

Evaluation of the bond strength of heat-polymerized and auto-polymerized silicone-based soft lining materials to injection-molded, milled, and 3D-printed denture base resins

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1. Introduction

Although modern dentistry aims to preserve natural teeth, the increase in life expectancy and the elderly population means that tooth loss will likely occur in the future [1]. Implant-supported complete dentures have been reported as a more effective treatment option for edentulous patients [2,3]. However, complete dentures remain effective due to anatomical, physiological, and financial constraints [4]. Since the first use of complete dentures, the aim has been to reduce errors during the production process and improve the base materials' properties [5]. Polymethyl methacrylate (PMMA) is today's most widely used denture base material due to its many positive properties, such as biocompatibility, ease of processing, low cost, and low water solubility [6,7]. Denture bases can be fabricated conventionally using compression or injection molding techniques or digitally using subtractive or additive manufacturing techniques [8,9].

The introduction and development of computer-aided technology in the production of complete dentures is expected to overcome the difficulties in traditional complete denture production methods and facilitate the production process [10–12]. Studies have shown that digital complete dentures offer several advantages over conventionally manufactured complete dentures, such as high hydrophilicity, less residual monomer, less microporosity, high color stability, and high adaptability due to the absence of polymerization shrinkage [13–22]. Digital complete dentures offer significant advantages for clinicians and patients, including reduced clinical appointment time and fewer visits [23,24]. Conventional methods for producing dentures typically require at least five appointments, whereas digital methods can be achieved in just two or three visits, depending on the system used [25]. Digital systems can produce a new prosthesis using archived data without needing clinic appointments in case of a broken or lost prosthesis [23,26].

Regardless of the denture fabrication technique, the long-term clinical success of complete dentures depends on several biological and functional factors. Factors such as retention, aesthetics, masticatory performance, stability, and patient comfort with the prostheses are considered important in evaluating the success of complete dentures. Soft lining materials can be used in complete dentures to homogeneously distribute stresses to the supporting tissues and minimize chewing pressure on the alveolar crests [27,28]. Soft liners are either acrylic- or silicone-based and can be heat-polymerized, light-polymerized, or auto-polymerized [29,30]. They can be used for temporary or permanent purposes [30]. One of the main issues encountered with soft liners during clinical use is the separation of the material from the acrylic base over time [9,31]. Soft lining materials are often replaced due to poor bonding strength to the prosthesis base, even if they have good physical properties such as flexibility, color, water absorption, and tear strength [32].

This *in vitro* study evaluated the tensile bond strengths of two different silicone-based soft liner materials, heat-polymerized and auto-polymerized, using prosthetic bases manufactured by injection, milling, and 3D printing. The null hypothesis was that neither the type of base materials nor the type of soft liner materials would affect the adhesive strength of the soft liners to the different denture base polymers.

2. Materials and methods

The base and soft lining materials are listed in Table 1. An *a priori* sample size calculation using G*Power software (Heinrich Heine University, Düsseldorf, Germany) determined that 60 tensile bond strength specimens ($n = 10$) were required. Therefore, 120 denture base specimens ($10 \times 10 \times 20$ mm) were fabricated, with the 10×10 mm surface serving as the bonding area. Every two specimens were joined at this

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Table 1
Materials used.

Brand	Manufacturer	Material Type	LOT
Ivobase High Impact (IHI)	Ivoclar Vivadent AG, Schaan, Liechtenstein	Autopolymerized denture base acrylic resin	LOT VT0765
Ivobase CAD (IC)	Ivoclar Vivadent AG, Schaan, Liechtenstein	CAD-CAM denture base acrylic resin	LOT YB3KKY
Dentabase (DB)	Asiga, Alexandria, NSW, Australia	3D printing denture base acrylic resin	LOT MO/13181
Molloplast B (MpB)	Detax GmbH & Co KG, Ettlingen, Germany	Heat polymerized silicone based soft liner	LOT 240201
Ufi Gel P (UgP)	VOCO GmbH Cuxhaven, Germany	Autopolymerized silicone based soft liner	LOT 2214430

surface by a 3-mm-thick soft liner to form one tensile bond strength specimen.

