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Effects of Thermocycling and Hydrolytic Aging on
The Flexural Strength of Additively Manufactured Restorative Materials

by

Steven Abu Al Tamn, BDS

A thesis submitted to the faculty of the graduate school,
Marquette University,
In Partial Fulfillment of the Requirements for
The Degree of Master of Science

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ABSTRACT

Effects of Thermocycling and Artificial Aging on The Flexural Strength of Additively Manufactured Restorative Materials

Steven Abu Al Tamn, BDS

Marquette University 2023

Introduction: Additive manufacturing is being integrated into various aspects of clinical prosthodontics due to its potential for use in various indications and the promise of improved clinical efficiency and properties.

Objective: The purpose of this in-vitro study was to evaluate the flexural strength of two 3D-printed resin composites in relation to a conventionally fabricated resin composite after being subjected to two different artificial aging methods: thermocycling and distilled water storage.

Material and methods: A total of 220 samples were fabricated (N=220); 72 samples were fabricated from Integrity composite (Dentsply Sirona) and 148 samples were fabricated from two 3D-printed composite materials MFH and CROWNTEC (NextDent). The conventional material (Integrity composite) was dispensed from an auto mixer into a mold adhering to ISO 4949 sample dimensions of 25x2x2 mm. The 3D-printed samples were designed to the same standard. The designing process was carried out on a CAD software Meshmixer (Autodesk) and printed using a NextDent 5100 (3D systems), followed by an alcohol rinse and post-printing polymerization. The samples were finished and divided into 3 sub-groups: non-aged, thermocycling, and distilled water aging. The non-aged group was preserved in distilled water for 24 hours, the thermocycled group was cycled for 700 cycles using a well-known protocol consisting of alternating water baths between 5°C and 55°C water bath, and the water storage group was stored in distilled water for 1 month. The samples were subjected to a three-point flexural test using a universal testing machine (Instron). Data were statistically analyzed using statistical software R at a 95% confidence interval.

Results: The results showed the mean \pm standard deviations for flexural values for Integrity was 76 ± 31 MPa, MFH 148 ± 16 MPa, and CROWNTEC 173 ± 21 MPa. One-way analysis of variance showed statistically significant differences among the groups ($p < 0.05$).

Conclusions: From the results of this study, it was concluded that all the materials performed above the clinical acceptability threshold of 60 MPA set by ISO standard 4049. FS was highest for CROWNTEC, followed by MFH then Integrity. FS was not affected by artificial aging techniques except for CROWNTEC which showed lower FS after water aging for 1 month.

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LIST OF SYMBOLS AND TERMINOLOGY

Guide to Acronyms

CAD/CAM	Computer Aided Design / Computer Aided Manufacturing
STL	Standard Tessellation or Triangulation Language
FDA	Federal Drug Administration

Explanation of Digital Dental Terms

General Term	Explanations or definitions
3D printing	Additive manufacturing is the process that builds 3D objects by adding layers of material on top of each other until the final object is produced.
CAD/CAM dentistry	Computer-aided design and computer-aided manufacturing Using computer technologies to design and manufacture different types of dental restorations.
Computer-Aided Design (CAD)	The use of computer software to create (2D or 3D) graphical representations of physical objects.

Milling	The machining process of using rotary cutters (burs) to remove material from a workpiece to fabricate dental restorations.
Standard tessellation language (STL)	File format that can be used on CAD software created by 3D Systems., STL files describe only the surface geometry of 3D object without any representation of color.
Tessellation	The division of a surface into smaller polygons.

CHAPTER 1: Introduction

The goal of prosthetic dentistry was eloquently said by M.M Devan to be the “*perpetual preservation of what remains is more important than the meticulous replacement of what is missing.*” The specialty of prosthodontics is the art and science that is principally concerned with prosthetic rehabilitation of lost teeth and prevention of further loss (GPT-9). Prosthetics were fabricated in ancient times by the Assyrians, and those basic prosthetics were made out of ivory, gold, and extracted teeth (Singh et al., 2017). The science of dental materials encompasses the physical, chemical, mechanical and biological properties of biomaterials and dental materials. One main importance lies in that it allows prosthodontics as a clinical specialty to advance. New dental biomaterials are developed to answer the demands of clinicians that stem from daily challenges due to the complex workflow and lack of efficiency and speed of manufacturing. These clinical demands have incentivized manufacturers to innovate new techniques to solve such challenges. Charles Hull invented stereolithography in 1983 (Hull, 1986) and started the digital additive manufacturing revolution. Nowadays, digitalized workflows offer more streamlined processes, thus more patient acceptance and greater efficiency. However, as the transition from the analog world to a more digitalized workflow occurs, the properties and environmental footprint should offset the initial learning curve (Joda and Brägger, 2014; Colorado et al., 2020). Since the invention of stereolithography in 1983, significant accomplishments have been achieved as manufacturers (Carbon printing, EnvisionTEC) utilized continuous 3D-printing, which significantly reduced print time and increased efficiency. Current advancements in additive manufacturing have virtually allowed the additive manufacturing of prosthetics in a wide range of biomaterials, from ceramics to

metals to various photoreactive polymeric resins. The continuous development of photoreactive resins allowed the manufacturing of a wide variety of dental prosthetics, which range from surgical templates, occlusal splints, implant metal frameworks, provisional crowns and bridge and implant restorations. The federal drug administration (FDA) recently approved materials for definitive restorations, such as denture bases and crown restorations [Leading Dental Materials for 3D Printing (2019), Retrieved January 5, 2023)]. Additive manufacturing has advanced the field of prosthetic dentistry by allowing office fabrication, providing clinicians with unprecedented control and less expense than subtractive manufacturing due to minimizing material waste during fabrication. Under certain circumstances, an additively manufactured full-arch prosthesis can be 3D-printed in just under 30 minutes. On the laboratory front, additive manufacturing continues to rise in numbers, with 77% of the laboratories utilizing additive manufacturing due to the reported benefits of achieving a fully digital workflow, increased efficiency, eliminating waxing and model pouring, increased accuracy, and reduced production and turnaround time (Carr, 2023). However, the properties of additively manufactured resins are still underreported and long-term success unknown (Suliman, 2019).

