Reparability of UDMA resin 3D printed for interim dental prostheses.

Thesis

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#### Abstract

**Purpose:** To evaluate the in vitro effects of the 3D printed Urethane dimethacrylate (3D-UDMA) resin flexural strength (FS) once repaired through different surface treatments and repair materials. Methods: 20 milled Poly-methylmethacrylate (mPMMA) and 60 3D-UDMA 2 x 2 x 25 mm rectangular blocks are divided randomly into 8 different groups according to surface treatment and repair material (PmP: mPMMA + monomer + selfcured PMMA; UvP: UDMA + Visio.Link + self-cured PMMA; UvC: UDMA + Visio.Link + flowable composite; UeC: UDMA + etch & bond + flowable composite), and if they were thermocycled or not (PmPt; UvPt; UvCt; UeCt). FS was measured using a universal testing machine at 1 mm/minute. Failure (adhesive, cohesive within the original material, cohesive within the repair material, or mixed) was assessed under x3.5 magnification. FS data was analyzed using 2-way-ANOVA (a = 0.05). Results: For the FS within the non-thermocycled samples, there was a statistically significant difference between UvP and all other 3 groups. Within the thermocycled samples, there was only a statistically significant difference between UvPt and UeCt. For the mode of failure within the non-thermocycled there was a statistically significant difference between UeC and UvP and the control repair (PmP). For the mode of failure within the thermocycled samples, there was only a statistically significant difference between UvCt and PmPt. Conclusion: UDMA repaired with Visio.Link primer and self-cure PMMA had statistically significant lower FS compared to all other repairs. The repairs with highest FS were achieved with flowable composite repairs.

# Dedication

This Master's Thesis is dedicated to my loving parents Carmen Demetrio and Javier Ibarra, who have been my foundation and guide for my 12 years of dental training, my sister Claudia, for her encouragement to keep pursuing my dreams, and to my fiancé, Edward Zimmer, for his everyday support through residency.

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Fields of Study

Major Field: Dentistry

Specialty: Advanced Prosthodontics

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#### **Chapter 1. Introduction**

#### **Provisional Restorations**

Interim restorations are a crucial component in the success of dental prostheses of a single or multi-unit, over teeth or implants.<sup>1</sup> These provisional restorations will guide the clinician in regards esthetics, phonetics, occlusion, as well as protecting the biologic components of the oral cavity and should serve as a prototype of the final prosthesis.<sup>2–4</sup>

Although they are meant to serve for a limited period of time, Burns et al.<sup>5</sup> and Yuodelis et al<sup>6</sup> describe several desirable properties for interim restorations. These include good marginal adaptation, retention and resistance form, stable dimensions, strength, comfort, esthetic, preservation of physiology, physiologic occlusion, ease of fabrication, ease of removal and cementation by the dentist, low cost and biocompatible.

Despite the fact that a great part of dentistry calls for direct fabrication of provisional restorations, there are many cases where these provisional prostheses are full arch or full mouth restorations in advance prosthodontics. These are more elaborate in their construction and will be in function for a longer period of time. This is true especially in situations of changes in vertical dimension in full oral rehabilitation, long-span fixed prostheses, temporomandibular joint dysfunction therapies or patients that exhibit parafunctional habits.<sup>3</sup>

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In these more complex cases, the clinician will most likely choose an indirect fabrication technique. Furthermore, it becomes critical to avoid fracture of the interim prosthesis, as it may result in unplanned appointments and could negatively affect the outcome of the fixed prosthodontic procedure<sup>7</sup> as well as the patient-doctor relationship.

In order to facilitate the fabrication of these complex interim prostheses, there has been an increase of use of computer-aided design/computer aided manufacturing (CAD/CAM) technology, CAD allows the clinician to digitally design the prosthesis and the CAM process utilized either subtractive (milling from a puck of material) or additive methods (such as 3D printing), reducing as well the total time of fabrication.<sup>8</sup> Beyond the potential for repairing fractures, being able to modify these prostheses by subtracting and/or adding material during the treatment is an important factor to contemplate when choosing the provisional material to use to achieve ideal design of the final prosthesis.<sup>4</sup>