Milling (IvoBase Cad; Ivoclar Vivadent Inc., Amherst, NY, USA) and printing (DentaBase; Asiga, Alexandria, NSW, Australia) specimens were designed using open-source CAD software (MeshMixer; AutoCAD) and saved in Standard Tessellation Language (STL) format. The design was placed on 98.5-30 mm PMMA discs (IvoBase Cad; Ivoclar Vivadent Inc., Amherst, NY, USA) using the software program (DentalCam 7; vhf camfacture AG, Ammerbuch, Baden-Württemberg, Germany) and milling was performed using a 5-axis milling unit (Vhf K5; vhf camfacture AG, Ammerbuch, Baden-Württemberg, Germany) (Fig. 1).

Specimens were produced using a printer with a 385 nm LED wavelength (MAX UV; Asiga, Alexandria, NSW, Australia). The printer produced the specimens in 99 layers, each with a thickness of 100 μ m, in just 14 min. After production, the specimens were washed twice for 3 min each in isopropyl alcohol to remove excess resin. They were then completely air-dried and placed in a glycerin bath to remove the oxygen-inhibiting layer. The specimens were polymerized for 1 h using the post-cure unit (Asiga Flash; Asiga, Alexandria, NSW, Australia), washed in 37°C water to remove glycerin, and dried according to the manufacturer's recommendations.

For injected (IvoBase High Impact; Ivoclar Vivadent Inc., Amherst, NY, USA) specimens, wax models were specifically prepared 10 $\times$ 10 $\times$ 20 mm and invested in metal flasks. After wax elimination, the flask was opened, and the softened waxes were removed. The specimens were processed using an injection system, following the

manufacturer's instructions.

A 3 mm thick soft liner was applied between the denture base specimens (Figs. 2A and 2B). To prepare specimens with Molloplast B (MpB) (Detax GmbH & Co. KG, Ettlingen, Baden-Württemberg, Germany), an adhesive (Primo Adeziv; Detax GmbH & Co. KG, Ettlingen, Baden-Württemberg, Germany) was carefully applied to the entire acrylic surface for bonding and left to dry for 60 min. The MpB soft liner material was placed in the gaps between the denture bases, and the polymerization process was completed following the manufacturer's instructions. For the specimens to be prepared with Ufi Gel P (UgP) (VOCO GmbH, Cuxhaven, Lower Saxony, Germany), the primer was applied to the acrylic surfaces to adhere and left to dry for approximately 1 min, after which the polymerization was completed following the manufacturer's indirect production method instructions.

All specimens were soaked in distilled water at 37°C for 24 h prior to the tensile strength test. Tensile tests were performed using a universal testing machine (Instron; Norwood, MA, USA) at a test speed of 5 mm/min (Fig. 3). The tensile bond strength values (in MPa) were obtained by dividing the maximum force value (N) recorded prior to failure by the area of the specimen bond surface (mm²) and were calculated for each specimen individually. The failure mode of each specimen was recorded as adhesive, cohesive, or mixed failure.

Scanning electron microscopy (SEM) (Quanta FEG 250; FEI Company, Hillsboro, OR, USA) was used to characterize the morphological features of the adhesive, cohesive, and mixed failure modes. For each failure mode, the specimen with the highest tensile bond strength was selected for SEM examination.

All statistical data were analyzed using statistical software (IBM SPSS V23 and Minitab V14). Compliance with normal distribution was analyzed using the Shapiro-Wilk test and kurtosis-skewness values. Two-way analysis of variance (ANOVA) was employed to assess the tensile bond strength values, with multiple comparisons analyzed using the Tukey Honestly Significant Difference (HSD) test. ($P < 0.05$).

3. Results

A summary of the two-way ANOVA results is presented in Table 2, and the mean tensile bond strength values of all groups are presented in Table 3. Among the denture base materials, a significant difference was observed between the IHI and DB groups. MpB showed significantly higher bond strength than UgP in the IHI and IC groups, whereas no significant difference was observed between the liners in the DB group.

