Modes of failure are presented in Table 2.

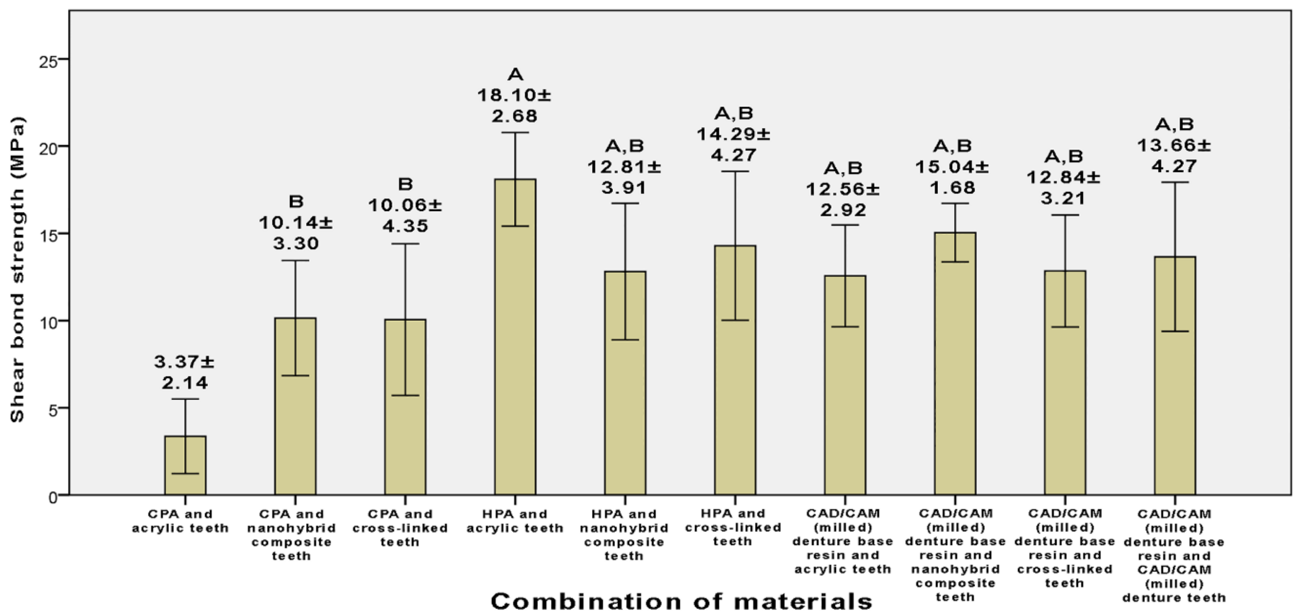

Figure 6. Means and standard deviations of shear bond strength for groups. Similar uppercase letters denote no significant differences between groups (Tukey post-hoc test, $P > .05$). CPA – cold-polymerized acrylics; HPA – heat-polymerized acrylics.

Table 2 Number of tested groups, combination of materials, number of specimens and mode of failure

Group	Combination of materials	n	Adhesive failure	Cohesive failure	Mixed failure
1.	Cold-polymerized denture base resin and acrylic teeth	8	7	-	1
2.	Cold-polymerized denture base resin and nanohybrid composite teeth	8	5	2	1
3.	Cold-polymerized denture base resin and cross-linked teeth	8	5	3	-
4.	Heat-polymerized denture base resin and acrylic teeth	8	1	7	-
5.	Heat-polymerized denture base resin and nanohybrid composite teeth	8	2	6	-
6.	Heat-polymerized denture base resin and cross-linked teeth	8	1	7	-
7.	CAD/CAM (milled) denture base resin and acrylic teeth	8	3	5	-
8.	CAD/CAM (milled) denture base resin and nanohybrid composite teeth	8	-	8	-
9.	CAD/CAM (milled) denture base resin and cross-linked teeth	8	1	6	1
10.	CAD/CAM (milled) denture base resin and CAD/CAM (milled) denture teeth	8	3	5	-

5.5 Discussion

The present study investigated the shear bond strength between different types of denture base resins and different types of prefabricated and CAD/CAM (milled) denture teeth, with an emphasis on digitally produced denture base resins. The null hypothesis was rejected because the statistical analysis showed differences among the study groups.

Shear bond strength is the strength of a material or component against the type of yield or structural failure when the material or component fails by shear force. Since a tooth can detach from a denture base for various reasons, it is important that the shear bond strength is as high as possible. In this study, the highest shear bond strength values (18.10 ± 2.68 MPa) were observed in group 4 (heat-polymerized denture base resin and acrylic teeth; Fig. 6). Because acrylic teeth can chemically bond to PMMA denture base resins, (7) these findings were expected, and they are comparable to the results of other studies (8, 11).