## Materials for provisional restorations

Materials used for the fabrication of interim restorations must satisfy biological, esthetic, and functional needs. Strength is probably one of the most relevant physical properties<sup>9,10</sup> contributing to clinical efficiency, as well as other characteristics such as ease of fabrication and low cost as mentioned previously.<sup>11</sup> The knowledge of the physical properties of each interim prosthesis is key consideration in each clinical case.<sup>7,12</sup> Flexural strength (FS), also known as moduli of rupture, is the stress necessary to cause a fracture under a static load.<sup>13</sup> It is important to highlight that FS is a complex measurement that is a combination of both tensile and compressive strength. The ANSI/ADA and ISO standards for resin based crown and bridge materials both dictate that the minimum FS of these materials shall be no less than 50 MPa.<sup>14,15</sup>

Most interim prosthesis are made of dental polymers, which are formed through chemical reactions that convert large numbers of low-molecular-weight molecules, known as monomers, into large, very high-molecular-weight long-chain macromolecules.<sup>11</sup>

One of the classifications of these materials is based on their composition:<sup>5,13</sup>

- Unfilled copolymers of methacrylates (mixed with a liquid monomer) such as:
  - o ethyl methacrylate (PEMA),
  - o methyl methacrylate (PMMA), or
  - o vinyl methacrylate (PVEMA), and
- Microfilled composite (bis-acryl paste formulations) materials such as:
  - o bisphenol A-glycidyl methacrylate (bis-GMA),
  - o triethyleneglycol dimethacrylate (TEGDMA) or
  - urethane dimethacrylates (UDMA).<sup>5,13</sup>

However other authors<sup>16,17</sup> tend to extend this classification into four groups:

- Polymethyl methacrylate,
- Polyethyl or butyl methacrylate,
- Microfilled bisphenol A-glycidyl dimethacrylate (bis-GMA) composite resin, and
- Urethane dimethacrylate (light-polymerizing resins).

Table 1 presents some examples of these dental materials for interim restorations, and their classification.

Material Classification	Product Name	Manufacturer
Ethyl methacrylate	Splintline	Lang Dental, Wheeling, III
Methyl methacrylate	Alike	GC America, Alsip III
Vinyl ethyl methacrylate	Snap	Parkell, Farmington, NY
Butyl methacrylate	TempPlus	Ellman Int, Hewlett, NY
Bis-acryl composite (auto- polymerized)	Intergrity	Lang Dental, wheeling, III
Bis-acryl composite (dual- polymerized)	IsoTemp	3M Dental, St. Paul, Minn.
Urethane dimethacrylate composite (visible light- polymerized)	Triad	Dentsply Int, York, Pa.

Table 1. Interim restoration materials classification and product examples.

A different classification of polymer-based crown and bridge materials comes from the ISO 10477<sup>15</sup> which is based on their activation system for polymerization:

**Type 1:** Polymerization started mixing initiator(s) and activator(s) which are commonly known as "self-cured" resins.

**Type 2:** Polymerization starts when an external source is applied such as heat and/or light or UV radiation. These "external-energy-activated" materials are subdivided into two classes.

Class 1: polymer-based materials without a light or UV-sensitive initiator. Class 2: polymer-based materials with a light or UV-sensitive initiator.

Type 3: Hybrid from type 1 and 2. Commonly known as "dual-cured" materials.

## Monomethacrylates

The chemical polymerization reaction of monomethacrylates will have their foundation on the breaking the C=C double bond of the monomer molecule, creating active free radicals with the potential of propagating the polymer chain growth. This prorogation is achieved by the active free radical breaking other double bonds and generating new free radicals. These polymers will create chain entanglements of individual polymer chains, however these linear chain configuration do not create any cross linking between each other.<sup>11</sup>



Figure 1. Methylmethacrylate molecule.

Poly(methylmethacrylate) (PMMA) (Figure 1)<sup>11</sup> is a frequently used acrylic thermoplastic material that is the product of the polymerization of the monomer methylmethacrylate (MMA).<sup>11</sup> PMMA based provisional restorations are one of the most widely used in dentistry<sup>3,4</sup> due to its chemical and mechanical properties such as a flexural strength (FS) of 60 MPa<sup>11</sup> or higher. PMMA's advantages also include ease of handling, good toughness, wear-resistance, it is able to be pigmented to a lifelike appearance, able to be sterilized, easily cleaned, biologically safe, and very durable.<sup>11</sup>

While PMMA was generally considered the gold standard for interim prostheses, it does possess disadvantages and its clinical use continues to decline compared to newer materials such as bis-acrylics.<sup>7</sup> One of the most known disadvantage is the greatly exothermic reaction, that might not only affect the tooth's vitality, but also impacts its polymerization volumetric change,<sup>4</sup> which is a shrinkage of around 21%.<sup>18</sup> Another

disadvantage is the ease of fabrication, as well as a possible allergic reaction to the MMA monomer liquid.<sup>4</sup>