All samples bonded with UgP had an adhesive failure, whereas cohesive failure was observed only in the IHI-MpB group. In the IHI-MpB group, 30% of the failures were cohesive and 70% were mixed. In the IC-MpB group, 60% were adhesive and 40% were mixed. In the DB-MpB group, 70% were adhesive and 30% were mixed. SEM images of all failure modes are shown in Figs. 4A, 4B, and 4C.



Fig. 1. IvoBase CAD PMMA disk after subtractive manufacturing, showing the milled denture base specimens.



Fig. 2A. The specimen with a 3 mm wax section was placed in the dental flasks.



Fig. 2B. 3 mm section removed and ready for soft lining reconnection.

4. Discussion

Two-way analysis of variance (ANOVA) performed in this study revealed that both the type of denture base material and the type of soft liner material had a statistically significant effect on the adhesion strength of soft liners to different denture base polymers. The effect of denture base material was found to be significant ($p = 0.001$). Similarly, the effect of soft liner material was also significant ($p < 0.001$). In addition, the interaction between denture base and soft liner material was found to be significant ($p < 0.001$). These findings indicate that both the main effects and the interaction effect affected the adhesion strength and support the rejection of the initially proposed null hypothesis.

Tensile, peel, and shear strength tests can be applied to evaluate the bonding of soft liner materials with denture base materials. Wright reported that the peel test was the most effective method for assessing the adhesion of soft liner materials [28]. Kutay, on the other hand, reported that the adhesion strength of silicone-based soft liner materials can be best determined by tensile strength test since they have lower tear resistance than other soft liner types [31]. In the shear test, stresses are assumed to be concentrated at the edges of the liner material, complicating the interpretation of bond strength and failure mode results. The most commonly used test in the literature is the tensile strength test. Özdemir et al. explained the reason for this as follows: the tensile test

provides more reliable results and is easier to apply [29]. Tensile testing applies a static, slow force to measure the bond strength between the base and soft liner material. In this study, tensile tests were performed.

When the tensile strength test findings were evaluated, statistically significant differences were observed between the mean values of the tensile strength (MPa) within the groups ($p < 0.05$). The highest mean value was observed for the IHI-MpB group (1.57 MPa). In contrast, the lowest value was observed for the IC-UgP group (0.39 MPa). It has been reported that the bond strength with the denture base material should be a minimum of 0.44 MPa for soft liner materials to be considered suitable for clinical use [33]. Accordingly, the mean tensile strength values of all groups bonded with MpB were found to be higher than the clinically acceptable bond strength value. Among the groups bonded with UgP, the mean tensile bond strength values of the IHI and DB groups exceeded the clinically acceptable bond strength value, whereas that of the IC group (0.39 MPa) was below this value. Looking at the literature, Choi et al. found this value to be 0.36 MPa and reported that it was not suitable for clinical use, similar to our findings [9]. They explained that silica and aluminum added to the IC base to compensate for thermal

Table 2
Summary of results of two-way ANOVA.

Source of Variation	Degrees of Freedom	Mean Square	F-ratio	P
Denture Base	2	5403.400	8.550	0.001*
Soft Liner	1	86310.500	136.530	< 0.001*
Denture Base x Soft Liner	2	9207.800	14.560	< 0.001*

* Statistically significant at $p < 0.05$.

Table 3
Tensile bond strength values (mean $\pm$ standard deviation) of all groups.

	MpB	UgP
IHI	1.57 $\pm$ 0.40	0.72 $\pm$ 0.13
IC	1.53 $\pm$ 0.19	0.39 $\pm$ 0.12*
DB	0.96 $\pm$ 0.35*	0.67 $\pm$ 0.19

IHI, Ivobase High Impact; IC, Ivobase Cad; DB, Dentabase; MpB, Molloplast B; UgP, Ufigel P. *Significant difference within group of same column ($p < 0.05$), 2-way ANOVA with Tukey post hoc.

