

EFFECT OF INTERIM FIXED PROSTHODONTICS MATERIALS AND
FLOWABLE COMPOSITE RESINS ON POLYMERIZATION
OF POLYVINYL SILOXANE IMPRESSIONS

by

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DEDICATION

This thesis is dedicated to my wife Athari Almalki; my parents, Mr. Darwish Alsayed and Mrs. Fatimah Alsayed, and my elder brother Mr. Mohammed Alsayed. Their utmost devotion and unconditional love provided a solid basis facing the challenges for me to succeed in my education.

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INTRODUCTION

Elastomeric impression materials have found their applications in restorative dentistry.¹⁻⁵ Polyvinyl siloxane (PVS) impression materials were first introduced in the 1970s. They have gained in popularity and acceptance in restorative dentistry because they produce accurate impressions, have good elastic recovery, dimensional stability, and sufficient tear strength.⁶

Despite the numerous advantages of PVS, there is potential for unfavorable interactions between PVS and other dental materials, which come into direct and indirect contact during restorative rehabilitations. These chemical interactions affect the polyvinyl siloxane accuracy and performance.^{6,7} For instance, it is well documented that PVS shows polymerization inhibition when it comes into contact with sulphur containing latex gloves.^{1,4,6,8} Moreover, studies have claimed that retraction cord medicaments such as ferric sulfate, ferric subsulfate and aluminum chloride cause polymerization inhibition of the PVS.⁹ However, other studies have shown no inhibitory effect with these types of medicaments.¹⁰

Flowable composite resins (FCR) have been widely used as preventive resin restorations for minimally invasive occlusal class I, class II, and class V non-cariou lesions. FCRs appear to be less time consuming and more esthetic than amalgam.¹¹ They are also an acceptable direct core material when enough tooth structure remains intact.¹² It has been recommended to reduce early failure rates of endodontically treated teeth.¹³ According to a clinical practice guideline done by Jean et al., FCRs resins are the first choice of material for pit and fissure sealants.¹⁴ The low material viscosity and injectability provide the clinician with superior handling properties.¹⁵

Interim fixed prosthodontic materials (IFPM) are an essential part of oral

rehabilitation. Biologically acceptable restorations should provide the prepared tooth with form and function resembling the definitive restorations.¹⁶ Besides the esthetic, functional, and protective advantages, they can be utilized diagnostically to assess different aspects in the planning of definitive restorations.^{17,18}

It has been found that certain types of IPFM and FCR materials have an inhibitory effect on the polyvinyl siloxane impression materials.^{19,20} It has been assessed with a visual scoring method only and research is lacking to validate the assessment of this phenomenon.

OBJECTIVE

The aim of this study was to test the inhibitory effect of certain types of IFPM and FCR materials with PVS impressions as quantified by weight change and qualified by Fourier transform infrared (FTIR) spectroscopy.

NULL HYPOTHESIS

IFPM and FCR will not cause significant inhibition of PVS impressions as quantified by weight change and qualified by FTIR spectroscopy.

ALTERNATIVE HYPOTHESIS

IFPM and FCR will cause significant inhibition of PVS impressions as quantified by weight change and qualified by FTIR spectroscopy.

REVIEW OF LITERATURE

IMPRESSION MATERIAL

The history of today's impression materials began in 1925 when Poller granted a British patent on the first hydrocolloid impression material.²¹ The introduction of hydrocolloids made the impression of undercuts possible. In the 1950s, polysulfides and condensation silicones were introduced as elastomeric impression materials and were commonly used in fixed prosthodontics.

The limitation of those impression materials is shrinkage over a period of several hours due to the evaporation of small molecules such as the water with hydrocolloids and the alcohol with condensation silicones.²² In the late 1960s, a hydrophilic impression material, a polyether was introduced in the market. Its hydrophilic mechanical properties and good elastic recovery were superior to those of hydrocolloid and condensation silicones. Ten years later, PVS impression materials were introduced. They have gained popularity and acceptance in restorative dentistry because they produce accurate impressions, have superior elastic recovery, good dimensional stability, and sufficient tear strength.⁶

Impression materials can be classified according to setting reaction, material properties, clinical application, and composition. Two types of setting reactions exist in impression materials, reversible and irreversible. Reversible reactions occur in which the material can revert to its previous state after the setting. For example, impression compound and agar soften and solidify by temperature without chemical reaction. On the other hand, irreversible reactions imply the material cannot return to its original state after the setting reaction is complete, such as in alginate, zinc oxide eugenol and impression plaster, which are set by irreversible chemical reactions. In

addition, non-aqueous elastomeric impression material set by polymerization have irreversible reactions.²³

Another way of classifying impression materials is by their mechanical properties, elastic and inelastic. The elastic impression materials can deform and return to the original form when unstressed. They provide advantageous clinical application when making an impression of soft and hard tissue undercuts. Examples of this category are alginate, agar, and non-aqueous elastomeric impression materials. On the other hand, inelastic impression materials are brittle and fracture when deformed, such as ZOE, plaster, and impression compound. They can be used with edentulous patients in the absence of tissue undercuts. Their rigidity offers clinical advantages in making interocclusal records and jaw relations for accurate articulation.^{23,24}

The elastic impression materials can be stretch and return to it is original form upon removal from hard and soft tissue undercuts, unlike inelastic impression materials. Inelastic impression materials when handled properly provide an advantage when used with edentulous ridges, as they don't exert pressure that might deform the soft tissue for complete denture therapy.²³

POLYVINYL SILOXANE IMPRESSION MATERIALS (PVSs)

PVS is an elastomeric impression material that is available in extra light, light, medium, heavy, and extra heavy (putty) consistencies. It is composed of two components, a base and catalyst. The base material has a polymethyl hydrogen siloxane copolymer, which is a moderately low molecular mass polymer with silane terminal groups.²⁵ The catalyst material has vinyl-terminated polydimethyl siloxane, which is also considered to be a moderately low molecular mass polymer with vinyl terminal groups.²⁵ During the mixing process, an addition polymerization reaction

between the vinyl and silane groups occurs with a platinum salt exhibiting minimal dimensional changes after the polymerization reaction is completed with no by-products.⁶ However, residual polymethyl hydrogen siloxane in the impression material can lead to another reaction to produce hydrogen gas. The production of hydrogen gas occurs in presence of (–OH) groups that exist in the water (H-OH) and the silane terminal group (Si-H) in the polymethyl hydrogen siloxane.²⁵ The hydrogen gas does not affect the dimensional stability of the PVS, but it can lead to pinpoint voids in gypsum casts poured after removal of the PVS from the mouth.²³

PVSs produce detailed reproduction and good long-term stability.²⁶ Johnson et al. demonstrated that the addition silicone and polyether were least affected with delays of 1 h, 4 h, and 24 h in pouring the impression.²⁷ Therefore, they became the material of choice for highly precise restorations in fixed prosthodontics.⁴ One of the limitations of the PVSs is their hydrophobicity. To provide a degree of hydrophilicity to the impression surface, nonionic surfactant can be added to the impression paste during manufacturing, which would make the impression surface more wettable by water.

POLYETHER IMPRESSION MATERIAL

Polyether is provided in light, medium, and heavy consistencies. It is supplied in the form of base and catalyst. The base paste contains polyether copolymer, filler such as colloidal silica, and plasticizer such phthalate or glycol ether. The catalyst paste contains alkyl-aromatic sulfonate, plasticizer, and fillers. The main polyether chain is a copolymer of ethylene oxide and tetrahydrofuran with aziridine rings at the end of the branched polyether molecules. The polymerization reaction is initiated by the opening of the aziridine rings in the presence of the initiator and an aromatic sulfonate ester. This result in cross-linking by cationic polymerization via charged

ethylene imine terminals forming a chain propagation polymerization reaction with a larger molecule.^{22,23,28}

INTERIM FIXED PROSTHODONTICS MATERIALS (IFPM)

IFPMs provide numerous adjunct clinical benefits to definitive fixed prosthetics treatment. Currently, there is no ideal material for all clinical conditions. Therefore, material selection and fabrication techniques should meet treatment objectives and requirements based on comprehensive knowledge of the material science.²⁹

Interim materials can be classified based on fabrication techniques, chemical compositions, and activation of the chemical reactions. IFPM can polymerize directly in the patient's mouth or indirectly in the laboratory. Also, it can be used directly/indirectly in a process by which the IFPM is polymerized indirectly in the laboratory and then relined directly intraorally. Intraoral matrices made of silicone putty, hard wax, or heat preformed resin sheets are usually used for the direct clinical method after crowns preparation. The indirect method allows the IFPM to be constructed on a stone cast.³⁰ Several studies suggest reinforcing the IFPM by incorporating different materials such as a cast metal substructure,¹⁷ orthodontic wires,³¹ or fiber³² improves the mechanical properties. With the emergence of digital technology, milled IFPM can be fabricated with the assist of an intraoral scanner.³³

IFPMs have been divided into several categories based on their activation process: heat-activated acrylic resins, chemically activated acrylic resins, and dual- (light and chemical) activated acrylic resin.³⁴ There are two main types of acrylic resins available for IFPM: 1) methacrylate resin such as methylmethacrylate, ethylmethacrylate, vinylmethacrylate, butylmethacrylate, and 2) composite resin such as microfilled bisphenol A-glycidyl dimethacrylate (bis-GMA), bis-acryl, urethane

dimethacrylate (UDMA).