Fortunately, most of these disadvantages, except for higher cost, are compensated with subtractive manufacturing method of milling the restoration from a prefabricated PMMA puck. Although, these prefabricated acrylic blocks are polymerized with a high degree of conversion, which result in better physical properties, there are instances where the size of the block is the limiting factor when designing a prosthesis.<sup>8</sup> In these cases the alternative option is relying in materials to be fabricated with additive methods, such us 3-D printed UDMA resin.

## **Dimethacrylates**

The chemical polymerization reaction of dimethacrylates will also have their foundation on the breaking the C=C double bond of the monomer molecule, creating active free radicals with the potential of propagating the polymer chain growth. In this case however, compared to the monomethacrylates, the dimethacrylates polymers will create chain entanglements of individual polymer chains that will later generate branched polymer configurations that will result in cross linking.<sup>11</sup> These crosslinks are chemical bonds that require high energy to break, therefore these polymers will have higher strength properties than the ones with linear configurations, however at the same time it makes them more brittle.<sup>7,11,12</sup>

Dimethacrylates are formulated to produce materials with slightly different properties including the unpolymerized resin viscosity which it is directly influenced by their degree of conversion. UDMA based resins (Figure 2)<sup>11</sup> have been created seeking to reduce this unpolymerized resin viscosity compared to other dimethacrylates which results in a higher degree of conversion. As a class, UDMA base resins encompass any monomer containing one or more urethane groups and two methacrylate end groups.<sup>11</sup>



Figure 2. UDMA molecule

Despite their disadvantages, studies have shown that PMMA resins have better physical properties than 3D printed UDMA,<sup>19</sup> especially after being subjected to environmental changes as saliva or other mediums. Gujjari et al.<sup>20</sup> found that after subjecting PMMA and bis-acrylic samples to different dietary beverages, PMMA had better color stability, and had no effect on its FS, compared to the ProTemp samples (a dimethacrylate resin based material).

#### Repairs. Surface treatment, primers, bonding agents and materials

As mentioned previously, provisional prostheses are a crucial element in the dental treatment, and it is important to preserve their integrity throughout the treatment. Unfortunately, there are occasions where dentists encounter fractures of these prostheses, and the clinician has to decide on whether to repair or to replace them. In complex prosthodontic cases like the ones mentioned earlier, due to their elaborate fabrication and cost, clinicians often opt for repairs.<sup>8,10</sup> In view of this, this study will try to guide clinicians towards the repair protocol least likely to break again.

Despite their advantages, bis-acryl materials have poor repairability properties,<sup>4</sup> even recommending the remake rather than repair of the interim prostheses due to the FS of the repair material being reduced to less than 50%.<sup>10</sup> In dentistry, the dimethacrylates are often fabricated with fillers, which will generate changes on the surface energy of the material. Materials with similar surface energy bond better, therefore it is likely that UDMA repaired with PMMA does not have the same results as UDMA repaired with a filled composite resin material.<sup>21</sup> This is one of the reasons why it is important to determine appropriate repair protocols for UDMA based provisionals.

Historically, PMMA prostheses has been repaired with PMMA due to its clinical success. Chemically, treating the repair surface with the liquid methyl methacrylate monomer to soften and swell the old resin, allows for new PMMA polymers chains to be added and facilitate bonding of the repair material.<sup>11</sup> However as mentioned before,

studies and clinical experience has shown that bis-acryl paste formulations are not able to be repaired in the same way with the same original material. Fortunately, resent studies have demonstrated good results of repairs with composite resins, different surfaces treatments, primers and bonding agents.<sup>22</sup>

One of the classifications of resin composites is by their manipulation characteristics, classified as Flowable or Condensable-packable composites. A modification of the small-particle composite and hybrid composite results in the flowable composites.<sup>23</sup>

Vega-Goncalves et al.<sup>24</sup> study found that the shear bond strength of repaired dental polymers (PMMA and Bis-acryl) was statistically increased when repairs were done utilizing composite primer, as well as utilizing flowable composite (conventional dimethacrylate-based composite)<sup>25</sup>, compared with packable composite and no primer.