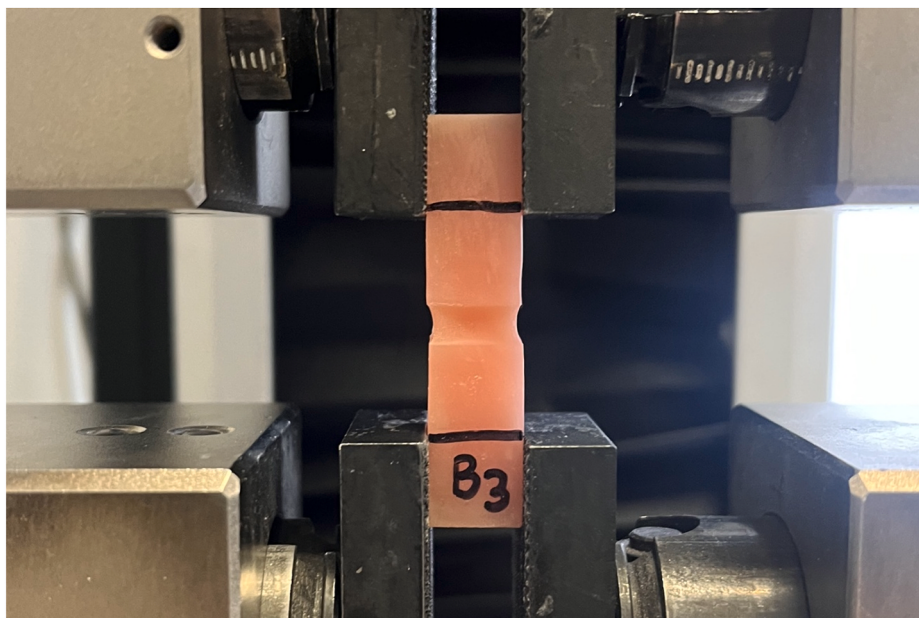


Fig. 3. Tensile bond strength testing of the denture base-soft liner specimen using a universal testing machine.

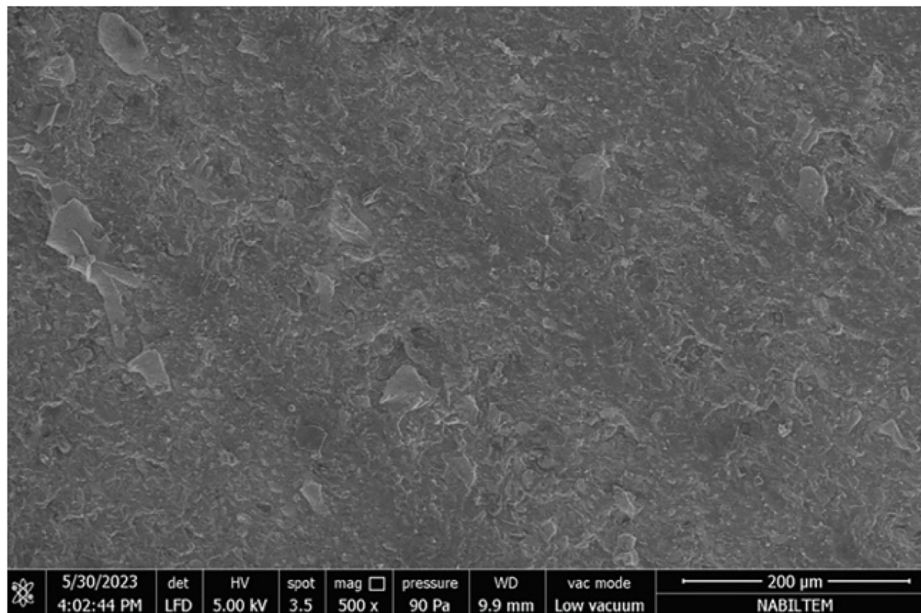


Fig. 4A. Scanning electron microscope images showing the highest tensile strength in the groups with adhesive, cohesive and mixed failure. Original magnification x500.

Ivobase CAD relined with Molloplast B showing adhesive failure. The soft liner was detached from the denture base, exposing a wavy and generally regular base surface with residual bonding material in some areas.