In the 1990s, flowable composite resin (FCR) was introduced in the market. Its desirable handling properties and injectability provided a simple placement procedure.³⁵⁻³⁷ The first generation of FCR had less filler content and higher volume of resin matrix when compared with nonflowable composite. Therefore, it was primarily used with cervical caries lesions due to a lower elastic modulus. Recent generations of FCR are claimed to have better mechanical properties due to their higher filler content, and they are indicated for large posterior caries lesions.^{38,39} Improvement of properties of the FCR and the bond strength of resin adhesives allow diverse applications of the flowable composite resin.

Dental erosive and abrasive lesions can have severe negative effect on the teeth. Early diagnosis and management can prevent debilitating factors such as the loss of tooth structure and subsequently the loss of occlusal vertical dimension (OVD). Prevention of progression of these lesions limits further destruction of the teeth and avoids comprehensive and expensive prosthetic care. A recent *in-vitro* study suggested that a conservative restorative of 150 µm FCR could provide sufficient protection against abrasive and erosive wear of enamel, dentin, and cementum for a period of two years.⁴⁰ Bartlett et al. in his *in-vivo* study evaluated the ability of the fissure sealant to protect palatal dentin surfaces of the maxillary anterior teeth. It was found that this conservative therapy could protect the palatal surfaces of the maxillary teeth from the erosive acids in the patient's mouth.⁴¹

Implant fixed supported prosthesis must fit the implants accurately to minimize the mechanical and biological complications. One of the factors that determine the prosthesis fit is the impression-making technique. There are several contradictory *in-vitro* and *in-vivo* studies concerning this factor.^{42,43} Papaspyridakos

et al. did a recent systematic review about different impression techniques with partially and completely edentulous patients. Seventy-six studies fulfilled their inclusion criteria featuring four clinical studies and 72 *in-vitro* studies. In completely edentulous patients, 22 laboratory studies and three clinical studies evaluated the accuracy of splinted implants impression copings and nonsplinted impression techniques. It was concluded from 12 *in-vitro* and three *in-vivo* studies that splinting of the impression copings is more accurate than nonsplinted impression techniques. However, nine *in-vitro* studies reported that there was no difference between both techniques, and one *in-vitro* study reported that the nonsplinted technique was more accurate than the splinted technique.⁴⁴ In a systematic review that included 17 studies comparing splinted and nonsplinted impression techniques, Lee et al. found similar conflicting outcomes. Seven studies reported more accurate results with splinted impression techniques; however, three studies found better accuracy without splinting, and seven studies reported no difference between the accuracy of splinted and nonsplinted techniques.⁴⁵ Therefore, a conclusive result cannot be reached from the literature due to the heterogeneity of those studies. Limitations were found with all studies in that they used chemically polymerized acrylic resins, and only one study used flowable composite to splint the impression coping. The studies also showed smaller three-dimensional linear distortion than the nonsplinted group during the fabrication of definitive casts.⁴⁶ Chemically polymerized acrylic resins have volumetric shrinkage of 6.5 percent to 7.9 percent after 24 hours with most of it occurring within 17 minutes of the fabrication.⁴⁷ Flowable composite resins have a high strength and low volumetric shrinkage and can be used for splinting impression copings before making the definitive impressions.^{48,49,50} Alqahtani et al. described a clinical technique to use flowable composite resin to splint impression copings with

partially edentulous patients before making a definitive impression.⁵¹

PVS INTERACTIONS WITH OTHER DENTAL MATERIALS

The accuracy of the definitive impression material is a crucial factor for reliable fabrication of indirect restorations and successful prosthetic rehabilitation.⁴ The selected impression material must provide precise and dimensionally stable replicas of the tooth preparation and the surrounding tissues to produce accurate working casts.⁵²⁻⁵⁴ Unfavorable interactions of the PVS with other dental materials have detrimental effects on the dimensional accuracy and surface definition of the working casts. It has been well documented that latex protective barriers such as rubber dam or gloves interfere with the polymerization reaction of the PVS impression materials. It was suspected earlier that corn starch powder used as a lubricant in gloves was the primary cause for the interaction.⁵⁵ A sulfur compound, zinc diethyl dithiocarbamate, has been identified as being a responsible factor for the polymerization inhibition. It is a preservative and vulcanizing accelerator used during latex gloves manufacturing.⁵⁶ Indirect contact of the PVS with latex has been reported to inhibit the polymerization reaction of the PVS.^{57,58} Kimoto et al. confirmed the indirect polymerization inhibition of the PVS with the sulfur containing particles, and there were no decontamination methods able to remove the contamination. Displacement cord medicaments such as ferric sulfate, ferric subsulfate, and aluminum chloride have been shown to inhibit the polymerization reaction of PVS.⁵⁹ They mostly affect the sulcular region of the impression.⁹ However, aluminum sulfate and epinephrine have no inhibitory effect on the PVS.¹⁰

Published reports have documented silicon contaminations to the bonding surface from fit-indicator⁶⁰ or PVS impression,^{61,62} which have negative effects on the bond strength and durability of the bonded restorations. PVS was advocated to be

used as a matrix to support composite resin when restoring missing parts of a tooth structure. A recent study by Chen et al. showed an unpolymerized composite resin surface when it was polymerized in contact with the PVS impression.⁶³ The degree of conversion and the Vickers microhardness values of the tested group were significantly lower than the control group, and the scanning electron microscope (SEM) showed 10 μm of the unpolymerized composite resin.

Moreover, resin-based restorative materials have an inhibitory effect on the polymerization reactions of the PVS. Some authors claimed that the oxygen-inhibited layer that forms on the surface of the material is a primary cause for the inhibition reactions.^{64,65} However, other studies have been shown to exclude the oxygen-inhibited layer as a possible cause.^{7,19,20} FCR had a significant effect on the PVS polymerization and continued to show inhibition even after removing the oxygen-inhibited layer.¹⁹ It was documented that certain types of IFPM such as bis-acryl, bis-glycidyl methacrylate and rubberized urethane cause strong inhibition after indirect contact with PVS.²⁰ Mild inhibition of PVS was also reported with polymethyl methacrylate and polyethyl methacrylate. In addition, polyether impression material did not have any polymerization inhibition and its surface showed complete polymerization when direct contact occurred with resin-based restorative materials.

Previous studies evaluated visually the PVS polymerization inhibition with flowable composite resins and interim fixed prosthodontics materials. The aim of this study was to test the inhibitory effect of certain types of interim fixed prosthodontic materials and flowable composite materials with polyvinyl siloxane impressions as quantified by weight change and qualified by FTIR spectroscopy.

MATERIALS AND METHODS

PART I

Material Selection and Sample Preparation for the Weight Change Test

The weight change measurements of the PVS and restorative materials were performed in a constant temperature room (21°C) with filtered ambient light that absorbed wavelengths between 380 nm to 520 nm. A fully charged multiple emission peak light emitting diode (LED) light curing unit (LCU) (Bluephase Style, Ivoclar Vivadent, Amherst, NY) that operated in the 385 nm to 515 nm wavelength range was used (Figure 1). The LCU was mounted on a Managing Accurate Resin Curing System-Resin Calibrator MARC-RC system (BlueLight Analytics, Halifax, Canada) to standardize a 2-mm distance between the light guide tip and the 4-mm bottom sensor using a mechanical arm with a metric gauge (Figure 2). The LCU tip was aligned with the MARC-RC crosshead on the surface positioned flat and perpendicular against the sensor to standardize the x-, y-, and z-directions (Figure 3). Specimens were made of microfilled flowable composite resin (FCR) Heliomolar Flow (HM, Ivoclar Vivadent, Schaan, Liechtenstein), and Flows Rite (FR, Pulpdent, Watertown, MA) with a square customized Delrin plastic mold 5×5×2 mm (Table I) (Figure 4).

Sixty specimens of FCR were fabricated and assigned to six groups (n = 10) (Figure 5). The mold was fabricated to fit the shape of the MARC-RC bottom sensor in order to prevent mold rotation and standardize the position of the specimens during the light curing. Mylar strips (Matrix Strips, DuPont MYLAR, Chester, VA) of 0.002-mm thickness were placed under the mold and the selected FCR was injected (Figure

6). One (1) mm-thick glass slides were also placed over the mold to condense and remove excess material. Then, the light curing time was set according to manufacturer instructions. In order to remove the oxygen-inhibited layer, the specimens were finished using a Struers RotoForce- 4 polishing unit (Struers, Ballerup, Denmark) with 1200-, 2400- and 4000-grit silicon carbide abrasive paper for 1 second, 8 seconds, and 19 seconds respectively. Then, specimens were rinsed with de-ionized water for 3 minutes, sonicated with de-ionized water for 3 minutes in an ultrasonic cleaning system (L&R Manufacturing Company, Kearny, NJ), and air-dried before testing with the selected elastomeric impression material. Throughout the fabrication process, powder-free nitrile gloves (Halyard, LLC, Alpharetta, GA) were used.