The results of the present study determined the lowest values in cold-polymerized denture base resins (Fig. 6). This finding is similar to previous studies, which compared the shear bond strength of heat-polymerized and cold-polymerized denture base resins with prefabricated teeth (8, 11, 26, 28). Although cold-polymerized denture base resins have been promoted as an alternative to heat-polymerized ones, (2) they are not capable of diffusing effectively into the denture tooth surface. According to Chung et al., (20) their shear bond strength can only reach one-quarter of the strength of heat-polymerized denture base resins. The closest investigation to the present study is the investigation by Choi et al. (9). These authors followed the same scientific question and compared heat-polymerized, CAD/CAM (milled), and 3D-printed denture base resins with four types of commercial denture teeth. They determined that the highest bond strength values were present in heat-polymerized denture bases, followed by CAD/CAM (milled), while 3D-printed resin showed the lowest bond strength values. The results are opposite of those of the present study, in which differences in CAD/CAM (milled) and heat-polymerized denture bases were not determined (Fig. 6). It must be considered that the study by Choi et al. (9) had a different experimental design; the authors used different combinations of materials and different bond strength tests (flexural bond strength), making comparison difficult. Different adhesives are used to overcome the difficulty in achieving adequate chemical bonding, (29) and their ability to bond various materials is well known (30, 31). In a study by Rosca et al., (29) the authors obtained adequate shear bond strength values between light-polymerized composite and PMMA using a universal adhesive. Yanikoglu et al. (10) examined the shear bond strengths of light-polymerized composites and cold-polymerized acrylics to acrylic teeth and reported that, if an adequate bonding agent is used, enhanced bonding can be attained with a composite material. In a systematic review, Mine et al. (32) concluded that materials containing methyl methacrylate improved the bonding of CAD/CAM PMMA resin materials. Previous research (10, 17, 32, 33) and the present study results (Fig. 6) suggest that PMMA-based bonding material is an option for denture teeth placement that is comparable to processing with heat polymerization.

Prefabricated teeth can be attached to a 3D-printed denture base using different techniques, including bonding with a light-polymerized bonding agent (11) or with cold or heat polymerization (19, 21). Since attaching of prefabricated teeth to the 3D-printed denture base using cold or heat polymerization is a customary way of finishing 3D printed digital dentures, in the first six groups of the present study, it was also aimed to evaluate the shear strength of different types of prefabricated teeth to the 3D-printed denture base. Consequently, from the present (Fig. 6) and previous studies (9, 11, 26) results, it is recommended to bond 3D-printed denture base resins and prefabricated teeth with heat polymerization to obtain optimal shear bond strength values. In average, lower bond strength values are expected with prefabricated teeth bonded to cold-polymerized acrylics (Fig. 6) (8, 11, 20, 26, 28). Therefore, such acrylics should be avoided when attaching prefabricated teeth to a 3D-printed denture base.

The mode of failure has been used as a measure of the performance of bonding (12). Adhesive failures have been considered the least acceptable, mixed failures acceptable, and cohesive failures perfect (12). In this study, it was found that, with higher shear bond strength values, higher percentages of cohesive and mixed failures were also evident, which is in accordance with a study by Akin et al. (13). CAD/CAM (milled) and heat-polymerized denture bases had similar percentages of cohesive and mixed failures, while coldpolymerized acrylics showed mostly adhesive failures (Table 2). Similar results to our study were obtained by Jain et al. (5) and Neppelenbroek et al., (6). who stated that the cohesive failure mode between heat-polymerized denture base resin and acrylic teeth occurred in 100% of instances. The prevalence of cohesive failures suggests that the bond strength between denture base acrylics and prefabricated teeth was higher compared with the resistance of each material alone (14).

The comparison between the results of the present study and previous studies is difficult because there has been no standardization of testing techniques in the literature, as well as because of the diversity of dental materials that were used (2). With emerging technologies, there are different ways of processing and finishing removable dentures. The present study aimed to compare different types of removable denture base materials and has included 10 different combinations with different denture teeth. Still, due to inaccessibility, not every possible combination was included, which is a limitation of the present study. In future, building on the present study results, similar studies could estimate the bond strength between the 3D printed denture base resin and artificial teeth, and also determine the most favorable option for attaching teeth to an appropriate denture base.