The pre-treatment of the repairing surfaces with mechanical surface treatment such as aluminum oxide-blasting, or chemical agents such as primers and bonding agents become critical when repairing dental prostheses, as studies show that repairs without it resulted in adhesive failures or reduction of flexural strength (FS).<sup>26,27</sup> A primer is a hydrophilic, low-viscosity resin that promotes bonding to an adherend substrate. They are solutions containing hydrophilic monomers dissolved in a solvent such as acetone, ethanol, or water.<sup>21</sup>

Usually, bonding agents are fabricated by combining different dimethacrylates with diluting monomers to control viscosity and to enhance wettability. They have no potential for adhesion, but they improve micromechanical bonding.<sup>21</sup>

Visio.Link composite-acrylic primer (Bredent GmbH & Co.KG)<sup>28</sup> is a combination of methyl methacrylate monomer, pentaerythritol tri-acrylate and pentaerythritol tetra-acrylate. This primer was fabricated in aims to create adhesive bonding of composite to plastics such as composite resins, PMMA materials and high-performance polymers.<sup>28</sup>

There are 3 modes of fractures that are utilized to evaluate a failure surface: cohesive, adhesive and mixed.<sup>29</sup> For this study, fractures were classified as cohesive within the original material (O), cohesive within the repair material (R), mixed fractures that involved both repair and original materials (M), and finally, adhesive fractures (A) which would involve a line fracture right in the junction of both materials and that it would mean a failure in the bond of the material. The location of fracture was statistically analyzed and related to the FS of each repair protocol through a logistic regression analysis, hoping to guide the clinician on predicting where a new fracture would occur if repairing with one protocol over another.

## Objective

Based on the above review, there is a lack of studies testing the repairs of 3D printed UDMA, which would guide the clinician into understanding if the repair material and/or the bond between the repair and interim material utilized has adequate strength.

Driven by the needs of prosthodontist specialists regarding long-term indirect fabricated interim prosthesis, the purpose of this study was to evaluate the in vitro effects of the 3D UDMA resin flexural strength (FS) once repaired through different surface treatments and repair materials. This would ultimately determine what repair protocol is the most effective for 3D printed UDMA resin, assessed by determining which repair protocol sample exhibits greatest flexural strength, as well as assessing if the sample experienced a cohesive, adhesive, or mixed fracture. Since the clinical gold standard interim prosthesis material is PMMA, milled PMMA (m-PMMA) repaired with selfcured PMMA was included as the control repaired sample.

# Hypotheses

The hypothesis of this study is that 3D printed UDMA primed with Visio-Link and repaired with self-cured PMMA will have the highest FS and will have the least amount of adhesive interface failure.

There are two additional sub-hypotheses in this study:

A null hypothesis of this study is that the repair protocol will not be affected by the thermocycling. The second null hypothesis is that the flexural strength is not correlated to the mode of failure.

#### **Chapter 2. Materials and Methods**

A total of 5 different materials were utilized. For the samples 3D printed UDMA (FormLabs)<sup>30</sup> blocks were fabricated as well as milled PMMA (ZirluxTemp – Henry Shein)<sup>31</sup> blocks, used as the controlled group. For the different surface treatments acrylic monomer (Alike liquid), 35% phosphoric acid (Ultra-Etch) and resin bond (G-aenial Bond – GC America), and acrylic-composite primer (Visio.Link – Bredent) were utilized. Finally, two different materials were used as repair materials; cold-cure PMMA (Alike), and flowable composite (G-aenial Universal Flo – GC America)<sup>32</sup>. FS of each sample and repair material (mPMMA<sup>31</sup>, 3D-UDMA<sup>30</sup> and flowable composite)<sup>32</sup> was gathered from their manufacturer's information, except for self-cured PMMA, as self-cured acrylic's FS will vary from dentist to dentist and mix to mix as Alp et al.<sup>33</sup> describe in their study (Table 2).

Table 2. Sample and repair materials' Flexural Strength.

	mPMMA	Self-cure PMMA	3D-UDMA	Flowable Composite
FS	100 MPa	~65 MPa	>50 MPa	167 MPa

20 mPMMA samples were milled from one single PMMA puck, and 60 3D-UDMA samples were printed utilizing FormLabs From 2 printer in a horizontal orientation (Figure 3), as Alharbi et al.<sup>34</sup> found in their study that the specimens that were printed perpendicular to the load direction had significantly higher compressive strength than in a parallel direction.



Figure 3. Milled PMMa and 3D-printed UDMA samples.