The enamel bovine blocks were used as negative controls in this *in-vitro* study (n = 10). Bovine incisor teeth were obtained from Tri State Beef Co. (Ohio). Teeth with surface flaws or cracks were excluded before preparation. Buccal surfaces of the crowns were cut into 5 x 5 x 2 mm specimens under continuous de-ionized water irrigation with a Buehler Isomet low speed saw (Isomet, Buehler Ltd, Lake Bluff). Superficial enamel irregularities were removed to establish a flat enamel surface using 1200-, 2400- and 4000-grit silicon carbide abrasive papers for 1 second, 8 seconds, and 19 seconds respectively while mounted on a Struers Rotopol 31 polishing unit (Struers). Each specimen was rinsed with de-ionized water for 3 minutes, sonicated with de-ionized water for 3 minutes in an ultrasonic cleaning system (L&R Manufacturing Company), and then stored at 4 °C under moist conditions in a sealable, labeled container. Moisture was provided by 0.1-percent thymol applied to paper towels laid in the bottom of the container, with DI water on top of the specimens until testing.

Weight Changes

Material loss of the PVS caused by inhibitory effect was quantified with a calibrated 0.0001-mg digital analytical balance (Mettler Toledo, Columbus, OH) (Figure 7). The scale pan was wiped with ethanol before each use. Baseline weights (S) for each restorative material specimen were determined before applying the impression materials. A polystyrene weighing dish (Fisher Scientific, Pittsburg, PA) was used to carry the specimen before the weighing (Figure 8). The specimen was placed into a circular plastic mold with dimension of 10 x 5 mm over a clean glass slab that was rinsed with DI for 5 minutes to standardize the application of the impression material (Figure 9). Two brands of light body PVS impression material Virtual, (VIT, Ivoclar Vivodent, Schaan, Liechtenstein), and Correct Plus, (CP, Pentron Clinical, Orange, CA) and one brand of polyether impression material Impregum Garant light body polyether impression (IMP, 3M ESPE, St. Paul, MN) were tested independently with each brand of FCR in this study (Table II). Then impression materials were applied on the surface of the polished specimens with GC Cartridge Dispenser II (GC America, Tokyo, Japan) after squeezing the initial mix out on a glass slab (Figure 10). The tested specimen and the impression material (Impression + specimen) were weighed together (SP) (Figure 11) to determine the pre-application weight of the impression material (P1) by applying the following equation:

$$P1 = PS - S$$

The Fourier transform infrared spectrophotometer (Jasco Inc., Tokyo, Japan) with a diamond attenuated total reflectance (ATR-MIRacle, Pike Technologies, Madison, WI) (FTIR-ATR) accessory device was used to determine the removal time of the PVS impression material based on the time when the peaks of the reacted

silicone hydride groups (Si-H) no longer changed in height, which was a representation of complete polymerization of the impression material. Each PVS impression (n = 3) was studied with FTIR at the time periods of 0 min, 3 min, 5 min, 10 min, 12 min, 13 min, 20 min, and 40 min after mixing. For the VIT, the Si-H group was stable at 12 minutes and it was at the same height as the completely polymerized PVS at 40 minutes. The Si-H group of the CP impression was stable at 10 minutes. An extra 2 minutes were added to each polymerization time of VIT and CP to be 14 and 12 minutes, respectively. For the polyether IMP, no reacted peaks were found; therefore, the manufacturer's recommendation time was multiplied by three (10 minutes) to ensure complete polymerization of the impression.

The impression surface that was in contact with the specimen was wiped off with a dry cotton tipped applicator (Henry Schein Inc., Melville, NY) three times to remove any inhibited impression material. The wiping was confirmed by FTIR-ATR to ensure removal of the inhibited impression material (Figure 12). After that, reweighing of the impression was done to determine the post-application weight (P2). The difference between the pre-application weight of the impression (P1) and post-application weight (P2) was considered as the amount of total inhibited impression (P3) by applying the following equation:

$$P3 = P1 - P2$$

The negative control group was specimens of bovine enamel (n = 10) that were tested with polyether IMP. Specimens were rinsed with DI for 3 minutes and then air-dried for 30 seconds before testing. The control group was tested following the same method mentioned previously. After that, the weight loss percentage was calculated.

PART II

Material Selection and Sample Preparation for Polymerization Inhibition Testing

The Fourier transform infrared spectroscopy (Jasco Inc.) with a diamond attenuated total reflectance (ATR-MIRacle) (FTIR-ATR) accessory device was used to explore the presence of inhibited PVS material when they come into contact with interim fixed prosthodontic materials or flowable composite materials (Figure 13). The test was performed in a constant temperature room (21°C) with filtered ambient light that absorbed wavelengths between 380 nm to 520 nm.

One hundred and fifty specimens were assigned to 15 groups made of five different substrates, two IFPMs two FCRs, and enamel from bovine teeth (Figure 14). The two types of IFPM selected for this testing were bis-acryl chemically activated (IN, Integrity, DENTSPLY International, York, PA, USA), and rubberized urethane dual activated Tuff-Temp Plus (TT, Pulpdent, Watertown, MA) (Table III). The FRC specimens were HM and FR. For the control group, enamel specimens of bovine teeth were utilized.

A fully charged multiple emission peak light-emitting diode (LED) light curing unit (LCU) (Bluephase Style) that operated with a 385 nm to 515 nm wavelength range was used. The LCU was mounted in a MARC-RC system (BlueLight Analytics) to standardize a 2-mm distance between the light guide tip and the 3-mm top sensor using a mechanical arm with a metric gauge. The LCU tip was aligned with the MARC-RC crosshead on the surface, then positioned flat and perpendicular against the sensor to standardize the x-, y-, and z-directions (Figure 15). Plastic Shim paper of 0.254-mm thickness with circular 6-mm diameter was used as a mold (Precision Brand Products, Inc., Downer Grove, IL). Mylar strips (Matrix Strips) of 0.002-mm thickness were placed under the mold. Then, the Shim paper was

stabilized with adhesive tape to prevent mold rotation and standardize the position of the specimens during the light cure (Figure 16). The selected FCR or IFPM was injected. One-mm-thick glass slides were also placed over the mold to condense and remove excess material. Then, the light curing time was set according to manufacturer's instructions (Figure 17).

Polymerization Inhibitions Testing using Fourier Transform Infrared Spectroscopy Measurements (FTIR)

The ATR-FTIR diamond was cleaned with an ethanol wipe (Figure 18). The spectra were taken for all unset PVS materials to determine the peaks of the unset impression materials. Spectra of the unset materials were recorded as follows: The edge of a thin 0.05-mm plastic Shim paper (Precision Brand Products, Inc.) was placed vertically on the diamond surface, separating the test area of the refractive element into two equal parts. Then, each part was covered with the corresponding unset material (base or catalyst) from each PVS impression. The spectra taken under these conditions corresponded to a 1:1 component ratio, without mixing to prevent activating the polymerization reaction. All spectra were subjected to baseline, ATR corrections, and the net peak absorbance intensities to locate the silicon hydride groups Si-H and methylsilyl groups Si-CH₃. The peaks of the reacted silicon hydride (P1) groups (Si-H) at 2158, which were reduced during polymerization, was selected as the analytical band, and the height of this band was measured, whereas the peaks of the (P2) groups (Si-CH₃) at 1258 that do not change during polymerization were chosen as the reference band. For the polyether IMP, two peaks of tertiary amine salts in the catalyst were located and measured. The peak height at 2794 does not have any overlap with FCR and IFPM specimens and it was considered as a reference peak (P2). While the peak height at 2856 (P1) had potential overlap with FCR and IFPM

specimens, it was considered as a reacted peak, because it was enhanced only after it came to into contact with the specimens. Then, the impression materials were applied as described in Part I.

The tested surface of the selected restorative material was studied with the FTIR to locate and measure PVS and polyether peaks heights of (P1) and (P2) groups. Then, the ratio of P2/P1 was calculated to evaluate the presence of inhibited impression materials.

Specimens made of bovine enamel were prepared as described in the weight loss test and were used as a control group with VIT, CP and IMP. The specimens were tested with the FTIR to locate peaks related to impression materials.

Microscopic evaluations of the inhibited specimens were made with light microscopy (Leica MZ 125, Leica Microsystems GmbH, Wetzlar, Germany). Images were captured with the integrated Leica DFC290 camera (Leica Microsystems GmbH) and processed with Leica Application Suite Software (Leica Microsystems GmbH). Selected specimens were chosen to be examined under reflective microscopy (Leco Metallograph, St. Joseph, MI).

Statistical Analysis

One-way ANOVA was used to compare the FCR -PVS combinations for differences in weight change. Two-way ANOVA was used for differences in P2/P1 ratios of the impression materials. A 5-percent significance level was used for all tests. Sample size justification: With a sample size of 10 for each FCR -PVS combination, the study had 80-percent power to detect a weight change difference of 1 mg between any two substrate-PVS combinations, assuming two-sided tests each conducted at a 5-percent significance level and a within-group standard deviation of 15. With a sample size of 10 for each substrate-PVS combination, the study had 80-

percent power to detect a 60-percent difference in the percentage of samples showing the presence of inhibition, assuming two-sided tests each conducted at a 5-percent significance level.

RESULTS

PART I

Weight Change

One-way ANOVA with effects for impression materials/flowable composite composite resins (FCR) material group (CP-FR, CP-HM, IMP-BOV (Control), IMP-FR, IMP-HM, VIT-FR, VIT-HM) for the outcome weight loss showed a statistical difference in weight loss (<0.0001). Mean values of weight loss (%) and standard deviation (%) are showing in Table IV. PVS CP impression material with FR FCR showed the highest amount of weight loss followed by PVS VIT impression material with FR FCR. Moreover, polyether IMP impression material with FR FCR was not statistically significant from the control group (IMP-Bov) (Figure 19).