5.6. Conclusion

Shear bond strength significantly depends on the selected combination of a denture base material and a denture tooth material. Materials with higher shear bond strength values (heat-polymerized resins and CAD/CAM [milled] denture base resins) show mostly cohesive and mixed modes of failure compared with cold-polymerized resins, which mainly exhibit adhesive modes of failure. Denture teeth glued to a CAD/CAM (milled) denture base using a PMMA bonding agent show similar bond strength compared with denture teeth attached to heat-polymerized denture base resin.

5.7 Acknowledgements

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6. INVESTIGATION OF MECHANICAL PROPERTIES OF CONVENTIONAL AND DIGITAL DENTURES MATERIALS

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6.1 Abstract

Introduction: Complete dentures have been used for many years, and they represent a gold standard for treating edentulism. Recent improvements in science and technology have provided digital methods for denture base production, including computer-aided design/ computer-aided manufacturing (CAD/CAM) and three dimensional (3D) printing. This investigation of impact strength is continuation of the study "Comparison of Mechanical Properties of 3D-Printed, CAD/CAM, and Conventional Denture Base Materials" (1) which determined flexural strength and surface hardness values.

Materials and methods: A total of 160 rectangular specimens were fabricated from three conventional heat-polymerized acrylics, three CAD/CAM produced, one 3D-printed, and one polyamide material for denture base manufacturing. The flexural strength test was the three-point flexure test, while hardness testing was conducted using the Brinell method. Impact strength was tested on the Charpy's bar. The data were analyzed using descriptive and analytical statistics ($\alpha = 0.05$).

Results: The flexural strength values ranged from 71.7 ± 7.4 MPa to 111.9 ± 4.3 MPa (1). The surface hardness values ranged from 67.13 ± 10.64 MPa to 145.66 ± 2.22 MPa (1), while the impact strength values ranged from 8.01 ± 3.52 kJ/m^2 to 44.68 ± 39.37 kJ/m^2. During flexural testing, the IvoBase CAD and Vertex ThermoSens specimens did not fracture during loading. CAD/CAM and polyamide materials had the highest flexural strength values. The 3D-printed material had the lowest flexural strength values. Two groups of CAD/CAM materials had the highest surface hardness values. Poylamide and CAD/CAM materials showed the highest values of impact strength.

Conclusions: CAD/CAM materials show better mechanical properties than heat-polymerized and 3D-printed acrylics do. Nevertheless, a materials polymerization type does not guarantee its optimal mechanical properties.

Keywords: acrylic resin; denture base; flexural strength; surface hardness; impact strength

7. GENERAL DISCUSSION

Studies dealing with mechanical properties of denture base materials have recently gained much interest. Among different materials and different technologies, milled CAD/CAM materials for denture base fabrication show enhanced mechanical properties when compared to heat-polymerized and 3D-printed materials. The first part of the present study examined the mechanical properties of denture base materials made with different technologies. The study emphasized digital technologies (CAD/CAM, milling, and 3D printing). Specifically, flexural strength, surface hardness, and impact strength of heat-polymerized acrylic resins, milled CAD/CAM materials, 3D printed material, and polyamide material were examined.

In accordance with ISO 20795-1:2013 (112), a three-point flexure test was used for analyzing flexural properties of denture base materials (75). The three-point flexure test simulates conditions which occur in patients' mouth during mastication and parafunctional activities (41, 75, 113, 114). The above-mentioned standard indicates that acrylic resins should accomplish values equal or above 65 MPa (75, 112). Since all the tested materials had 65 MPa or higher flexural strength values (Chapter 4, Figure 1), it follows that all materials used during the present study are convenient for clinical practice.

Denture base resins are exhibited to complex stomatognathic stresses in the oral cavity (4). Correspondingly, optimal mechanical properties are necessary for the functional implementation of denture base resins which must have high flexural strength values to endure the forces of mastication without deformity or failure (4). Prosthodontic materials should serve in a stomatognathic system without unfavorable outcome on the oral tissues. Accordingly, the PMMA used as a denture base material must be biocompatible, without causing irritations to the surrounding tissues (4).

Flexural strength represents one of the main determinants of the mechanical properties of denture base acrylic resins, and is influenced by the degree of accomplished polymerization (41, 75). When comparing flexural strength values of acrylic denture base resins, the results suggest that materials with a lower degree of conversion demonstrate poorer mechanical properties (41, 75). Since CAD/CAM (milled) denture base materials are made under high heat and pressure, the degree of conversion is higher compared to the conventional ones (75). Accordingly, it is expected that flexural strength values of CAD/CAM (milled) denture base materials to be higher than in heat-polymerized acrylics (72, 75). Previous studies showed that flexural strength of CAD/CAM (milled) and conventional denture base materials differ (71, 74–76). Steinmassl et al. (74) determined heterogeneous results whereas different milled denture base resins demonstrated higher, similar, or lower flexural strength values when compared to a heat-polymerized group. Pacquet et al. (76) and Ayman (71) obtained lower flexural strength values in CAD/CAM (milled) denture base materials than in a heat-polymerized acrylic resin. Opposite results were obtained in a study by Aguirre at al. (75), where CAD/CAM (milled) denture base materials demonstrated higher flexural strength values when compared to compression molded denture base resins, which is in accordance with the present study results (Chapter 4, Figure 1).