Study Objectives:

The objective of this study was to investigate the flexural strength of additively manufactured materials with and without two artificial aging methods and compared to a control group.

Null Hypotheses:

H01: There is no statistically significant difference among the three different tested materials

H02: The aging protocols have no statistically significant effect in the flexural strength of the investigated materials.

CHAPTER 2: REVIEW OF LITERATURE

1-Resin Methacrylates in Dentistry

A tremendous advancement in dentistry occurred from the 1800s to 1975 when a transition occurred from ill-fitting dentures fabricated from natural resins to modern synthetic resins (Peyton, 1975). In 1947, methacrylate resins were used as direct restorations, and continuous development occurred. Traditional methacrylates are not commonly used as direct definitive restorations due to the poor mechanical properties required for definitive restorations. However, they are widely used for provisional restorations due to adequate stiffness and relining potential. These materials include PMMA, PEMA, VEMA, etc. (Gracis et al., 2000).

In 1952, Dr. Ray Bowen introduced bisphenol A glycidyl methacrylate (BIS-GMA), which was a self-curing methacrylate with dispersed ceramic particle fillers. These resins were developed to have a photoinitiator that allowed an ultra-violet light to activate and initiate the polymerization reactions resins. The ultraviolet resins were replaced with blue visible light photo-polymerizable resins. In the 1970s, a category of improved resin composite was introduced with macro-filled resin composite, characterized by large silica particles and quartz, which improved mechanical properties, offered less water sorption, polymerization shrinkage, and thermal expansion than previously unfilled (Anusavice, 2013). This composite type did suffer dullness and wear of the softer organic matrix, and these inferior properties were due to unbonded fillers, which were added to improve the physical and mechanical properties of the resin composites (Singh et al., 2017). A

significant improvement was the addition of silane to chemically adhere the inorganic filler particles to the organic resin matrix.

Multiple generations of resin composites were introduced, and many advancements were due to the filler type size and amount (Ferracane, 2011). The product of development resulted in microfilled, hybrid, micro-hybrids and nanohybrids, low shrink formulations, and self-adhesive flowable (Ferracane, 2011). In the 1980s, additive manufacturing utilized similar UV and blue visible light to polymerize additively manufactured resins in a similar fashion to photopolymerized resins used.

2-The Provisional Restoration

The provisional restoration is a critical step in prosthodontic rehabilitation. It should protect soft tissues and fulfill functional and esthetic requirements (Rosenstiel, 2016). It is particularly important in implant dentistry as utilization periods may exceed that of natural tooth-borne restorations. The reported average restoration provisional utilization is 200 days (Drago, 2015) in full arch implant dentistry, while it is reported that 37.5 days for crowns and fixed partial dentures is the average utilization for tooth-borne restoration (Luthardt, et al., 2000).

The provisional restoration serves an important role in both demonstrating esthetic and functional changes to patients and serving as a trial template in complex full arch restorations when increasing the vertical dimension of occlusion is evaluated. Most interim restorations are still fabricated with conventional methods. Examples of those materials are BIS-GMA, PMMA, EMMA based materials (Clinicians Report, 2023; Baroudi, 2015; Freedman, 2007). The properties of conventional materials as flexural strength, modulus of elasticity, toughness, hardness, and their repair potential, have been widely reported

(Balkenol, 2007; Balkenol 2008; Thompson and Luo, 2014). Generally, PMMA and Bis-GMA have been the most popular materials used by clinicians due to immediate chairside availability, ease of use, and lower cost (Thompson and Luo, 2014; Clinicians Report, 2023).

Light initiated polymers (BIS-GMA) offer less odor, thermal, and shrinkage. However, they have limitations regarding marginal accuracy, color stability, and mechanical properties (Tjan, et al., 1997; Rutunkas, et al.; 2010; Alp et al., 2010).

Furthermore, data surrounding CAD/CAM provisional restorations is limited (Joda and Bragger, 2014 Joda and Bragger 2015: Suliman, 2019). Emerging in vitro data favors the strength of CAD/CAM 3D-printed materials. A study by Lee et al. found that a 3D-printed denture base of Bisphenol A dimethacrylate resin composite showed higher impact strength than heat-cured PMMA (Lee et al., 2022). The flexural strength of a 3D-printed methacrylate ester resin composite (NextDents' C&B) was not significantly different from the Bisacrylate resin composite (Dentsply Sirona's Integrity). However, the modulus of elasticity for the 3D printed resin composites was lower than that of Integrity Bis-GMA resin material (Tahayeri et al., 2018).

Despite the lack of long-term data, there has been a shifting trend towards fully digitalized workflows due to numerous clinical advantages (Joda et al., 2014; Joda et al., 2015; Joda et al, 2017) demonstrated a reduced number of visits as well as optimized visit time while using a fully digitized workflow. Another advantage is the limited material waste achieved by additive CAD/CAM technology.

3-Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM)

Computer-aided design involves using software to sketch a design. The design can be utilized for the purpose of demonstration or execution by using computer-aided manufacturing, which involves computer numerals to control the fabrication of the design into three-dimensional objects.

The first CAD/CAM program was attributed to Renault in 1966. Dentistry would follow other industries' suit in attempts to digitalize fabrication. In 1980, the basic concept of operation to-mill chair-side inlays was developed by Mormann and Brandestini (Morman, 2006). The first commercially available unit capable of scanning and producing chair-side inlays was introduced in 1985 (Mormann, 2006).

CAD/CAM can be subdivided into subtractive manufacturing and additive manufacturing. Subtractive milling utilizes blanks of a chosen material and burs that move in several axes to mill the object. The additive manufacturing process is defined as the process of joining materials to make objects from 3D model data, usually layer by layer (Standard terminology for additive manufacturing, 2015).