PART II

Polymerization Inhibition Testing using Fourier Transform Infrared Spectroscopy Measurements (FTIR)

Two-way ANOVA with effects for impression material (CP, VIT, IMP), and flowable composite resins (FCR) or interim fixed prosthodontics (IFPM) materials (FR, HM, TT, IN, Control) for the outcome P2/P1 ratio showed statistical difference (<0.0001). The summary of the basic statistical analysis is shown in Table V. FTIR – ATR was able to detect inhibited impression material on the surface of the experimental specimens of FCR and IFPM. PVS VIT impression material with FR FCR showed the highest significant difference among the groups, followed by PVS CP impression material with TT IFPM. In addition, polyether IMP impression material was not statistically significant from the control group and didn't cause any inhibitory effect with any of the experimental specimens (Figure 20).

PVS VIT, CP impression materials were inhibited when in contact with FCR (FR, HM) and IFPM (TT, IN). PVS VIT impression material peak height of Si-H (P1) at 2158 and Si-CH₃ (P2) at 1258 were detected when tested by FTIR-ATR. None of these peaks were evident on the control bovine specimens (Figure 21 and Figure 22). For the polyether impression material (IMP), peaks of completely polymerized impression material were evident on the experimental specimens that were reduced in height (FR, HM, TT and IN) and on the surface of the control group (BOV) (Figure 23).

The microscopic evaluations of the experimental specimens FCR (FR, HM) and IFPM (TT, IN) made with microscopy confirmed the inhibition of the PVS impression material (VIT, CP) on the surface of the specimens (Figure 24 and Figure 25). FRC (FR, HM) and IFPM (TT, IN) showed matte surfaces after the application of polyether impression material, and there was no evidence of inhibited impression material on the surface of experimental specimens (Figure 26).

Further examination of selected specimens, made by reflective microscopy at a higher magnification, revealed similar inhibition patterns for the PVS impression materials (VIT, CP). Moreover, completely polymerized polyether impression (IMP) was found in the small surface microporosities of the experimental specimens, which explains the matte surfaces that were found with the microscopic evaluation (Figure 27 and Figure 28).

TABLES AND FIGURES

TABLE I
Flowable composite resins type and composition

Product Name	Manufacturer	Type	Shade	Compositions
Heliomolar Flow (HM)	Ivoclar Vivadent, Schaan, Liechtenstein	Microfilled	A1	Bis-GMA <20% Ytterbium trifluoride 10-13% Urethane dimethacrylate 3-5 % 1,10-decandiol dimethacrylate 3-5 %
Flows Rite (FR)	Pulpdent, Watertown, MA	Microfilled	A1	Uncured methacrylate ester monomers 55-65% Amorphous silica 5%

TABLE II
Impression materials type and consistency

Product Name	Manufacturer	Type	Consistency
Virtual (VIT)	Ivoclar Vivodent, Schaan, Liechtenstein	PVS	Light body
Correct Plus (CP)	Pentron Clinical, Orange, CA	PVS	Light body
Impregum Garant (IMP)	3 M ESPE, St. Paul, MN	Polyether	Light body

TABLE III
Interim fixed prosthodontics materials types and compositions

Product Name	Manufacturer	Type	Shade	Compositions
Integrity	DENTSPLY International, York, PA	Chemically polymerize Bis-Acryl	A1	Barium glass >35% Silica amorphous, fumed <10 % Glycol methacrylate > 35 % Multifunctional methacrylates <15 % Malonylurea derivative <1 % Sulfur Oxides
Tuff-Temp Plus	Pulpdent, Watertown, MA	Dual cure rubberized urethane	A1	Uncured methacrylate ester monomers 55-65% Amorphous silica 5%

TABLE IV
Summary of the basic statistical analysis for the weight loss

Impression Material	Flowable Material	N	Mean (mg)	SD (mg)	SE (mg)	Media n (mg)	IQR (mg)		Min (mg)	Max (mg)
CP	FR	10	3.9060	0.6042	0.1911	3.9600	3.4500	4.1500	3.0500	4.9100
CP	HM	10	1.1690	0.1797	0.0568	1.1650	1.0500	1.2600	0.8900	1.5500
IMP	BOV (Control)	10	0.0750	0.0378	0.0119	0.0750	0.0400	0.1000	0.0300	0.1300
IMP	FR	10	0.1620	0.0688	0.0217	0.1700	0.1200	0.2100	0.0500	0.2800
IMP	HM	10	0.1120	0.0585	0.0185	0.1100	0.0700	0.1800	0.0200	0.1900
VIT	FR	10	1.5770	0.3377	0.1068	1.4850	1.3500	1.7700	1.2300	2.2900
VIT	HM	10	0.4480	0.1323	0.0418	0.4550	0.3500	0.5400	0.2200	0.6400

TABLE V
Summary of the basic statistical analysis for the P2/P1 ratio

Impression Material	Flowable or Provisional Material	N	Mean (%)	SD (%)	SE (%)	Median (%)	IQR (%)		Min (%)	Max (%)
CP	FR	10	58.56	4.91	1.55	57.40	54.40	62.40	53.71	66.81
CP	HM	10	33.52	6.29	1.99	33.88	29.88	37.67	22.37	44.52
CP	IN	10	38.77	7.22	2.28	39.76	33.47	41.31	27.49	50.69
CP	TT	10	32.13	14.27	4.51	27.87	22.79	45.29	12.43	52.03
IMP	FR	10	30.79	13.81	4.37	29.60	20.42	40.33	11.83	51.23
IMP	HM	10	35.16	8.45	2.67	31.82	28.94	43.14	25.21	49.59
IMP	IN	10	55.59	4.42	1.40	56.15	54.68	57.33	45.93	62.70
IMP	TT	10	78.80	3.54	1.12	79.02	76.75	81.84	71.94	83.33
VIT	FR	10	36.33	11.42	3.61	39.22	24.33	46.43	18.45	49.39
VIT	HM	10	22.50	2.99	0.95	23.64	19.61	25.03	17.75	25.95
VIT	IN	10	38.78	6.96	2.20	39.75	33.86	44.24	28.80	48.71
VIT	TT	10	29.42	16.43	5.20	23.85	22.62	28.34	18.15	74.94
Control	FR	10	36.33	5.42	1.71	36.34	31.71	39.20	30.37	48.30
Control	HM	10	35.16	8.45	2.67	31.83	28.94	43.14	25.21	49.59
Control	IN	10	54.92	4.64	1.47	55.55	52.93	57.77	45.93	61.61
Control	TT	10	66.37	3.36	1.06	66.69	63.71	69.42	61.21	71.08



FIGURE 1. Multiple emission peak light emitting diode (LED) light curing unit (LCU).

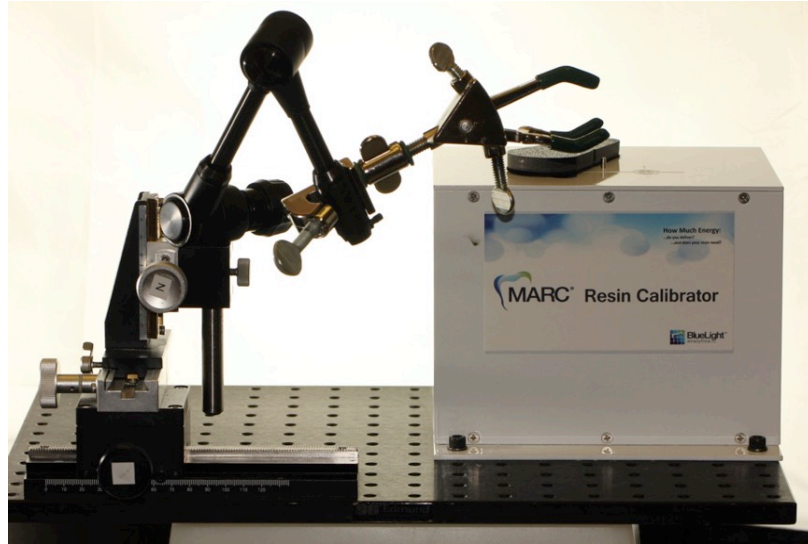


FIGURE 2. The MARC-RC System.



FIGURE 3. LCU tip was aligned with the MARC-RC crosshead on the surface positioned flat and perpendicular against the sensor to standardize the x-, y-, and z-directions.



FIGURE 4. A square customized Delrin plastic mold $5 \times 5 \times 2$ mm.