The results of the present study (Chapter 4, Figure 1) showed that polyamide has higher flexural strength values than two CAD/CAM (milled) denture base materials (Interdent CC disc PMMA and Polident CAD/CAM disc basic). Previous studies that investigated polyamide as a denture base material obtained different results (115–118). Yunus et al. (28) determined flexural properties of polyamide denture base material and compared the results with a conventional compression-molded heat-polymerized denture base resin. Polyamide showed lower flexural strength when compared to two compression-molded PMMA denture base resins (28). Takabayashi (118) compared characteristics of six thermoplastic denture resin materials. In the study flexural strength of polyamide materials did not meet ISO requirements for flexural strength (65 MPa) (118). However, apart from the studies by Yunus et al. (28) and Takabayashi (118), literature agrees that polyamide materials have higher flexural strength values than heat-polymerized denture base materials (115–117). This is apparent in clinical practice where polyamide materials show energy absorption when a patient drops a denture (67). On the other hand, the flexibility of polyamide materials questions the possibility of the material to show enough rigidity to distribute the forces uniformly over the dental arch (67). Accordingly, higher flexibility could represent a shortcoming from a clinical aspect (24).

In 3D printed material (NextDent Base) the lowest flexural strength values were determined when compared with other study groups (Chapter 4, Figure 1). This finding is in agreement with the study by Gad et al. (119). The authors assumed that lower flexural strength in 3D printed acrylics could be due to different material composition (119). Flexural strength of 3D printed, cold-polymerized, and heat-polymerized denture base materials was also compared in the study by Perea-Lowery et al. (120). The results of the study showed inferior flexural strength values of 3D printed denture base resin when compared to heat-polymerized, and are in agreement with the present study results (Chapter 4, Figure 1).

3D printed materials use monomers based on acrylic esters and have relatively low double bond conversion compared to conventional acrylic resins (121). Even though 3D printed materials showed the lowest flexural strength values in the present study (Chapter 4, Figure 1), they met ISO requirements for flexural strength (65 MPa) (112). With higher or equal values to ISO's 65 MPa limit for denture base materials, today's 3D printed materials have enough flexural strength to be used in a clinical practice. The reason why 3D printed materials have lower flexural strength values could be due to layering build in a direction parallel with the load direction (119). Still, with frequent accidental or functional fractures of denture bases seen in practice, most clinicians would prefer safer options (materials with higher flexural strength). Having this in mind, 3D printed denture base materials need to develop more in order to suppress the use of conventional material.

Even though the enhanced flexural strength values of CAD/CAM (milled) denture base resins showed promising results (Chapter 4, Figure 1), future studies ought to estimate other mechanical properties.

In accordance with ISO 2039-1:2001 (122) surface hardness was tested with Brinell's method. Hardness is defined as the resistance to indentation (123). By testing surface hardness, forces that a denture base resin can resist during mastication can be simulated (123). Dentures with low surface hardness might be defected by eating or brushing thus causing plaque retention, which could then reduce the duration of dentures.

In the present study (Chapter 4, Figure 2) two groups of milled CAD/CAM material (Interdent CC disc PMMA and Polident CAD/CAM disc basic) showed the highest surface hardness values. These results are in accordance with the study by Ayman (71), where the author reported higher hardness values for CAD/CAM materials when compared to heat-polymerized materials. However, the third tested milled CAD/CAM material (IvoBase CAD) showed lower hardness values than most of the tested groups (Chapter 4, Figure 2). Similar results were obtained by Becerra et al. (124), where the authors examined compression-molded, high-pressure polymerized, and milled denture base materials. In the study milled denture base material (Ivobase CAD) showed the lowest hardness values when compared to the other tested groups (124). It could be concluded that specific milled denture base material (Ivobase CAD) has lower hardness values than other tested CAD/CAM (milled) denture base materials, and that denture base resins cannot be studied only via polymerization processing. Chang et al. (125) compared hardness of polyamide and milled denture base material. The milled material had higher hardness values when compared to polyamide, (125) which is in accordance with the present study results (Chapter 4, Figure 2).