4- Additive Manufacturing in Restorative Dentistry

Additive manufacturing can be categorized into four main subcategories. Digital light processing (DLP), Stereolithography (SLA), Material Jetting (MJ), and Material Extrusion (ME). MJ and ME have the ability to print a variety of materials supplied in filament form and the distinct ability to print colored materials (Revilla, 2018). SLA and DLP are most used in clinical dentistry due to their size, efficiency, resolution of the print objects, and range of materials that can be printed (Revilla, 2018). In 1983, Charles Hull invented stereolithography, in which a building platform is immersed in liquid resin to be

photopolymerized by an ultraviolet laser (Hull, 1986; Hull, 1991). The laser is focused using a set of lenses and then reflected by two motorized mirrors (Revilla, 2018). Larry Hornbeck of Texas Instruments created the DLP technology in 1987, and it is very similar to SLA in the fact that it is liquid vat polymerization. The difference is that DLP utilizes digital micromirror representing a pixel or more, which when projected on liquid resins, activates and initiates the polymerization reaction. DLP technology does not use motorized scanning mirrors to reflect UV as in SLA technology (Hornbeck, 2009).

5-Mechanical Properties (Flexural Strength and Aging)

Flexural strength is defined mathematically as the force per unit area at the instant of fracture (Anusavice, 2013). It's exemplified by a 3-unit fixed dental prosthesis where the terminal units are fixed, and a central force is applied on the suspended pontic area. Three-point flexural stress is important in dentistry as it mimics a fixed dental prosthesis under function where both compression and tension occur, the stress produced is termed complex stress.

In 2018, Tahayeri et al. investigated the flexural strength and elastic modulus of 3D-printed resin composite (Nextdent C&B, Nextdent) vs conventionally fabricated bis-acrylate (Integrity, Dentsply Sirona) and PMMA (Jet, Lang). The resultant flexural strength of the 3D printed composite and Integrity (Bisacrylate) was statistically similar and significantly greater than that of PMMA (Jet, Lang). Alp et al. (2019) compared the flexural strength of different (CAD/CAM) PMMAs and conventional (PMMA) polymers and conventional resin composite interim materials after thermocycling. Milled PMMA had higher strength than Bis-acrylate and conventionally fabricated PMMA.

Artificial aging is a process that attempts to simulate clinical environmental conditions artificially (Pires-de-Souza, 2009; Turgut S, 2011). Different laboratory methods were devised including thermocycling, load cycling, time lapse (immersion in distilled or deionized water), and exposure to acid challenge. Several studies have shown a significant effect on the flexural strength and fracture toughness of Urethane and bis-acrylate interim materials (Zuccari, 1997; Kerby, 2013). Thermocycling remains a common method to artificially age resin composites (Hikel, 2013; Gale et al., 1999). Despite the different methods, no one method is superior and more accurate in simulating clinical situations.

CHAPTER 3: Materials and Methods

Study Design, Variables, and Controls:

To address the research hypotheses, an in-vitro study was designed that included provisional restorative materials and aging as variables. A sample size $N=18$ per group was calculated, based on a pilot study, to estimate statistical differences at a 0.05 confidence interval. Three materials were investigated ($n=220$): a conventionally fabricated material auto-mixed multi-methacrylates based resin composite served as a control ($n=72$), Integrity (Dentsply Sirona, Charlotte, NC), and two 3D-printed resin composites were investigated in this study ($n=148$), CROWNTEC (NextDent, Soesterberg, Netherlands), C&B MFH (NextDent, Soesterberg, Netherlands). A bis-acrylate resin composite Integrity (Dentsply Sirona) was selected to serve as a control. The composition of the materials and handling is described in Table 1.

Table 1 Material composition and method of manufacturing

Manufacturer	Material (Lot)	Composition	Reported FS	Fabrication method
Dentsply Sirona	Integrity (00114346)	Acrylates and methacrylates (bis- and multifunctional), Barium boro alumino silicate glass and Silicon Dioxide	95 MPA	Conventional automixed
NextDent	C&B MFH (WX382NO2)	Methacrylic oligomer, Glycol Methacrylate, Phosphine oxide	107 MPA	Additively manufactured
NextDent	CROWNTEC (E391)	BisEMA, Trimethylbenzoyldiphenylphosphine oxide	>150 MPA	Additively manufactured

Sample preparation:

The control material (Integrity, Dentsply Sirona) was fabricated according to the manufacturer's instructions for use by dispensing the resin from an auto-mixing dispenser into a putty mold (Fig 1), and a glass slab was applied to the putty mold and allowed to set in self-cure mode for 5 minutes as recommended by manufacturers. After 5 minutes of setting, the materials were retrieved, and excess flash was trimmed using bard parker handle number 6 with number 25 blade mounted on to it. Following flash removal, the

samples were further refined using silicon carbide paper P400 (320 grit) (Buhler, Lake Bluff, IL, USA).

The 3D-printed materials investigated in this study were CROWNTEC (NextDent, Soesterberg, Netherlands) and NextDent C&B MFH (NextDent, Soesterberg, Netherlands). The 3D-printed samples were designed using CAD software Meshmixer (Autodesk, Mill Valley, CA) (Fig 2) to meet the ISO 4049 standard dimensions of (25 × 2 × 2 mm). The designs were then transferred to NextDent's segmentation software using Standard Tessellation language file format (STL). 3D Sprint version 2.0 (3D systems, Rockhill, SC) segmentation software (Figs 3 & 4) was utilized to give the print order. The specimens were aligned at 0° orientation as the highest flexural strength was observed at zero degree print. Auto-support generation was selected for the samples, uniform segmentation was used at a 50 µm z-axis layer height. NextDent printer 5100 (3D systems, Rockhill, SC) (Fig 5) was used to print the two 3D printed investigated resins, CROWNTEC (NextDent, Soesterberg, Netherlands) and MFH C&B (NextDent, Soesterberg, Netherlands). Post-printing processing included immersion in 99% isopropyl alcohol for 3 minutes air-drying and post-print polymerization using a photopolymerization unit LC-3D Printbox (NextDent, Soesterberg, Netherlands) (Fig 6) for 30 minutes per the manufacturers' instructions. The samples' dimensions were confirmed by using a digital caliper (Mitutoyo, Tokyo, Japan) to a resolution of 0.001 mm (Fig 7).