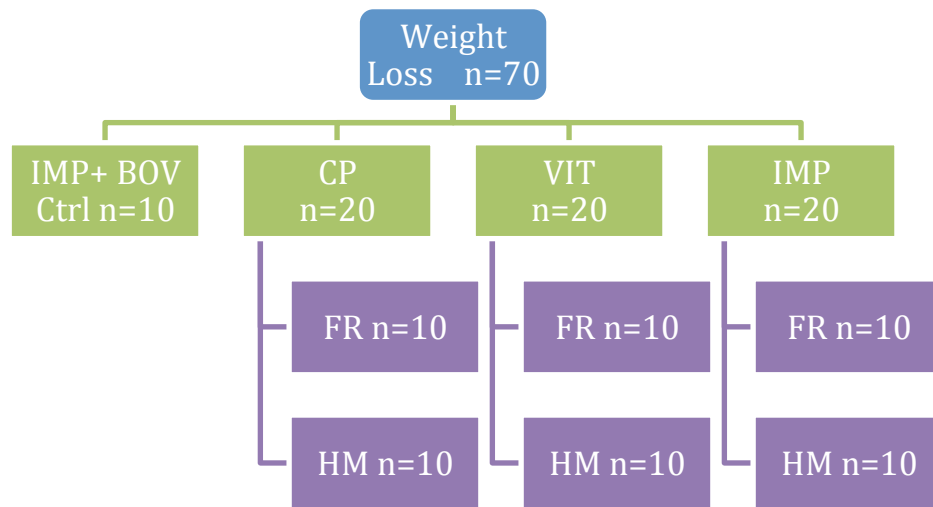


FIGURE 5. Sixty specimens of flowable composite were fabricated and assigned to six groups (n = 10).

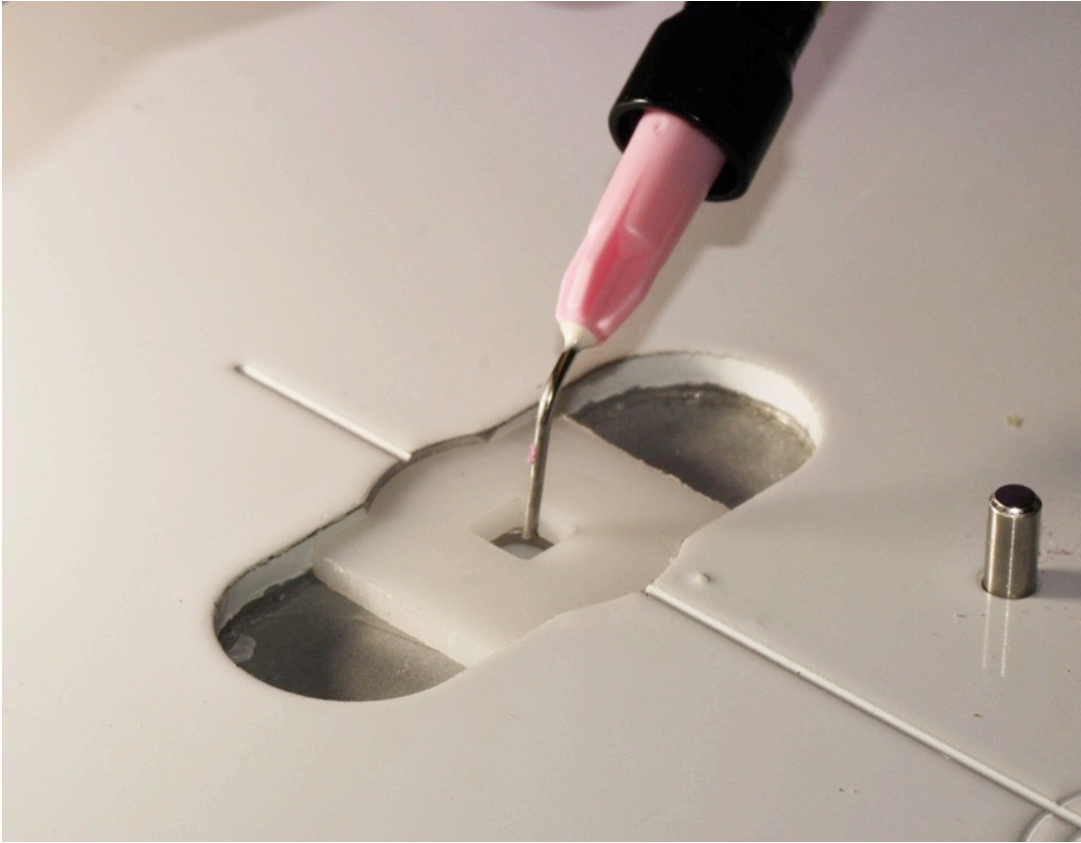


FIGURE 6. The selected FCR was injected.



FIGURE 7. Digital analytical balance (Mettler Toledo, Columbus, OH).

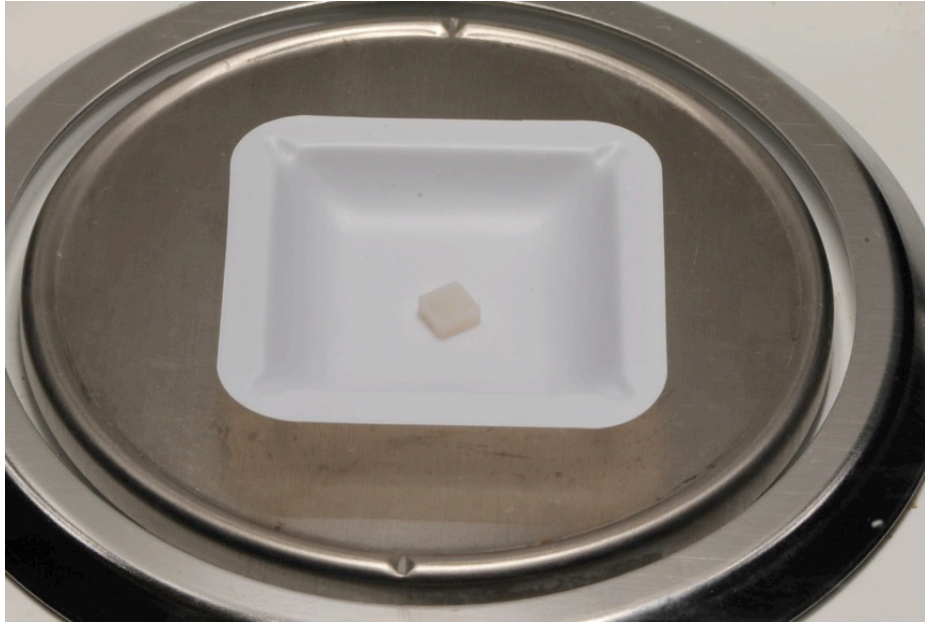


FIGURE 8. Polystyrene weighing dish was used to carry the specimen before the weighing.

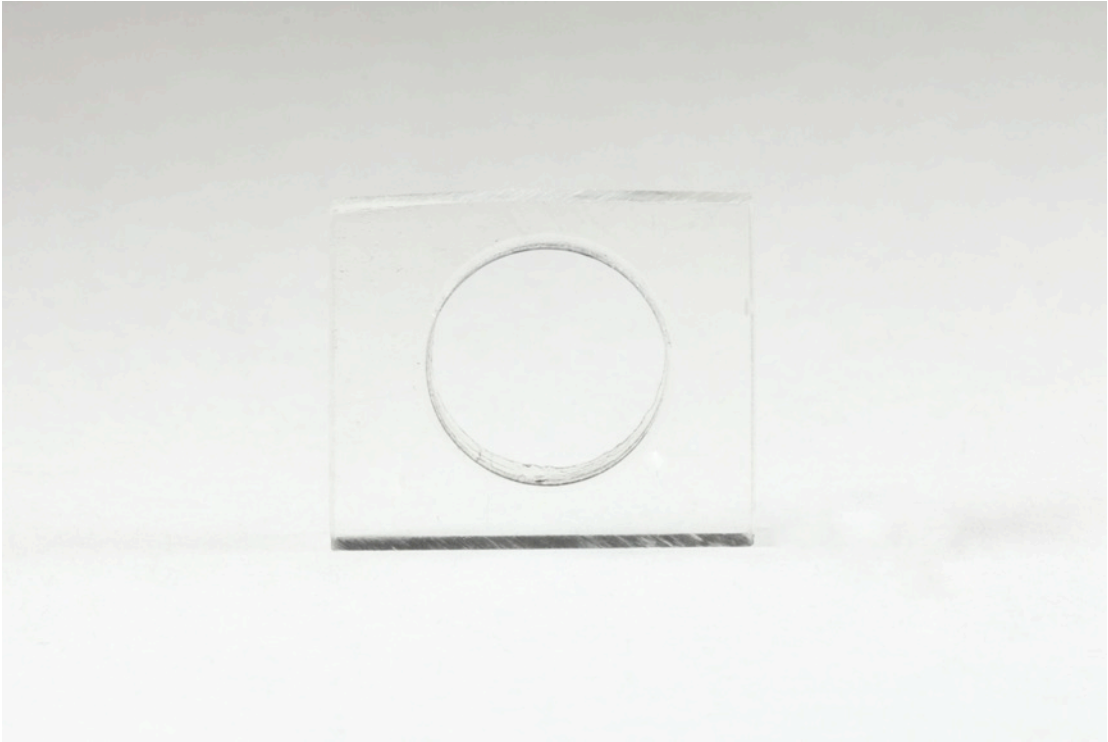


FIGURE 9. Circular plastic mold with dimension of 10 x 5 mm.