The present study results also showed that polyamide has the lowest values of surface hardness (Chapter 4, Figure 2). These results are in accordance with the study by Ucar et al. (67), where the authors compared hardness of polyamide denture base material with conventional compression-molded PMMA. In the study polyamide showed much lower values than other materials (67). The authors concluded that polyamide material is not as hard as other tested materials (67). Shah et al. (126) showed that PMMA material has higher hardness values than a flexible resin. This could be due to a high monomer-polymer ratio, and the presence of methyl-methacrylate monomer (126). Furthermore, PMMA material may contain cross-linking agents (126). Flexible resins have lower amounts of cross-linking agents when compared to PMMA material, which can imply that the cross-linking agent may affect surface hardness (126).

3D printed material (NextDent Base) showed lower surface hardness values than most of the other tested acrylics (only Ivobase CAD had lower surface hardness values, Chapter 4, Figure 2). The finding is similar to the study done by Gad et al. (119). The authors investigated flexural strength and hardness of 3D printed denture base materials, and determined that 3D printed material has lower flexural strength and hardness values than a heat-polymerized denture base resin. Lower hardness values of 3D printed denture base materials could be explained by material composition and water sorption with thermal stressing (119). With a deficiency of similar studies, future research is necessary for a better insight in 3D printed denture base resins properties.

In accordance with ISO 179-1:2010 (127) impact strength was tested on the Charpy's bar. Impact test is utilized to simulate experimental fractures and identify the amount of energy that acrylic denture base resin can absorb before fracturing (128). Most frequently it is used as an indicator of the ability of denture base structure to resist fracture when given a sudden shock, mostly outside the patient's oral cavity (128, 129).

In the present study (Chapter 6) polyamide material (Vertex ThermoSens) showed the highest impact strength values. These results are in accordance with the study by Helal et al. (129) , where authors reported higher impact strength values of polyamide, when in comparison with CAD/CAM (milled) and 3D printed denture base resins. This can be assignable to lower amount of cross-linking agents which enhance the flexibility (129).

When comparing impact strength of CAD/CAM (milled) and 3D printed denture base resin, it could be noted that CAD/CAM (milled) denture base resins (IvoBase CAD, Polident CAD/CAM, Interdent CC disc PMMA) demonstrated higher impact strength values (Chapter 6). Findings of the present study are in accordance with the study by Helal et al. (129). Above-mentioned can be explained with homogeneous structure and free of porosities or air bubbles of CAD/CAM (milled) denture base resins (129). In addition, the lower values of 3D printed denture base resins could be due to layered-printing of the specimens in a direction that parallels the impact load (119).

Chhabra et al. (130) and Lee et al. (131) compared impact strength of 3D printed and heat-polymerized denture base resins. Heat-polymerized denture base resins demonstrate higher impact strength values(130, 131). This is in agreement with present study - 3D printed material (NextDent Base) showed the lowest impact strength values when compared to all study groups. Further studies are essential to improve the mechanical properties of 3D printed denture base resins which could launch the accessibility of improved 3D printed materials when compared to conventional ones on the market (130).

In the second part of the present study, the shear bond strength between denture base resins and different types of prefabricated teeth, and milled CAD/CAM teeth was examined. The study emphasized digitally fabricated denture base resins and milled denture teeth. Specifically, three types of prefabricated teeth (acrylic, nanohybrid composite, and cross-linked) were attached to a cold-polymerized denture base resin, a heat-polymerized denture base resin, and a CAD/CAM (milled) denture base resin. Also, one group tested milled denture teeth and a milled denture base resin.

Shear bond strength is the strength of a material or component against the type of yield or structural failure when the material or component fails by shear force (Chapter 5). A debonding of prefabricated teeth from denture base resin can be frustrating for patients and dentists as well (92). Since a prefabricated tooth can detach from a denture base for different causes, it is essential that shear bond strength values are highest possible. 22 % to 30 % of denture repairs include a debonding of an artificial tooth, especially in the anterior portion of a denture. The above-mentioned detachment could be assignable to a minor ridge lap surface area available for bonding, and the direction of stresses during mastication (81). Nowadays, scientists have found methods to increase the shear bond strength between denture base resins and artificial teeth with chemical, and physical changes of artificial teeth and polymers (92). The ridge lap surface of the artificial teeth used in the present study was intact in order to exclude other influence on shear bond strength apart from the studied material combination. Most of the efforts to strengthen the bond between acrylic denture base resins and denture teeth include chemical treatment and mechanical customization of the ridge lap surface of an artificial tooth (79). The given techniques involve wetting using methyl methacrylate monomer, sandblasting, laser irradiation, bur grooving, or a combination of those (86).