Diagrams of technique

Figure 1: Integrity material dispensed from an automixing dispenser.



Figure 2: Sample design.

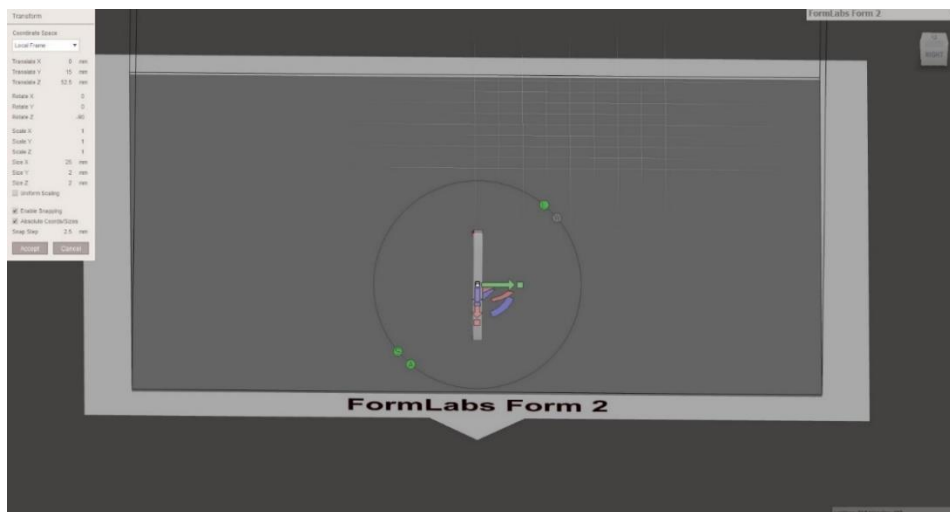


Figure 3: CROWNTEC Samples imported into 3D Sprint software.

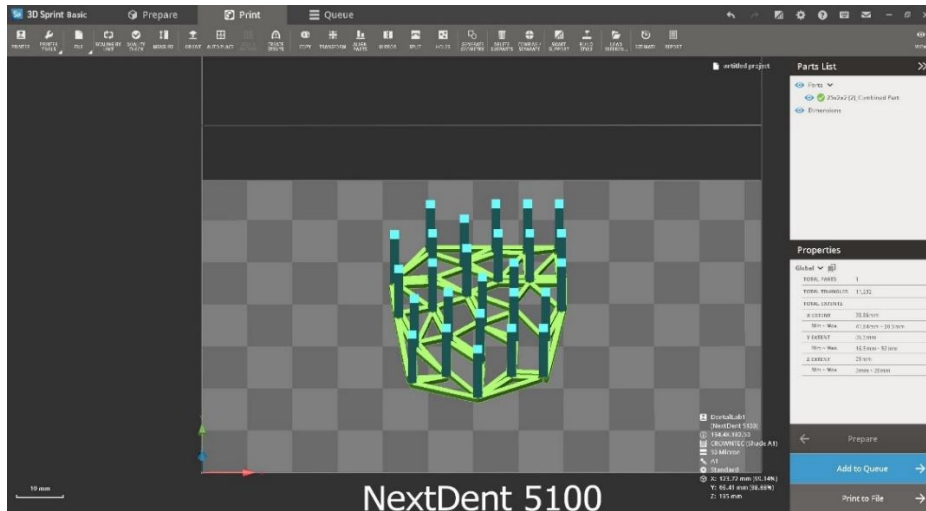


Figure 4: MFH Samples imported into 3D Sprint software.

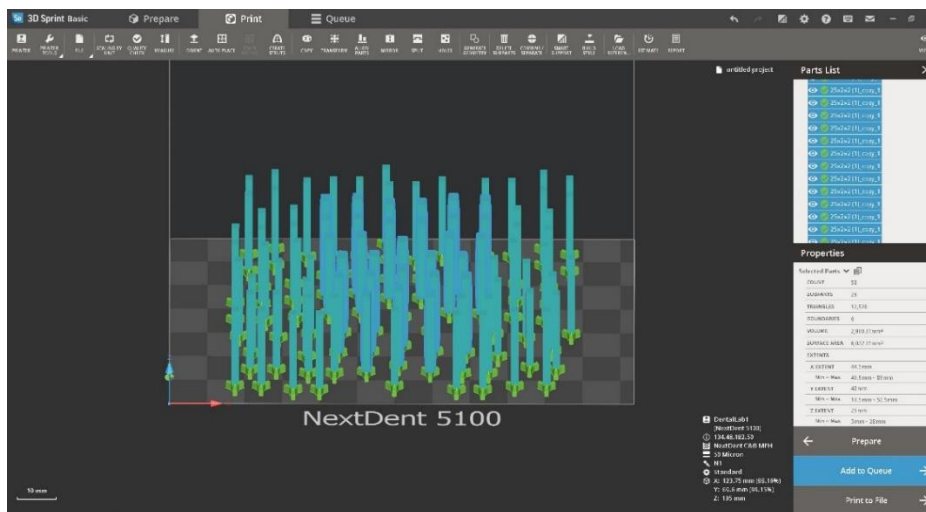


Figure 5: NextDent printer 5100.



Figure 6: LC-3D Printbox.



Figure 7: Mitutoyo caliper verifying dimensions.



The specimens were divided according to aging protocols: (1) No aging, (2) immersion in water for 30 days, and (3) thermocycling for 700 cycles. The non-aged groups were stored for 24 hours in 37°C distilled water, the artificially aged group was stored for 30 days in distilled water, and the accelerated aging used a thermocycling protocol of 700 cycles of 5°C (30 seconds dwell time) and 55°C (30 seconds dwell time) using a Sabri thermocycler (Fig 8).