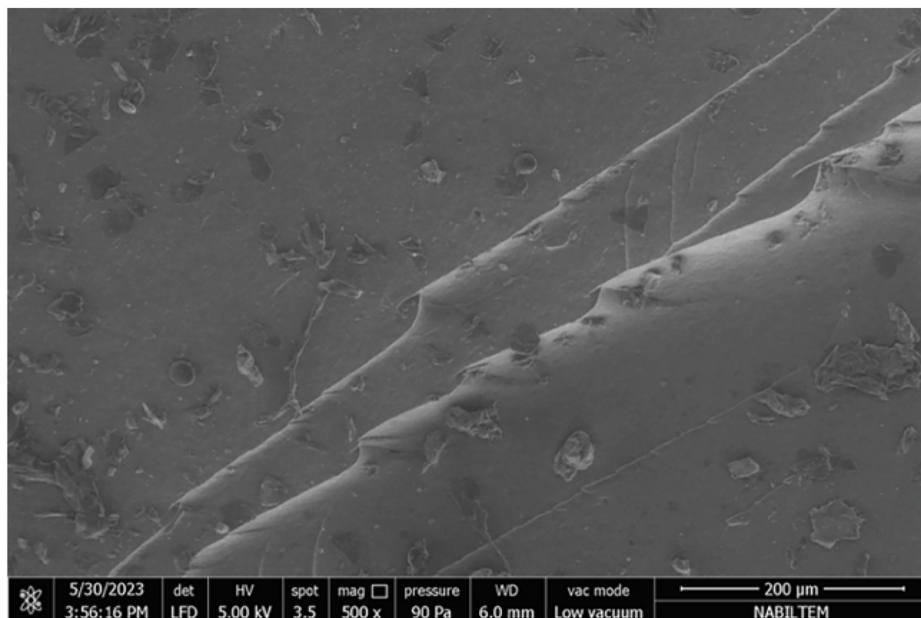


Fig. 4B. Scanning electron microscope images showing the highest tensile strength in the groups with adhesive, cohesive and mixed failure. Original magnification x500.

Ivobase High Impact relined with Molloplast B showing cohesive failure. The denture base surface remained covered by soft liner material, with strip-shaped tears and folds indicating fracture within the liner.

stresses during the milling process may have affected adhesion to soft lining materials, leading to lower tensile strength values [9]. Wemken et al. reported this value as 1.3 MPa, and this value, which is different from our findings, is above the minimum value reported for clinical use [26].

MpB was observed to adhere more strongly to IHI and IC bases than to UgP ($p < 0.05$), whereas no significant difference was observed with the DB base ($p > 0.05$). Both are silicone-based materials. However, MpB hardens with heat, whereas UgP hardens at room temperature. As

silicone is already a polymer, it hardens via a cross-linking process instead of a polymerization process, and this cross-linking can be achieved with heat using benzoyl peroxide or at room temperature using tetraethylsilicate. PMMA denture base resins and silicone-based soft lining materials have different molecular structures and do not allow chemical bonding. The bond between the two materials is micro-mechanical. An alkyl-silane coupling agent (adhesive) or silicone polymer dissolved in a solvent (primer) is used to improve the bond [30]. Primers are more commonly used in the direct technique (chair-side