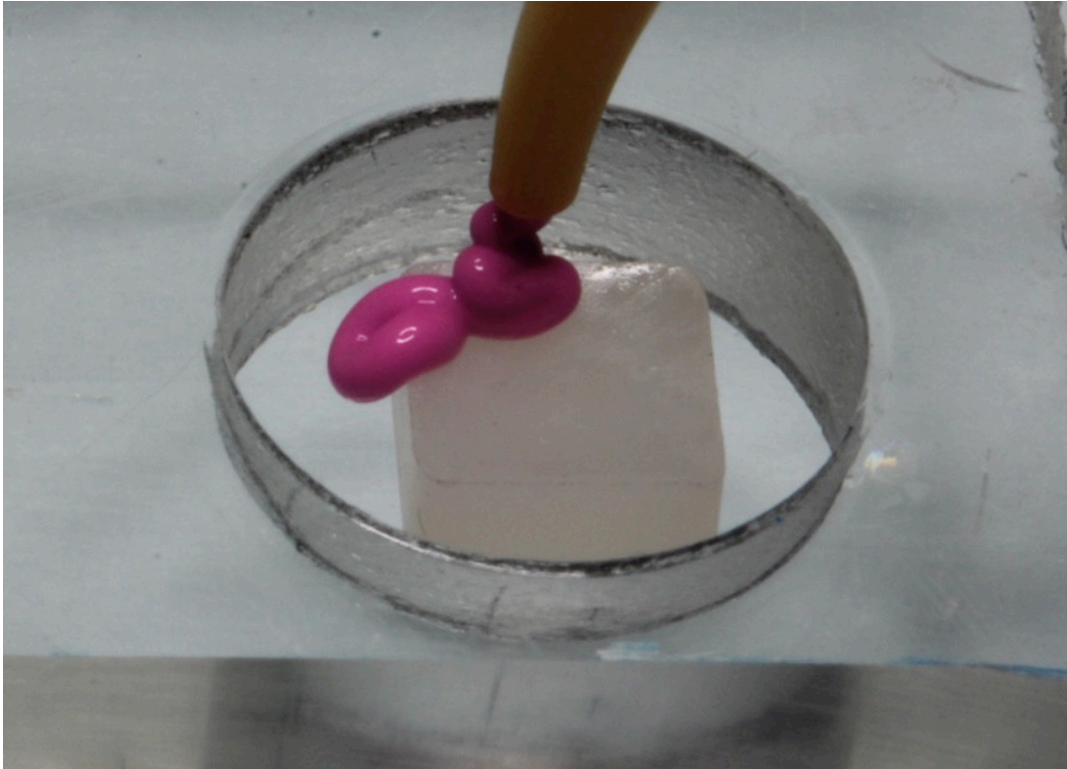


FIGURE 10. Impression materials were applied on the surface of the polished specimen with GC Cartridge Dispenser II.



FIGURE 11. The tested specimen and the impression material (Impression + specimen) were weighed together (SP) to determine the pre-application weight of the impression material (P1).

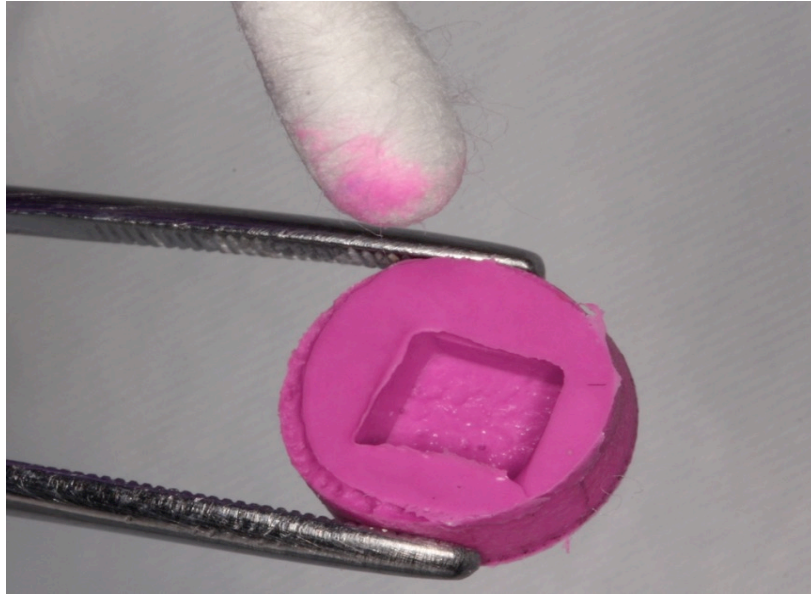


FIGURE 12. The impression surface that was in contact with the specimen was wiped off with a dry cotton-tipped applicator.



FIGURE 13. The Fourier transform infrared spectrophotometer with a diamond attenuated total reflectance attachment (FTIR-ATR).

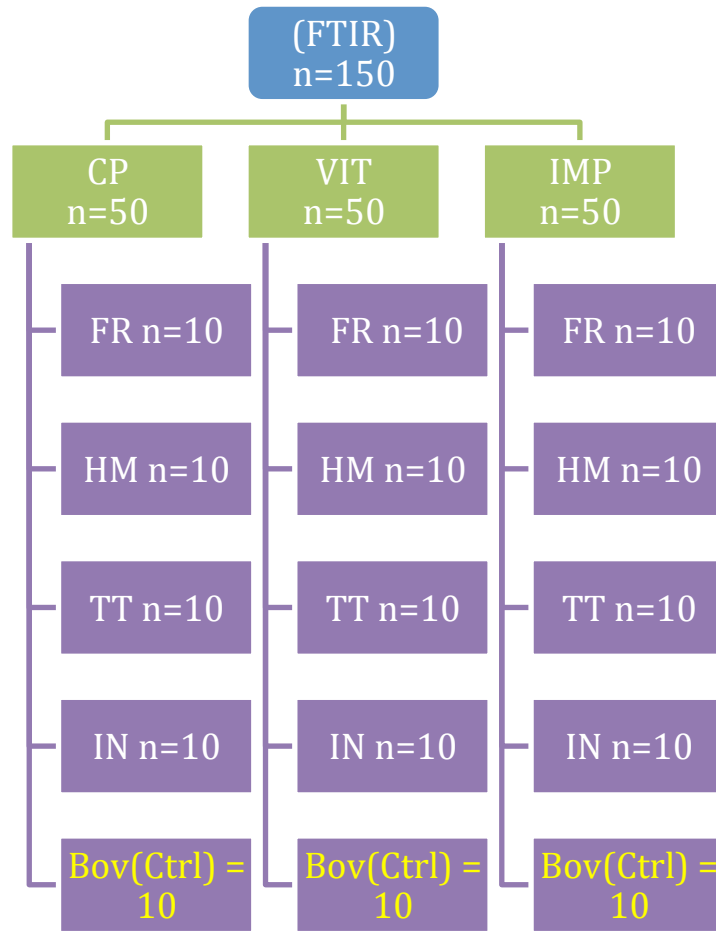


FIGURE 11. One hundred and fifty specimens assigned to 15 groups.



FIGURE 15. LCU tip was aligned and positioned flat and perpendicular against the MARC-RC sensor to standardize the x-, y-, and z-directions.

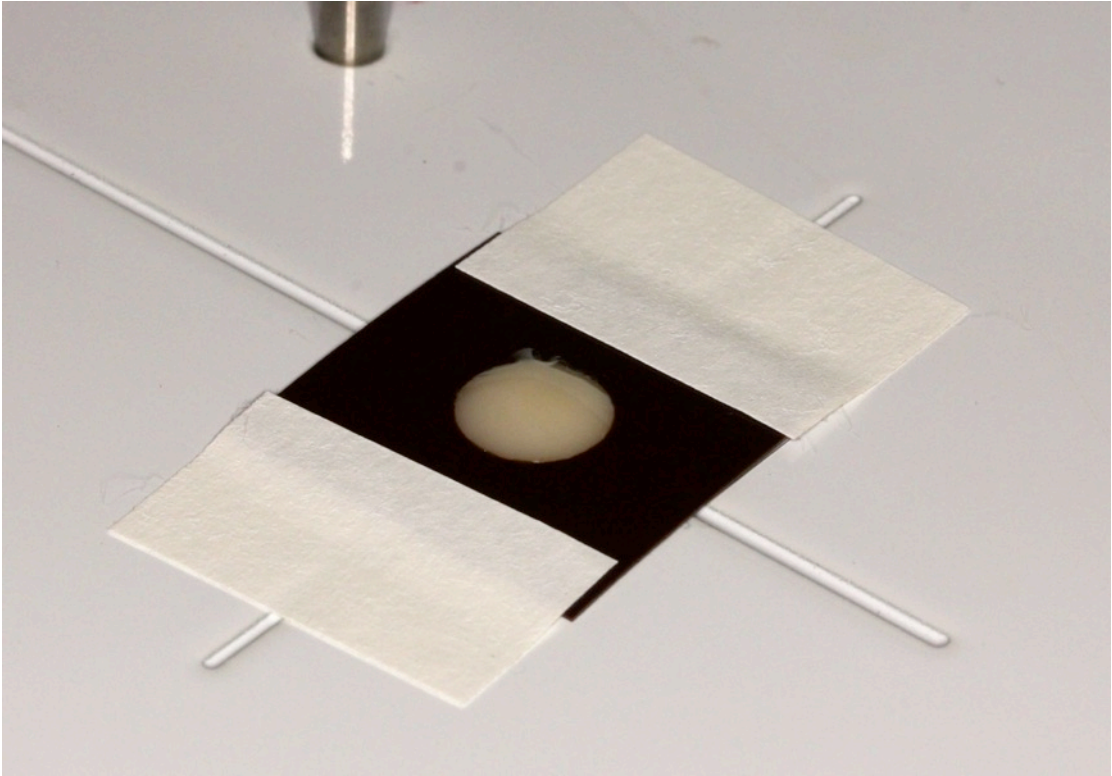


FIGURE 12. Shim paper was stabilized with adhesive tape.