Figure 8: Sabri thermocycling machine.



Flexural strength:

A flexural strength test was used following a well established research protocol (Thompson and Luo, 2014). The final dimension of each specimen was recorded. Specimens were placed on a standard 3-point bending apparatus with a bottom support span of 20 mm. A universal testing machine (Instron Model 5500, Instron, Norwood, MA), a 500-Newton load cell (Fig 9), and top support rod were used to perform the three-point flexural test (Fig 10) using a 0.7 mm/min crosshead speed. Specimens were loaded until fracture. The peak force to fracture samples was recorded.

Figure 9: Universal testing machine (Instron 5500).



Figure 10: Chisel mounted on universal testing machine



The Max function on the excel CSV files was used to locate the maximum force to calculate flexural strength. The flexural strength was calculated manually using the following formula $FS = \frac{3FD}{2Wh^2}$, where F is the maximum load at failure (N); d is the distance between support spans (mm); w is the width at the center of the specimen (mm); and h is the height at the center of the specimen (mm).

Statistical analysis:

The data were organized in a single excel sheet and statistical analysis was performed using statistical software R version 4.2.2. The means and standard deviations were calculated for each group. One-way analysis of variance, pairwise comparison was performed. A set confidence interval of 95% was used, analysis of variance was performed to detect if differences exist between materials, and pairwise comparisons were performed to detect flexural strength changes post-aging.

CHAPTER IV: Results

Upon reviewing the observations between the materials and aging methods, the flexural at baseline strength for Integrity was 76 ± 31 MPa, MFH 148 ± 16 MPa, and CROWNTEC 173 ± 21 MPa.

One-way analysis of variance showed statistically significant differences among the groups at $p < 0.05$ (Table 2).

Table 2 Analysis of variance table

Analysis of Variance Table						
Response: MPA						
	Df	Sum Sq	Mean Sq	F value	Pr(>F)	
materials	2	126965	63482	116.52	< 2.2e-16	***
Residuals	72	39226	545			

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1						

Table 3 Descriptive results of flexural strength include means and standard deviations.

Material (Intervention)	Mean (MPa)	SD (MPa)
Integrity (control)	76	31
Integrity (1-month)	70	30
Integrity Thermocycling	77	45
CROWNTEC (control)	173	21
CROWNTEC (1-month)	154	22
CROWNTEC Thermocycling	173	23
MFH (control)	148	16
MFH (1-month)	154	18
MFH Thermocycling	150	20

The intervention's effect depended on the type of intervention and material type. All the materials performed above the set clinical threshold for clinical use, according to the American Dental Association specified requirements Specification 49, which specify that a minimum flexural strength of 60 MPAs is required for clinical use.

Analysis of variance:

Analysis of variance at 24 hours showed a statistical difference between the materials ($p < 0.005$).

Analysis of variance for the thermocycled groups showed a statistical difference between the materials ($p < 0.01$).

Analysis of variance for artificially aged groups showed statistical differences between the materials ($p < 0.01$).

Pairwise Comparisons:

Pairwise comparisons were performed to compare the effects of thermocycling and aging in relation to the control. There was no statistically significant difference for Integrity when subjected to thermocycling ($P > 0.05$) and the same statistical result was found for artificial aging ($P > 0.05$).

There was no statistically significant difference for MFH when subjected to thermocycling ($P > 0.05$) and artificial aging ($P > 0.05$).

CROWNTEC showed a statistically significant difference between 1-month artificial aging compared to 24 hours ($p < 0.05$).

Figure 11: Observation of the materials at baseline (24 hours)