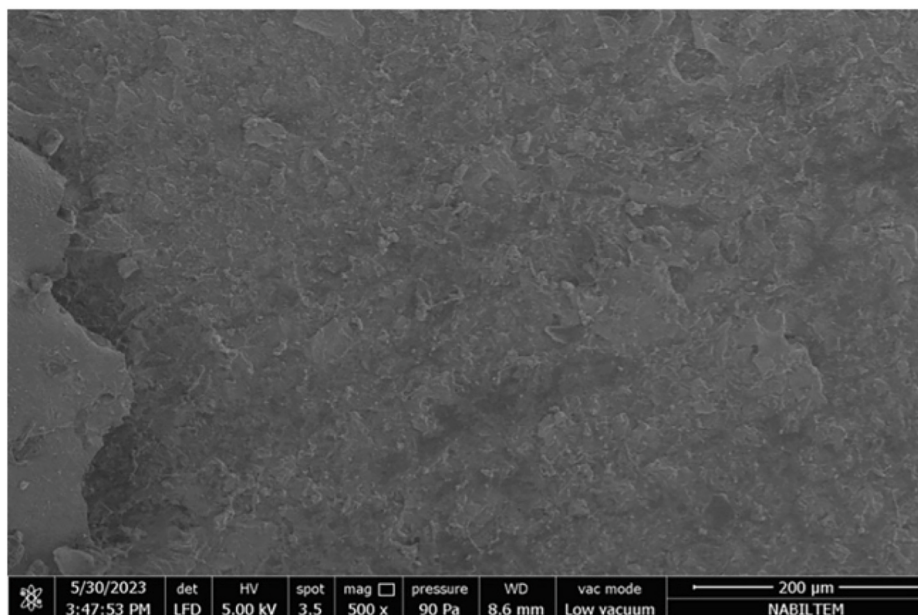


Fig. 4C. Scanning electron microscope images showing the highest tensile strength in the groups with adhesive, cohesive and mixed failure. Original magnification x500.

Ivobase High Impact relined with Molloplast B showing mixed failure. Both exposed denture base and residual soft liner material were observed, with a widespread, generally regular structure and partially stripped areas, indicating combined adhesive and cohesive failure.

application). In contrast, adhesives are used in the indirect technique (laboratory application) because of the long drying time [34]. The difference in bonding between MpB and UgP is thought to be due to this. Primo adhesive used with MpB contains methacryloxypyril-trimethoxysilane, an organosilane commonly used in dentistry. The methacrylate end of the adhesive bonds to the methacrylate groups suspended on the surface of the denture base, and the reactive end bonds to the soft lining material. The primer used with UgP consisted of a reactive polymer, special silane, and 2-butanone as the solvent. The reactive polymer contained hydrocarbon chains and reactive Si-H- and vinyl groups. The solvent in the primer dissolves the upper layers of the denture base. After the solvent evaporated, the hydrocarbon chains bonded to the denture base, whereas the reactive groups bonded to the soft lining material. Unlike UgP, MpB adheres to the denture via the methacrylate group on the silane (A 174) [35].

Many studies have shown that autopolymerized soft liner materials exhibit lower bonding to PMMA bases than heat-polymerized soft liner materials. Awad et al. evaluated the bonding of 5 different soft liner materials to conventional, milling, and 3D printing bases. They reported that autopolymerised soft liners showed the lowest bonding value to all bases [24]. A similar study was conducted by Alfaraj et al., and it was reported that the autopolymerised group showed the lowest values [12]. In their study, Köseoğlu et al. concluded that there was no significant difference between heat-polymerized and autopolymerised groups, which differs from our findings [22].

MpB had a significantly lower bond strength in the DB base than in the IHI and IC bases ($p < 0.05$). It is thought that different amounts of suspended methacrylate groups in different denture bases affect the bond strength. At the same time, no significant difference in bond strength was observed between IHI-MpB and IC-MpB. However, cohesive failure was observed in the IHI-MpB group. When the failure mode is cohesive, a force higher than the tensile strength of the soft liner is assumed to be measured; however, this provides less meaningful information about the adhesive bond strength. All specimens in the UgP group showed adhesive failure. A silicone liner has no plasticizer but does contain a filler [36]. The filler absorbs water in the material and increases its softness, thus increasing its elasticity and decreasing its bond strength [9].

One limitation of this study is that the test conditions did not fully simulate the clinical situation. The specimens were stored in water for only 24 h before testing, and thermocycling, long-term ageing, and cyclic loading were not performed. Therefore, the findings represent short-term bond strength and may not reflect the long-term clinical performance of the materials. Factors such as the processing method, water absorption, hardness, tear resistance, color stability, and *Candida* adherence are properties of soft liner materials that should be investigated to better predict their clinical performance.

5. Conclusions

- 1) The highest tensile value was observed in the IHI-MpB group.
- 2) The lowest tensile strength was observed for the IC-UgP group. This value (0.39 MPa) is below the minimum value required for clinical use (0.44 MPa).
- 3) MpB showed higher tensile strength than UgP in IHI and IC bases, with no significant difference observed in the DB base.
- 4) Failure modes varied among the Molloplast B groups, whereas all Ufi Gel P specimens exhibited adhesive failure.

CRediT authorship contribution statement

Cagla Nur Gedikli: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Tayfun Bilgin:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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