In the present study the highest shear bond strength values (18.10 ± 2.68 MPa) were measured between heat-polymerized denture base resin and acrylic teeth. These findings were expected because acrylic teeth can chemically bond to PMMA denture base resins (132). Moreover, Gharebagh et al. (92) compared shear bond strength values between artificial teeth (acrylic Ivoclar, Apple composite, and B-Star nanocomposite) and heat-polymerized denture base resin. The results showed that Ivoclar acrylic teeth have the highest shear bond strength values (92), which is in accordance with the present study results (Chapter 5, Figure 6).

The lowest shear bond strength values were measured in cold-polymerized denture base resins, especially between cold-polymerized denture base resin and acrylic teeth (3.37 ± 2.14 MPa) (Chapter 5, Figure 6). Some previous studies compared the shear bond strength of cold-polymerized and heat-polymerized denture base resins with artificial teeth and obtained similar results; cold-polymerized denture base resins and artificial teeth showed low shear bond strength values (79, 81, 85, 133). Shear bond strength of cold-polymerized denture base resins to prefabricated teeth can only get a quarter of the shear bond strength than heat-polymerized denture base resins can reach (101).

Contrary to the present study results (Chapter 5, Figure 6), Choi et al. (99) determined the highest bond strength values in heat-polymerized denture base resins, followed by CAD/CAM (milled), whilst 3D printed denture base resins demonstrated the lowest bond strength values. Choi et al. (99) compared the above-mentioned denture base resins with four types of artificial teeth (unfilled PMMA, double cross-linked PMMA, PMMA with nanofillers, and 3D printed resin teeth). It must be noticed that the authors (99) used a different bond strength test (flexural bond strength) and different combinations of materials, which makes the comparison difficult.

Prefabricated teeth can be attached to a 3D printed denture base resin with a bonding agent (79) or by using heat or cold polymerization (9, 134). Since heat or cold polymerization is a common procedure of finishing digital dentures, the first six groups of the present study (shear bond strength testing) also evaluated the shear bond strength of different types of artificial teeth to a 3D-printed denture base resin (Chapter 5). Based on the present study (Chapter 5, Figure 6) and some previous studies' results (79, 85, 99), it is advisable to bond prefabricated teeth and 3D-printed denture base resins by using heat polymerization to attain adequate shear bond strength values. Lower shear bond strength values are expected when prefabricated teeth are bonded to cold polymerized acrylics (79, 81, 85, 101, 133). Consequently, cold-polymerized acrylics should be averted when attaching prefabricated teeth to a 3D-printed denture base resin (Chapter 5).

Furthermore, mode of failure is used for the categorization of the performance of bonding (Chapter 5). After the testing process, every specimen was observed by a stereomicroscope (Olympus SZX10, Olympus) and categorized into one of three groups:

1 Adhesive failure means a total detachment between a prefabricated tooth and a denture base resin

2 Cohesive failure means a total break in a prefabricated tooth or a denture base resin

3 Mixed failure means both adhesive and cohesive failure occurring at the same time (Chapter 5).

The least eligible type of failure was adhesive failure, while cohesive failure was considered ideal. Mixed type of failure was considered admissible (Chapter 5). The present study showed that the higher shear bond strength values were measured, the higher percentages of mixed and cohesive failures were perceived (Chapter 5). The above-mentioned present study results are in accordance with the study by Akin et al. (88). Thean et al. (93) and Korkmaz et al. (135) interpret that cohesive failure happens when the bond strength of the interface surpasses the strength of subject material, which demonstrates tenable bond. Similar amount of mixed and cohesive failures (Chapter 5) was determined in heat-polymerized and CAD/CAM (milled) denture base resins. On the other hand, cold-polymerized denture base resins showed chiefly adhesive type of failure. Jain et al. (136) and Neppelenbroek et al. (137) attained equivalent results. The authors (136, 137) adduced that a cohesive failure between a heat-polymerized denture base resin and acrylic teeth occurred in 100% of cases. Altogether, the frequency of cohesive failures between denture base resins and denture teeth suggests that when well bonding is achieved, failure strength is defined by the strength of denture teeth (138).

Polymethyl methacrylate (PMMA) polymers are also used for occlusal devices' fabrication due to its easy usage. So far, the standard way to fabricate occlusal devices include vacuum thermoforming foil and an autopolymerizing PMMA resin. As a result of the advancement in technology, occlusal devices can be fabricated by using subtractive or additive manufacturing (Chapter 3). According to Brandt et al. (139) and Yuzbasioglu et al. (140), subtractive digital techniques have been depicted as more efficient when compared to the conventional ones.