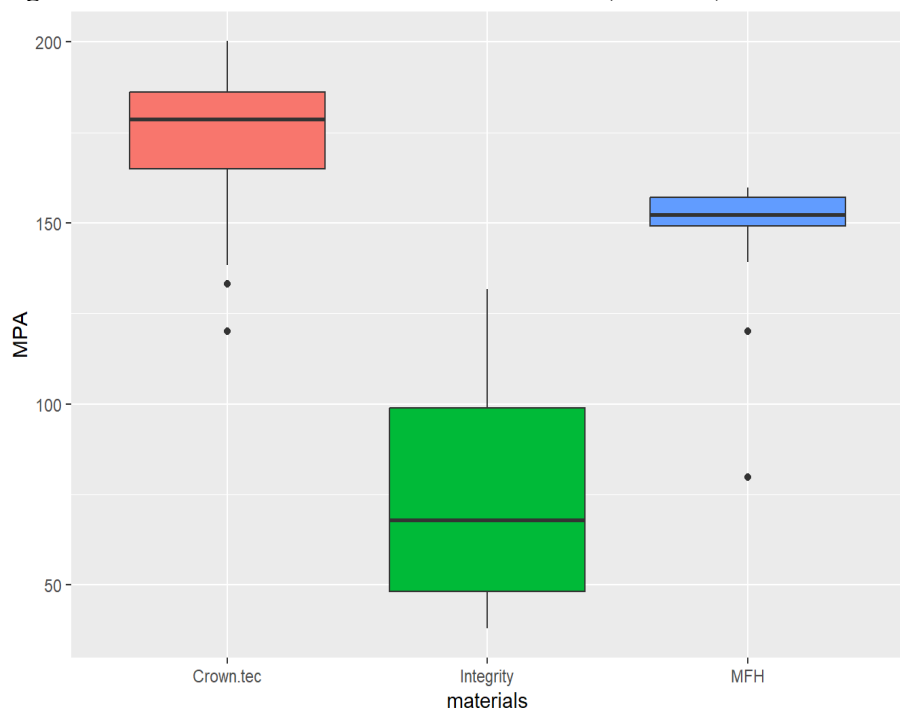


Figure 12: Observation of the materials after 700 cycles of thermocycling

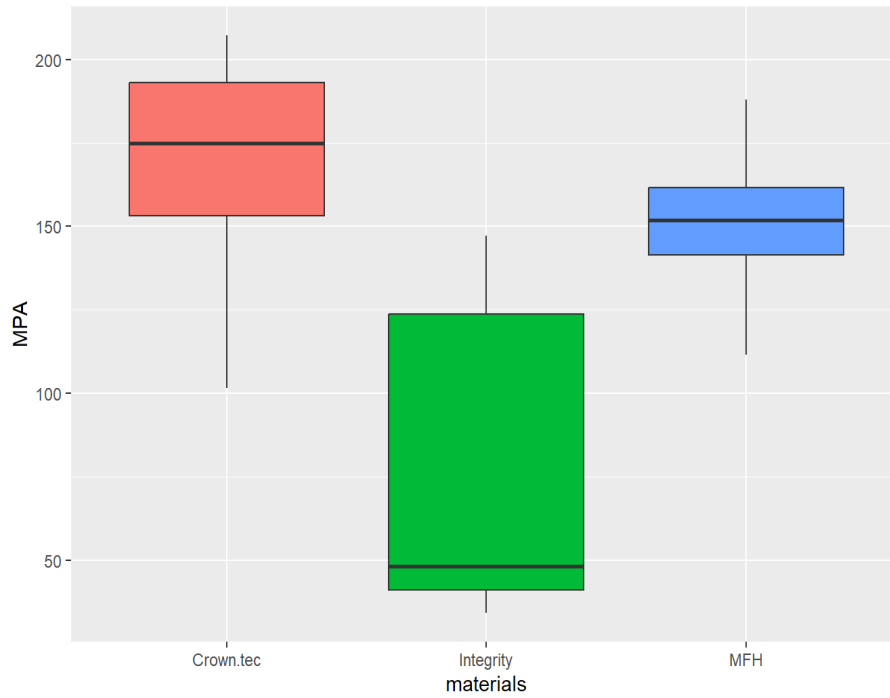


Figure 13: Observation of the materials after 1-month of artificial aging

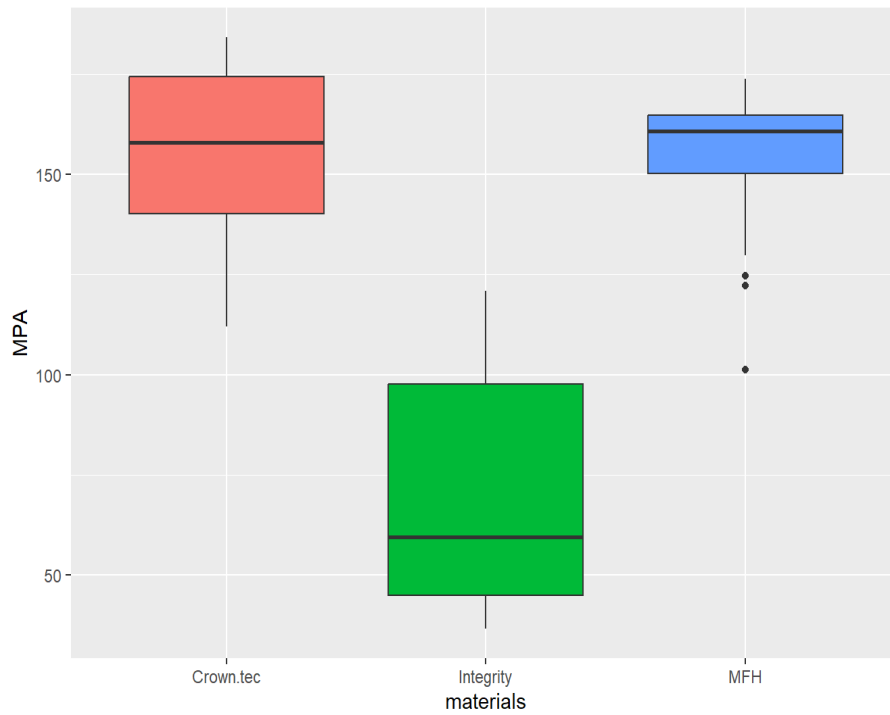


Figure 14: Pairwise tests at different conditions (Integrity).

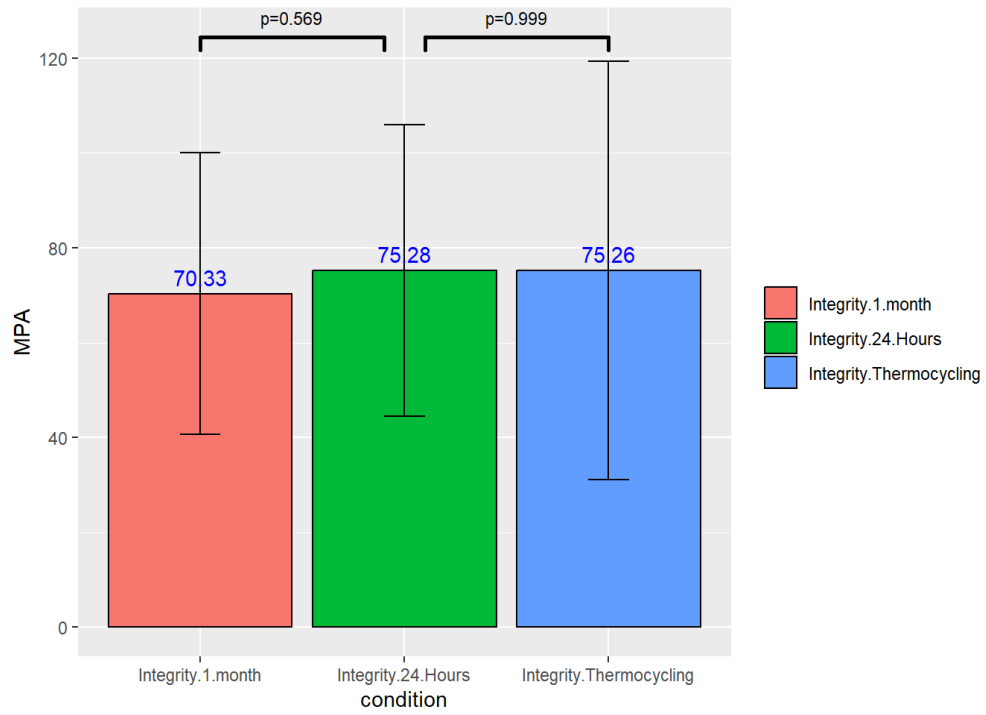


Figure 15: Pairwise tests at different conditions (CROWNTEC).