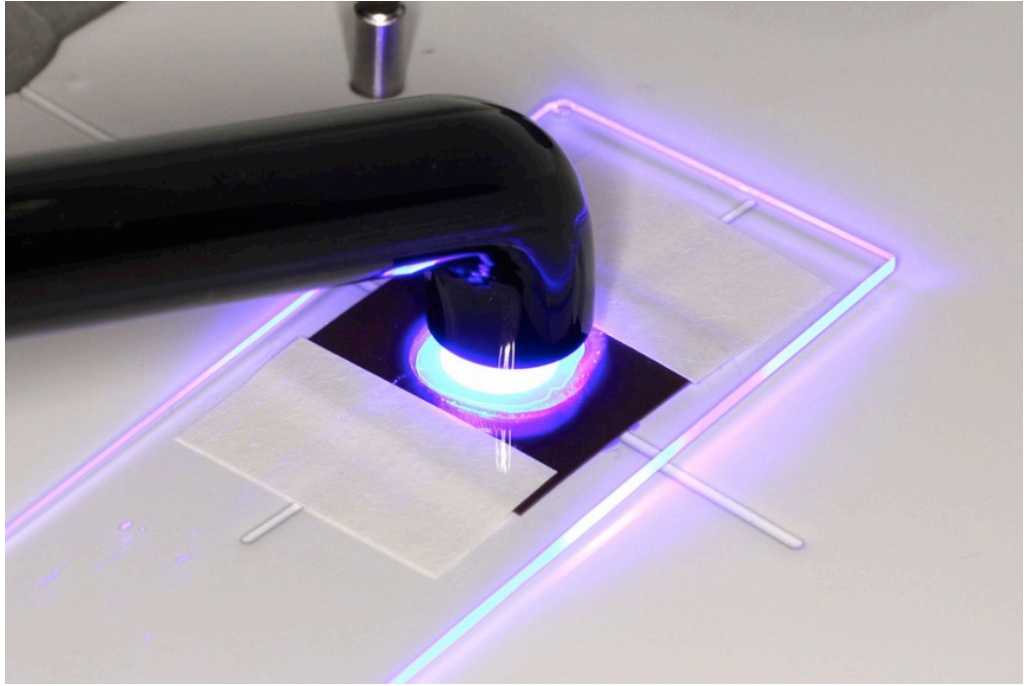


FIGURE 13. Light curing time was set according to manufacturer's instructions.



FIGURE 14. The ATR-FTIR diamond was cleaned with an ethanol wipe.

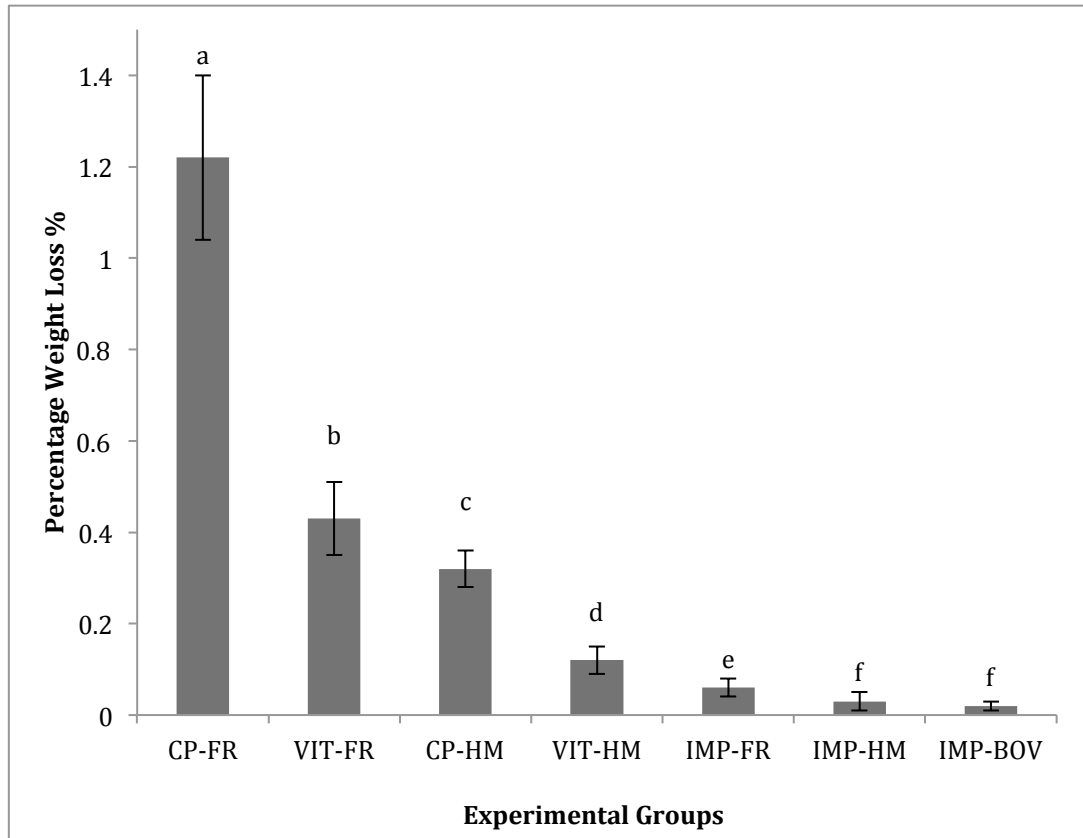


FIGURE 15. Mean and standard deviations for the amount of weight loss (%). Different lowercase letters indicate statistically significant differences in the amount of weight loss.

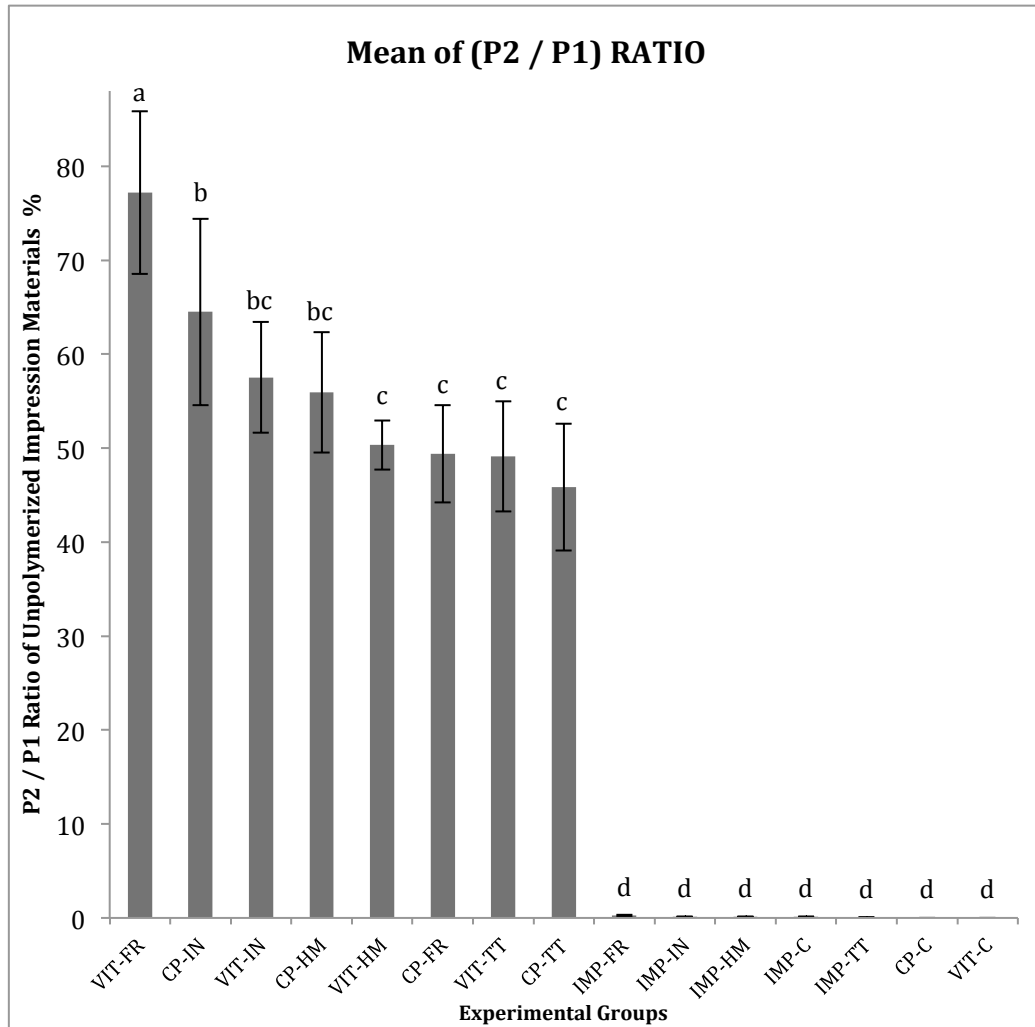


FIGURE 20. Mean and standard of errors for the P2/P1 ratio of unpolymerized impression material (%). Different lowercase letters indicate statistically significant differences in the P2/P1 ratio.