All sample sizes were made in accordance of flexural strength testing of resinbased and polymer-based crown and bridge materials of ISO no.  $10477^{15}$  and ANSI/ADA no. 27 (Figure 4)<sup>14</sup>, with dimensions of 2 x 2 x 25 mm.



Figure 4. Sample Dimensions

Both mPMMA and 3D-UDMA samples were divided randomly into two treatment groups: thermocycled and not. Gale et al. mention that there is no concrete evidence that failures occur as a result of thermal stresses, however if the intention of the test is not to determine material serviceability but to investigate the mode of failure which is the case with this study, then thermocycling would be appropriate.<sup>35</sup> The samples to be thermocycled were put into metal boxes (Figure 5) and later put through standard cyclic regimen alternating between 55°C and 5°C for 10,000 cycles to mimic 1 year of physiologic wear in the oral cavity, on a thermocycler machine with cycle periods of 60 seconds. Following 24 hours, surface roughness was standardized (0.16 µm) with aluminum-oxide grit blasting.<sup>24,27</sup> Ultimately, groups were divided randomly within each sample material, and named as shown on Table 3.



Figure 5. Four metal boxes for thermocycling. Box At had the m-PMMA samples.

Group	Sample	Thermocycled	Surface	Repair	Number of
Acronym	material		treatment	material	samples (n)
PmP	m-PMMA	No	Monomer	РММА	10
PmP <i>t</i>	m-PMMA	Yes	Monomer	РММА	10
UvP	3D-UDMA	No	Visio.Link	РММА	10
UvP <i>t</i>	3D-UDMA	Yes	Visio.Link	РММА	10
UvC	3D-UDMA	No	Visio.Link	Flowable Composite	10
UvCt	3D-UDMA	Yes	Visio.Link	Flowable Composite	10
UeC	3D-UDMA	No	Etch + Bond	Flowable Composite	10
UeC <i>t</i>	3D-UDMA	Yes	Etch + Bond	Flowable Composite	10

Table 3. Testing groups, their acronyms, and description.

Blocks then were cut into two sections with a diamond disc, in the following fashion: a 1 x 2 mm section in the middle portion was measured. From this middle section, 2 x 2 mm cubes on each side were measured to later draw a 45° diagonal through these squares. Ultimately these lines allowed for cuts on each sample to achieve a parallelogram space of 3 x 5.65 mm, as shown on Figure. 6. This protocol of sample cut was designed and adapted from Singh et al.<sup>1</sup>



Figure 6. Sample cut protocol

The middle space on the metal jig which would receive the repair material was designed to have two equal opposing angles, this way when applying the 3-point-bend test the repair material and bonding surface to the original material would be under both tension on one side and compression on the other, following the analysis of FS, measurement that is a combination of both tensile and compressive strength. This would be important when analyzing the mode of fracture.

Surfaces were treated and repaired according to their group on a metal jig (Table 3):

**PmP:** Bonding surfaces were primed with monomer liquid with a microbrush. Prepared cold-cured PMMA on the vibrating machine and put into a monojet syringe, then injected into the metal mold. Once fully polymerized, excess cold-cure acrylic was eliminated with 1000 grit sandpaper.

**UvP:** Bonding surfaces were primed with Visio.Link with a microbrush, airbrushed to remove excess and light-cured. Prepared cold-cured PMMA as with PmP on the vibrating machine and put into a monojet syringe, then injected into the metal mold. Once fully polymerized, excess cold-cure acrylic was eliminated with 1000 grit sandpaper.

**UvC:** Bonding surfaces were primed with Visio.Link with a microbrush, airbrushed to remove excess, and photo-polymerized. Flowable composite was injected into the metal mold and photo-polymerized. Excess composite was eliminated with 1000 grit sandpaper.

**UeC:** Bonding surfaces were primed with hydrofluoric acid etchant and resin bond was applied with a microbrush, airbrushed to remove excess, and photo-polymerized. Flowable composite was injected into the metal mold, and photo-polymerized. Excess composite was eliminated with 1000 grit sandpaper.

After measuring each sample's exact length, width and thickness, their FS was tested with the 3-point-bend test (Figure 7) in a universal testing machine with a crosshead speed of 1 mm/minute, under a 1kN static load cell. FS data was collected from Universal Testing Machine into a statistics software (Figure 8). Results were reported as average +/- standard deviation, and *p*-values depicted as \* for p<0.05.<sup>19</sup> All fractured samples were preserved and organized in a sheet to later assess failure type (adhesive, cohesive or mixed) assessed under x3.5 magnification (Figure 9 and 10).