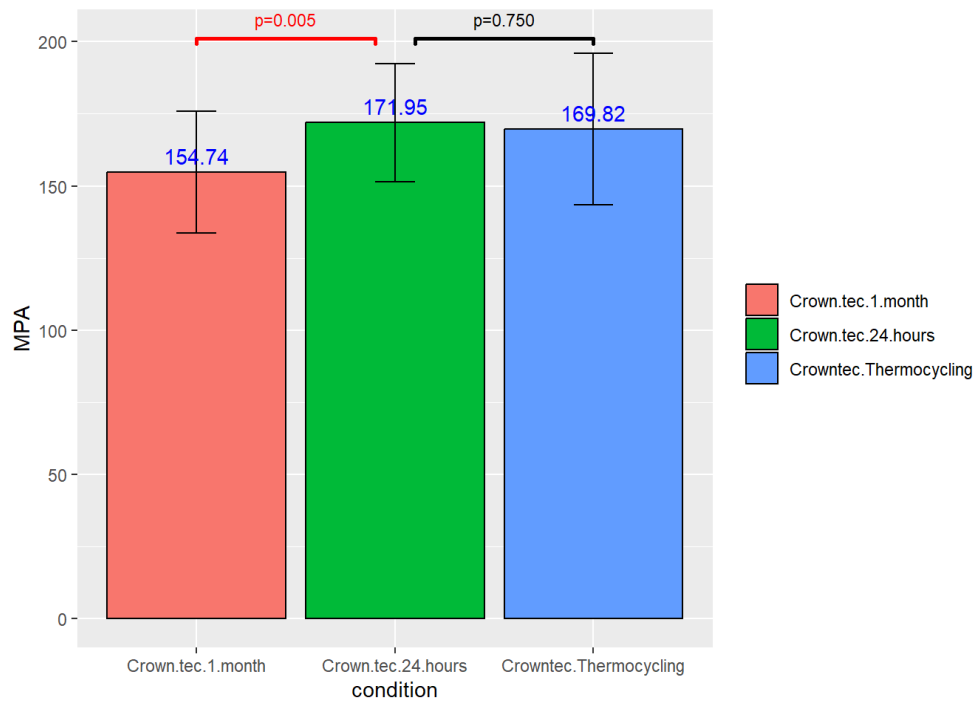


Figure 16: Pairwise tests at different conditions (MFH)

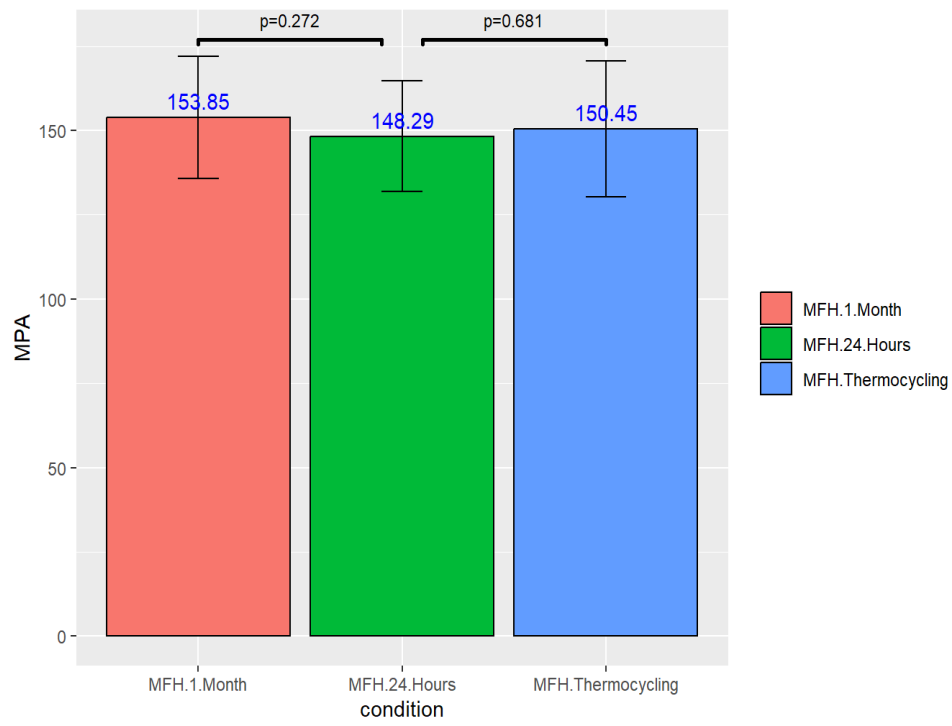
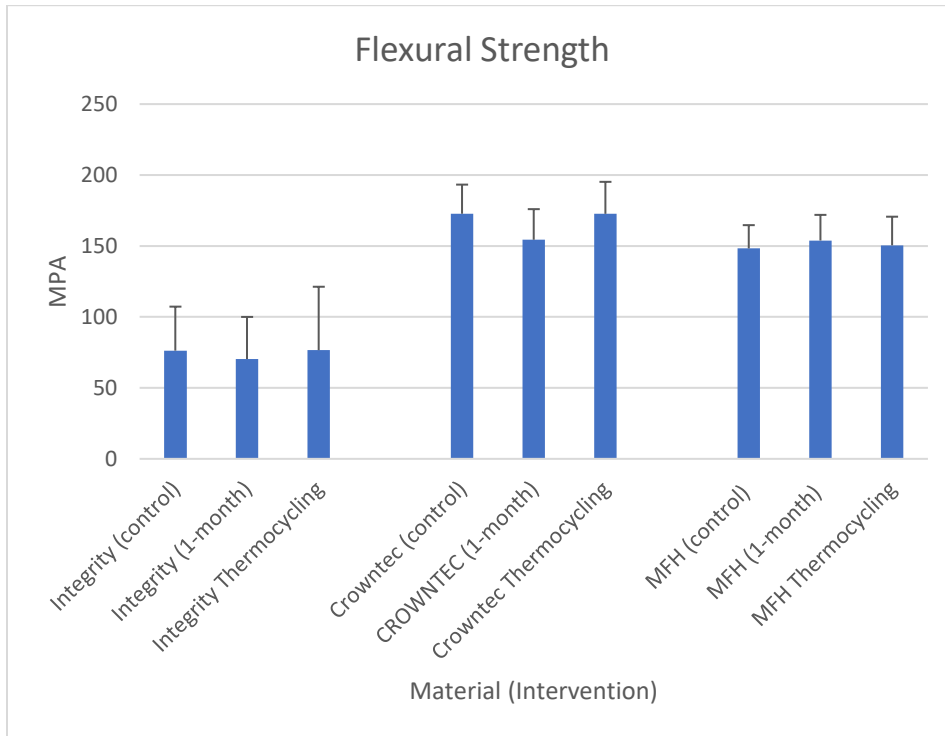