A subtractive production of occlusal devices requires three major items: information received throughout intraoral scanners, a software meant to create virtual restoration, and a CAD/CAM (milling) device. A completely digital production can be modified with the use of a conventional impression. After the impression taking, the impression or corresponding cast is scanned to obtain necessary information in a digital form.

The category of additive technology used for 3D printed occlusal device fabrication is mostly stereolithography (SLA). During the polymerization process SLA photopolymers turn from liquid to solid stage under UV light. In the mentioned process, an object is being manufactured layer by layer until the definitive item (occlusal device) is acquired (141).

Technology similar to SLA is a digital light processing (DLP) (56). In DLP a liquid photopolymer is exposed to light from a projector. The projector shows an image of a 3D model on a liquid photopolymer. In the technology mentioned, an object is pulled up from the liquid resin and the procedure is repeated until the definitive object is built. The basic difference between SLA and DLP is a source of light (56).

Occlusal devices are frequently used for the treatment of temporomandibular disorders (TMD). TMD includes a large group of clinical signs and symptoms which involve the temporomandibular joint (TMJ), masticatory musculature, and surrounding tissues (bony and soft) (142).

In the present pilot study (Chapter 3), the mechanical properties (flexural strength and surface hardness) of occlusal device materials made with different technologies were examined. The study emphasized CAD/CAM (milled) and 3D printing technology. Particularly, flexural strength and surface hardness of 3D printed, CAD/CAM (milled), and conventional autopolymerizing occlusal device materials were examined. In accordance with ISO standard (ISO 20795-1:2013) (112), a three-point flexure test was used for analyzing flexural properties of occlusal device materials. None of the specimens of cross-linked polyamide material (CopraDur) and nonacrylic lightpolymerizing resin for additive manufacturing (VarseoWax Splint) fractured when loaded with the end limits of the possible movement of the penetrant (Chapter 3), which is in accordance with the study by Ucar et al. (67). A few studies examined polyamide materials for denture base fabrication (67, 115–117, 142–145), even though the mentioned studies did not investigate materials for occlusal device fabrication. The studies by Hamanaka et al. (115), Takahashi et al. (116), and Sasaki et al. (117) showed that polyamide materials are more flexible when compared to acrylic resins, and similar results were obtained in the present study (Chapter 3, Figure 2).

Even though flexibility is essential for energy absorption when a patient drops a splint (67), nonflexible occlusal appliances deemed to be a better choice for patients suffering bruxism (146–148). The clinical implications of mechanical properties of digitally produced occlusal devices are not definite so far. Berli et al. (149) compared the mechanical properties (flexural strength and hardness) of 3D printed, pressed, and milled materials for occlusal device fabrication. The authors reported higher flexural strength values in milled materials than in 3D printed and pressed materials, while hardness values could not be measured in 3D printed materials since they met hardness values below ISO requirements (112, 149). The determined values of flexural strength (149) are not in accordance with the present study (Chapter 3, Figure 2), Still, similar to Berli et al. (149), the present study obtained lower values of surface hardness in 3D printed materials (Chapter 3, Figure 1).

Apart from a cross-linked polyamide material group and a nonacrylic lightpolymerizing resin for additive manufacturing group, all other specimens used in the present study fractured during the flexure testing (Chapter 3, Figure 2). Two conventional materials for occlusal device fabrication and one milled CAD/CAM PMMA material demonstrated equivalent flexural strength values, which is not in accordance with the study by Alp et al. (150). Nevertheless, all the examined materials met ISO requirements for flexural strength testing (65 MPa).

In accordance with ISO 2039-1:2001 (122) surface hardness of occlusal device materials was tested with Brinell's method. Surface hardness describes the density of a material and its resistance to wear, which can affect prosthodontic restorations during the usage. Due to occlusal forces during the functional or parafunctional activities that can be higher than 785 N, the materials' resistance to wear in occlusal device fabrication is imperative (151). In the present study, three conventional cold-polymerized materials and a CAD/CAM (milled) occlusal device material showed the most constant values of surface hardness. On the other hand, nonacrylic light-polymerizing material for additive manufacturing demonstrated the lowest values of surface hardness (149). The values of one milled (Ceramill Splintec) and one 3D printed material (Ortho Rigid) for occlusal device manufacturing from the present study (Chapter 3, Figure 1) are consistent with the hardness values of corresponding materials tested in the study by Reymus et al. (152). Consequently, based on the results of the published studies (149, 152) (Chapter 3, Figure 1) it can be concluded that 3D printed materials for occlusal device fabrication have inferior surface hardness.