Figure 7. 3-Point-Bending test for FS.



Figure 8. Universal Testing Machine.

St	AMPLES	Teste	D.
A	6	Au	u
2.	8	2.	Remained A.
1	9		9
(B) 1 344		Ct.	
2	6 2	2	*
3 3	8		
5	10.	5	10
0	le Press	4	6
2	3	2 <u></u>	1
4	9		
5	10	Pt	6
1	6	2	
3	s		
5	10.		a

Figure 9. Fractured samples preserved for fracture location analysis under x3.5 magnification.



Figure 10. (a) Mode of failure diagram. O: cohesive failure within de original material, R: cohesive failure within de repair material, M: mixed failure, A: adhesive failure. (b) Example of O mode of failure. (c) Example of R mode of failure.

## **Statistical Analysis**

FS,  $\sigma_B$ , was calculated from the following equation:<sup>15</sup>

$$\sigma_{\rm B} = \frac{3Fl}{2bh^2}$$

where:

*F* is the maximum applied load, in Newtons;

*l* is the distance, in millimeters, between the supports, i.e. 20 mm;

*b* is the width of the test specimen, in millimeters;

*h* is the height of the specimen, in millimeters.

FS analysis was done with two-way-analysis of variance using a log-normal response distribution and restricted maximum likelihood estimation technique. If a significant effect was found, then a pairwise comparison within groups for thermal cycled, non-thermal cycled, and between thermal cycled and non-thermal cycled within the same repair group, a pairwise analysis would be done using a Bonferroni corrected Student's t-tests.

For the Mode of Failure analysis (Figure 10), the data was ranked according to how much of the failure was in the repair material. When the failure was within the original material, it had a rank of 0, when the failure was partially on the original material and partially in the repair material a rank of 1 was given, and finally, when the failure was within the repair material, a rank of 2 was given to the sample. Once this data was ranked, it was subjected to the non-parametric 1 way ANOVA Kruskal Wallis test with subsequent pairwise comparisons using Bonferroni-corrected Wilcoxon Two-Sample test.

To determine where a statistically significant difference was both for the FS and Mode of failure, the Stepdown Bonferroni test was be applied to different repairs within the non- thermal cycled, to different repairs within the thermal cycled, and finally to the same groups thermal cycled and non-thermal cycled.

Finally, for the correlation of mode of fracture, using rank data according to how much of the failure was in the repair material, to the FS of the repaired samples, a logistic regression analysis was performed.

# **Chapter 3. Results**

The repaired sample with greatest FS was UvC with 69.35 MPa, followed by UeCt with 69.22 MPa and UeC with 63.88 MPa (Table 4). The means and 95% confidence intervals are given in Figure 11.

Non-Thermal cycled	FS (MPa)	Thermal cycled	FS (MPa)
Repair Group		Repair Group	
PmP	58.65	PmPt	59.24
UvP	33.48	UvPt	45.28
UvC	69.35	UvCt	56.74
UeC	63.88	UeCt	69.22

Table 4. Means of flexural strength (FS) of repaired samples in MPa.



Figure 11. Means and 95% confidence intervals of the Flexural Strength of the test groups.

# FS between non-thermocycled groups

There was a significant difference between: PmP & UvP: a=0.0002; UvC & UvP: a<0.0001; UvC & UvP: a<0.0001 (Table 5). Between thermocycled groups, there was a significant difference between: UeCt & UvPt: a=0.0455 (Table 6).

Repair group 1	Repair group 2	Bonferroni <i>p</i> -value	Statistical Significance
PmP	UeC	1.0000	No
PmP	UvC	1.0000	No
PmP	UvP	0.0002*	Yes
UeC	UvC	1.0000	No
UeC	UvP	<0.0001*	Yes
UvC	UvP	<0.0001*	Yes

Table 5. Pairwise comparisons of FS means between the non-thermocycled groups.

Table 6. Pairwise comparisons of FS means between the thermocycled groups.

Repair group 1	Repair group 2	Bonferroni <i>p</i> -value	Statistical Significance
PmPt	UeCt	1.0000	No
PmPt	UvCt	1.0000	No
PmPt	UvPt	0.4764	No
UeCt	UvCt	1.0000	No
UeCt	UvPt	0.0455*	Yes
UvCt	UvPt	1.0000	No

# FS between thermocycled and non-thermocycled groups

There was no statistically significant difference between same repair protocol before or after thermocycling process (Table 7).