Figure 17: Bar graph of the collective flexural strength.



CHAPTER V: Discussion

The present study compared the flexural strength of 3D-printed resin composites to a conventionally fabricated resin composite subjected to 3 aging protocols (no aging, 700 thermocycles, and 1-month distilled water storage).

The null hypothesis of this study was that there would be no statistical differences between the materials. Flexural strength was used to test this hypothesis. The null hypotheses were rejected as there were statistical differences among materials and aging protocols.

The results have shown that the flexural strength for the tested materials is as follows CROWNTEC > MFH > Integrity. This trend is in agreement with prior published findings (Ellakany, 2022). In that study, a milled PMMA and two 3D-printed resin composites and conventionally fabricated resin composite were tested for flexural strength in a 3-point bending apparatus. The following trend was found for flexural strength milled PMMA > SLA 3D-Printed > DLP 3D-printed > conventionally fabricated resin composite. On the other hand, this project's findings are not in agreement with the findings of Tahyri et al. (2008) that found 3D printed resin composites (NextDent's C&B) have similar flexural strength as the conventionally fabricated auto mixed-resin composite (Integrity). While the current study showed lower flexural strength for auto mixed-resin composite (Integrity) when compared to the two 3D-printed resin composites (MFH and CROWNTEC), this could be explained by the usage of a filled resin NextDent MFH as opposed to its predecessor NextDent C&B which was an unfilled 3D-printed resin composite which was used by Tahyri et al. Furthermore, the authors of the said article have eliminated the post-printing polymerization step which may negatively impact the strength values of the tested 3D printable resins. In addition to that the newer generation of 3D-printed resin composites

contain higher filler loading percentages. Another factor is air bubble incorporation during the 3D printing process which may vary among printers as it could also contribute to lower flexural strength (Tahyri et al., 2008).

Artificial aging is a process to simulate clinical conditions to estimate the clinical durability of provisional restorations. Provisional restorations function in the oral environment for over one month. A plethora of acceptable aging protocols exist including thermocycling, water storage, and UV irradiation. In the current study, distilled water storage and thermocycling were used. The findings of this study showed that flexural strength was material dependent and dependent on the method of aging (Fig 16). CROWNTEC at one month of artificial aging showed a statistically significant decrease in flexural strength when compared to MFH (Fig 15) and Integrity. This finding could be explained by degradation of the resin matrix and the possible loss of adhesion between the organic and inorganic content. Another reason is the higher diffusion coefficient for water in the CROWNTEC material than the other test materials (Matsukawa, 1994) as water adsorption in polymethyl methacrylate chains would push polymer chains apart, thus causing expansion and the effect of the water as a plasticizer, causing polymeric materials generally to present with lower flexural strength over time (Matsukawa, 1994). However, the time to reach equilibrium strength is related to the material (Takahashi et al., 1999; Balkenol, 2008). The results from the study do align with Takahashi et al (1999). Flexural strength (FS) is essential for achieving clinical success (Balkenhol, 2008). Balkenhol evaluated the FS of different materials stored at different times after mixing and reported that FS of polymeric resin materials is dependent on the storage time and curing mechanisms and chemical nature, which can explain the differences between auto-mixed

resin composite (Integrity) and the two 3D-printed resin composites (CROWNTEC and MFH). In the present study, there was a statistically significant difference between materials.

There are several strengths noted for this study. Being an in vitro study, it allowed for the control of study variables and the utilization of an FDA-validated workflow and equipment which eliminated doubt from data. The results from this study were in agreement with comparable published literature for similar additively manufacturing and manufactured materials. The utilization of the two widely used artificial aging methods allowed the assessment of simulated performance of the materials. There were also limitations to the study, including assessment of a singular mechanical parameter and no fracture pattern assessments that precludes comprehensive assessment of the modes of failures. Future studies should consider the inclusion of more comprehensive mechanical properties and dynamic testing protocols to assess simulated clinical performance, including modulus of elasticity, hardness, and wear testing.

CHAPTER VI: Conclusions

Within the limitations of this in vitro study, the following conclusions can be made:

1. All the provisional restorative materials investigated performed above the required flexural strength threshold required for clinical use according to ISO 4049 of 60 MPa
2. The observed flexural strength trend is as follows CROWNTEC>MFH>Integrity. where there were statistically significant differences between the materials.
3. The flexural strength of the three investigated materials was not significantly affected by artificial aging using thermocycling and water storage, with the exception of CROWNTEC which showed statistically significant decrease in flexural strength mean values post-water storage aging.

CHAPTER VI: Bibliography

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