Two polyamide materials which were used as a denture base material were tested by Nguyen et al. (143). The authors reported lower values of surface hardness of polyamide materials when compared to PMMA materials (143). Similar results were obtained by Hamanaka et al. (144) and Ayaz et al. (145), which is similar to the present study results (Chapter 3, Figure 1). It follows that polyamide materials for occlusal device fabrication possess lower surface hardness values when compared to PMMA materials.

Huettig et al. (153) investigated the wear resistance and polishability of materials for occlusal device manufacturing (cold-polymerized, milled, and 3D printed). In the study, all the tested materials showed comparable surface polishability and a similar scale of wear (153). In addition, few studies found a connection between wear resistance and surface hardness (95, 98, 109), while some authors are in opposition with that statement (144).

In the present study, two light-polymerized resins (VarseoWax Splint and Ortho Rigid) processed with additive manufacturing and two resins (Ceramill Splintec and CopraDur) processed with milled technology differed significantly (Chapter 3, Figures 1 and 2). Determined diversities between materials are most easily explained with a different chemical structure. The mechanical properties of occlusal devices depend more on the material than specific technology.

At the end of the chapter, several recommendations for future studies are listed:

With today's rapid technology development, the evolution of materials is also expected. For clinical practice it is important to have defined properties and behavior of available materials. Future studies should focus on mechanical and other properties of the newly developed materials for denture bases and occlusal splints.

The behavior of materials in oral conditions were not tested, which is a limitation of this study. Future studies should include different testing conditions (wet vs. dry) and different testing media (water or air).

Due to inaccessibility, not all possible combinations of denture base material and artificial tooth were tested. Future studies ought to include different combinations in order to get better insight of shear bond strength values.

8. CONCLUSIONS

Within the limitations of the present study, the following can be concluded:

- A polyamide resin and nonacrylic light-polymerizing resin for additive manufacturing of occlusal devices have lower surface hardness values, but their flexural strength values are higher when compared to acrylic resin.
- Acrylic resins for occlusal device fabrication show the most consistent values of surface hardness exclusive of the used technology.
- Polyamide and CAD/CAM (milled) materials for denture base manufacturing demonstrate a higher flexural strength compared to heat-polymerized and 3D-printed acrylics.
- The materials for denture base fabrication with the same polymerization type can demonstrate diverse mechanical properties.
- 3D-printed acrylics for a denture base fabrication demonstrate lower mechanical properties compared to the majority of other denture base materials.
- A chosen combination of denture base material and denture teeth material greatly affects shear bond strength values.
- In a shear bond strength test, heat-polymerized resins and CAD/CAM (milled) denture base resins demonstrate mainly cohesive and mixed type of failure compared to cold-polymerized resins, which mostly demonstrate adhesive type of failure.

9. LITERATURE

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10. CURRICULUM VITAE

Vladimir Prpić graduated from the School of Dental Medicine, University of Zagreb, in September 2016. During the 5th and 6th year of undergraduate study he was a student assistant for preclinical and laboratory removable prosthodontics. During that time, he published his articles in students' journals "Sonda" and "Fissura". In 2015 he participated student exchange in Bratislava as a part of the European Visiting Program (EVP). In the academic year 2015/2016 he submitted work for the Rector's Award made at the Department of Removable Prosthodontics, School of Dental Medicine, University of Zagreb, entitled "Influence of Light Conditions and Light Sources on Clinical Measurement of Natural Teeth Color using VITA Easyshade Advance 4,0 ® Spectrophotometer. Pilot Study". Promptly after the graduation, he started a postgraduate doctoral course and began working on a doctoral thesis entitled "The Effect of Technological Manufacturing Process on the Mechanical Performance and Binding Strength of Building Materials of Dentures" supervised by the associate professor Samir Čimić. Since 2016, he has actively attended various domestic and international conferences, such as the 3rd, 4th and 7th International Congress of the School of Dental Medicine, University of Zagreb, and CED-IADR/NOF Oral Health Research Congress held in Wien. Vladimir started his specialization in dental prosthodontics in July 2021, and is currently working as a research assistant at the Department of Fixed Prosthodontics, School of Dental Medicine, University of Zagreb.

List of publications:

Prpić V, Schauperl Z, Glavina D, Ćatić A, Čimić S. Comparison of Shear Bond Strengths of Different Types of Denture Teeth to Different Denture Base Resins. J Adv Prosthodont 2020;12:376–382.

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