Non-thermal cycled	Thermal cycled	Bonferroni p-value	Statistical
repair group	repair group		significance
PmP	PmPt	1.0000	No
UeC	UeCt	1.0000	No
UvC	UvCt	0.6070	No
UvP	UvPt	0.2255	No

Table 7. Pairwise comparisons of FS means between thermal cycled and not of the same repair.

From our control group (PmP/PmPt) 95% of the mPMMA samples repaired with cold-cured PMMA fractured within the repair material. Of the 3D-UDMA samples, 56.67% fractured within the original material (34/60), 38.3% fractured within the repair material (23/60), and 5% had mixed fractures (3/60) (Table 8).

	PmP	PmPt	UvP	UvPt	UvC	UvCt	UeC	UeCt	Total
0	0	1	1	5	5	10	7	6	35
R	10	9	9	5	4	0	2	3	42
Μ	0	0	0	0	1	0	1	1	3
Α	0	0	0	0	0	0	0	0	0
Total	10	10	10	10	10	10	10	10	80

Table 8. Mode of failure per repair group. O: Cohesive within the original material fracture. R: Cohesive failures within the repair (R) material fracture. M: Mixed fracture. A: Adhesive fracture.

## Mode of failure.

From the two-way-analysis of variance pairwise comparison we can state that there was a significant difference between PmP & UeC with a=0.008; UvP & UeC with a=0.0480; and PmPt & UvCt with a=0.0015.

UDMA samples (n=60) fracture modes are as follows:

- 56.67% had cohesive failures within the original (O) material.
- 38.3% had cohesive failures within the repair (R) material.
- 5% had mixed (M) fractures.

Fractures within the R material (n=42):

- 45.23% of the PmP samples
- 33.33% of the UvP samples

- 9.52% of the UvC samples
- 11.9% of the UeC samples

Samples and the number of fractures they had within each group was compared. The only groups with two or more fractures were within the non-thermocycled samples repair with composite (Table 9). It can be sugested that the multiple fractures within the original material (UDMA) is influenced by the high flexural strength of the repair material, making the UDMA the weak point for new fractures. However, this high flexural strength from the composite did not affect the number of fractures in the samples that were put through thermo-cycling.

Repair Group	1 Fracture	2+ Fractures
PmP	10	0
PmPt	10	0
UvP	10	0
UvPt	10	0
UvC	8	2
UvCt	10	0
UeC	6	4
UeCt	10	0

Table 9. Number of fractures per repaired sample.



Figure 12. Plot of Rank vs FS. Location of failure using ranked data; ranking by ammount in repair material

With the hypothesis that fractures being in different locations would influence the FS of the repaired sample, Figure 12 shows the plot graph revealing no statistically significant difference (a=0.0548) and therefore no correlation between both.

## **Chapter 4. Discussion**

The hypothesis that 3D printed UDMA repaired with Visio.Link and self-cured PMMA had the highest FS was rejected, as it had statistically significant lower FS compared to all other repairs.

The null hypotheses of this study were accepted as the repair protocol was not statistically significantly different between the repair protocols with or without thermocycling, and the flexural strength was not correlated to the mode of failure.

Although the shear bond strength was not tested in this study, it can be assumed that the bond created on each surface of any of the repair protocols tested will withstand forces to not have adhesive failures, however, the repair material will influence the repaired prosthesis' FS.

The unexpected result of UvP having the lowest FS values could be explained by a few factors, taking into consideration that UDMA samples repaired with PMMA did not have any adhesive or mixed fractures and the majority of fractures were within the repair material, compared to the ones repaired with flowable composite.

The first factor to consider is the delivery and mix of the repair material. Flowable composite is delivered in a prefabricated controlled environment syringe delivery system,

and this dispensary method allows for an accurate and consistent mix; conversely, the working and resultant biophysical properties of self-cured PMMA can be influenced by the monomer-powder ratio, which can vary from mix to mix and from dentist to dentist.<sup>33</sup>

The low FS and fractures mostly on the repair material also might be affected by the nature of self-cure PMMA's exothermic polymerization reaction, which makes it prone to bubbles as well as air bubbles as a result of manual mixing that will act as weak points.<sup>3,9</sup>

Another factor to consider is the material's water sorption and solubility, as this could also influence the final FS of the prosthesis. PMMA tends to absorb water by imbibition, as monomethacrylates absorb water due to their linear polymer network and air spaces in their structure.<sup>3,11,36</sup> In contrast, dimethacrylates, as 3D printed UDMA have a ridged central structures that limits water absorption to 0.8% by weight of water.<sup>3</sup> These polymer materials' water sorption is reduced by the crosslinking, which provides large of bridges between linear macromolecules to form a three-dimensional network.<sup>11</sup>

Provisional restorations' clinical expected function and durability can be anywhere from ~1-2 weeks to up to ~6-9 months depending on the type of prosthesis, number of teeth involved, purpose, lab work turn-around and/or implant osteointegration period (i.e., Maxillary implant supported fixed complete denture –ISFCD– immediate load interim prosthesis, where the full osseo-integration of implant in some cases could take around 6 months).

In this study, it was decided to recreate a clinical situation in where the patient would wear their interim prosthesis for an extended period of time until an unexpected fracture, where patients would schedule an appointment as soon as possible to have it repaired or replaced. With this in mind, the thermocycling process was done in accordance to the traditional dental clinical trials to mimic 1 year of wear,<sup>35</sup> knowing that clinically, these interim prosthesis are in function for less than a year, and there was no second thermocycling process after the fracture/cut of the samples, since the patients usually get them repaired as soon as possible.

In this research, although not statistically significant, the different materials were affected by the thermocycling shifting from the repair material to fractures in the original material. For example, in the case of group UvP, fractures were shifted from 90% within the self-cured PMMA repair material to a more even 50-50% fractures within the original and repair material. Similarly, samples repaired with composite as in the UvC group, fractures shifted from an even repair-original material location, to 100% of the fractures within the original UDMA material. This suggests a decrease of strength of UDMA after thermocycling, as well as greater water sorption of UDMA compared to flowable composite, as increased filler load of the material, decreases water sorption.<sup>1,11</sup>

The fact that only the samples repaired with composite had more than 1 fracture might be explained by the difference in modulus between the two materials influencing the stress concentrations and where the new fractures occur. Although there was no statistically significant difference when analyzing the dependence of the location of the fracture to the FS (Figure 12), there was a positive influence of the composite repair material on the final FS of the repaired samples. This modulus difference may also explain why the UDMA repaired with self-cured PMMA exhibited the lowest FS. Interesting area of study for a further research.

Ultimately, the purpose of this study and the methodology performed was to evaluate if the strength of the repaired 3D printed UDMA interim prosthesis is at least as strong as an unrepaired provisional. If the FS of the repaired prosthesis had statistically significant lower FS than the original prototype prosthesis or less than the 50 MPa dictated by the ADA specification no. 27,<sup>14</sup> then the clinician should take into consideration that it will take less force for this interim prosthesis to break again. Finally, the dentist will have to evaluate the time, effort, and financial investment to either repair or fabricate a new interim prosthesis.

## Limitations

With the above in mind, a limitation of this study is not including the analysis the material's solubility and how this would correlate on predicting where a new fracture would occur in a repaired prosthesis.

Within the same topic, another limitation included timing and number of thermal cycles, which does not necessarily correspond to clinical situations and could affect the outcome of the statistical analysis. However, as mentioned before, in a clinical setting, patients are seen to have their interim prosthesis repaired or replaced as soon as possible after the unexpected fracture occurs. Therefore, a second shorter thermocycling process after the initial fracture probably would have not significantly influence the prostheses' FS.

This research also assumed the accuracy of manufacturers' specifications, which were taken from their safty data/material properties sheets.

Lastly, FS is a stress unit measured in homogenous materials. FS in this case was applied to repaired samples in the assumption that both the repair and original material had the same modulus, knowing this is not the case. Although the standard deviation of the results did not indicate a large difference between the materials, it may be more clinically meaningful to report the force (in Newtons) that it took for the repair samples to break.

## **Chapter 5. Conclusions**

Understanding the limitations of this study, prosthodontists and clinicians in general should be aware that, even though, there was no failures on the adhesion interface, the FS of repairs of 3D printed UDMA restorations was affected by the repair material protocol, and the FS of the repair interim prosthesis was influenced positively by repairs with composite materials.

With the results of this study, the clinical recommendations to achieve a high FS repaired interim prosthesis would be to use flowable composite as repair material together with either surface treatment (etch and bond, or Visio.Link primer).

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