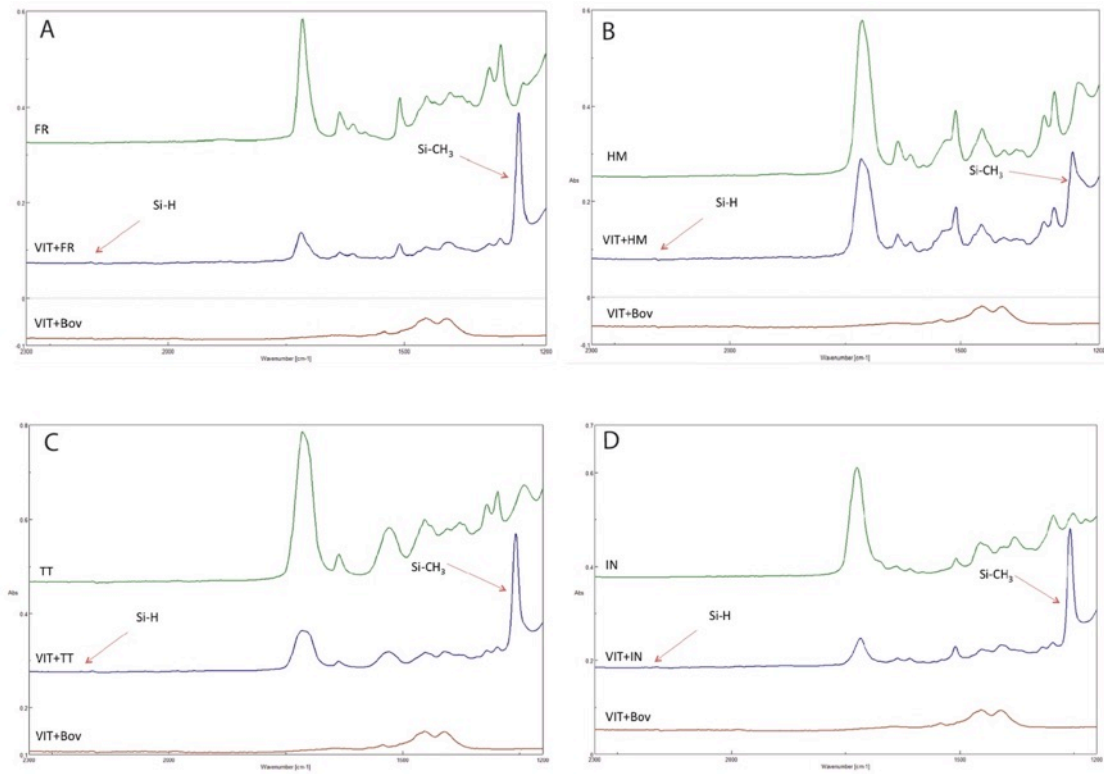


FIGURE 21. Three spectral evaluations are shown per experimental group. From the bottom to the top, control after contact with VIT, experimental specimen with VIT, and experimental specimen alone. A) FR FRC; B) HM FRC; C) TT IFPM; D) IN IFPM.

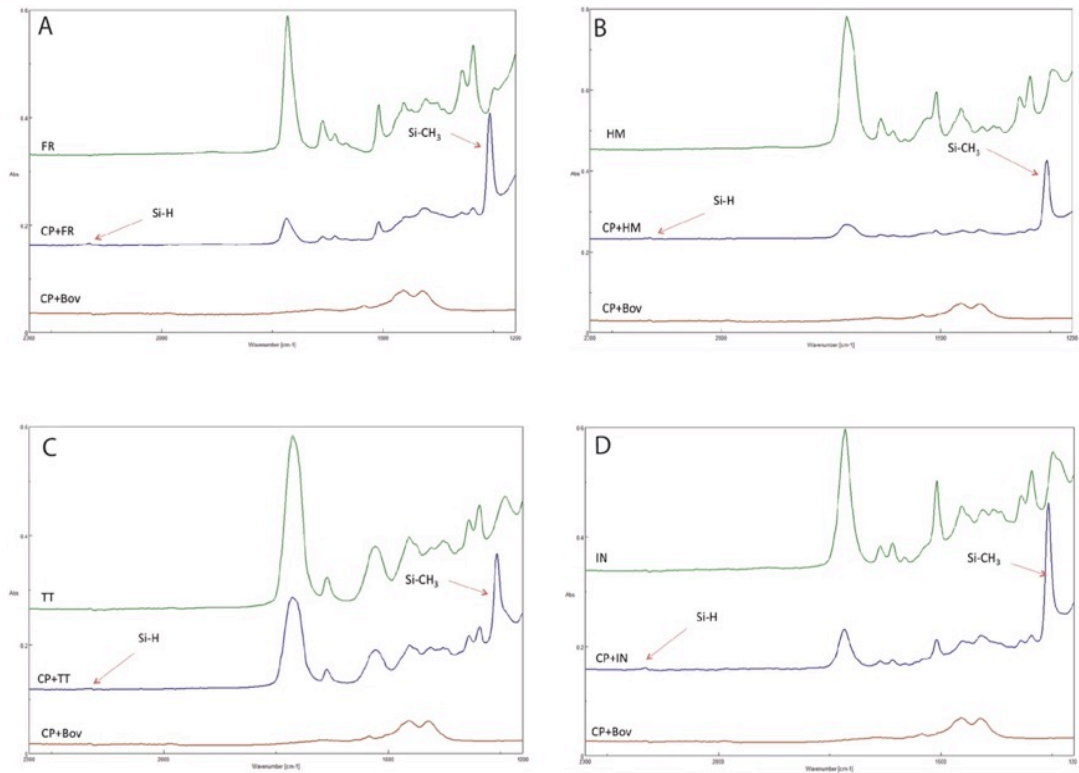


FIGURE 22. Three spectral evaluations are showing per experimental group. From the bottom to the top, control after contact with CP, experimental specimen with CP, and experimental specimen alone. A) FR FRC; B) HM FRC; C) TT IFPM; D) IN IFPM.

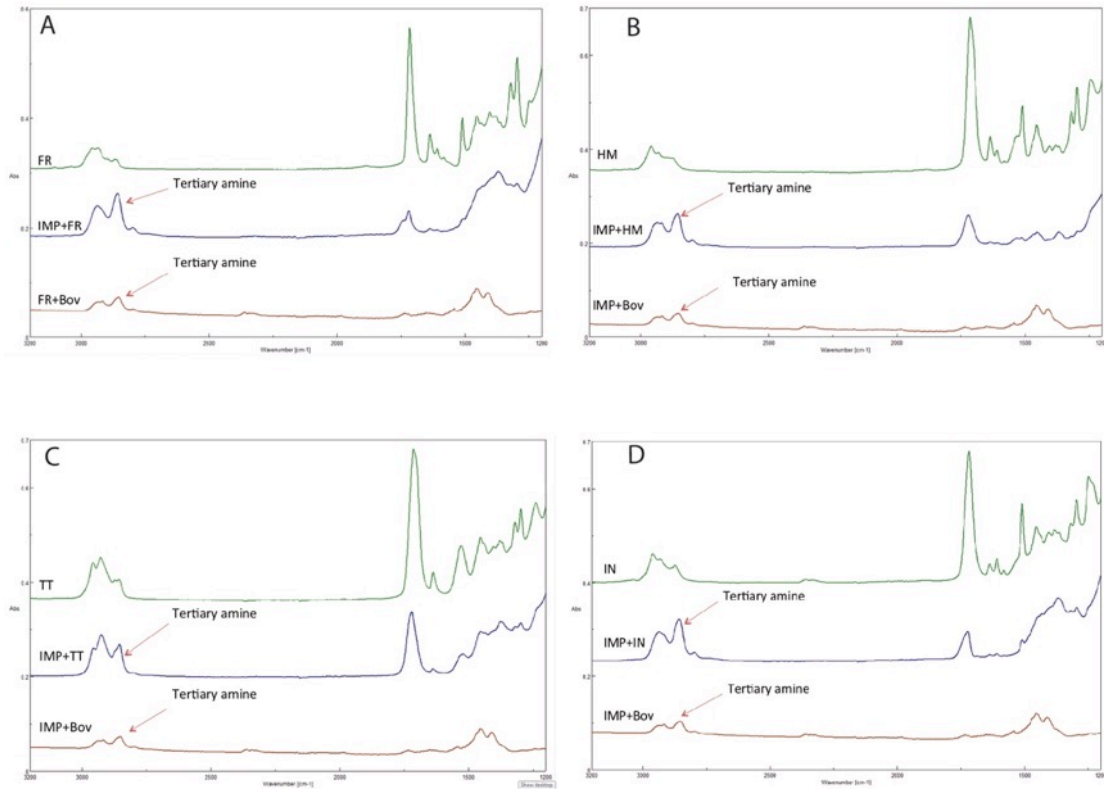


FIGURE 23. Three spectral evaluations are showing per experimental group. From the bottom to the top, control after contact with IMP, experimental specimen with IMP, and experimental specimen alone. A) FR FRC; B) HM FRC; C) TT IFPM; D) IN IFPM.

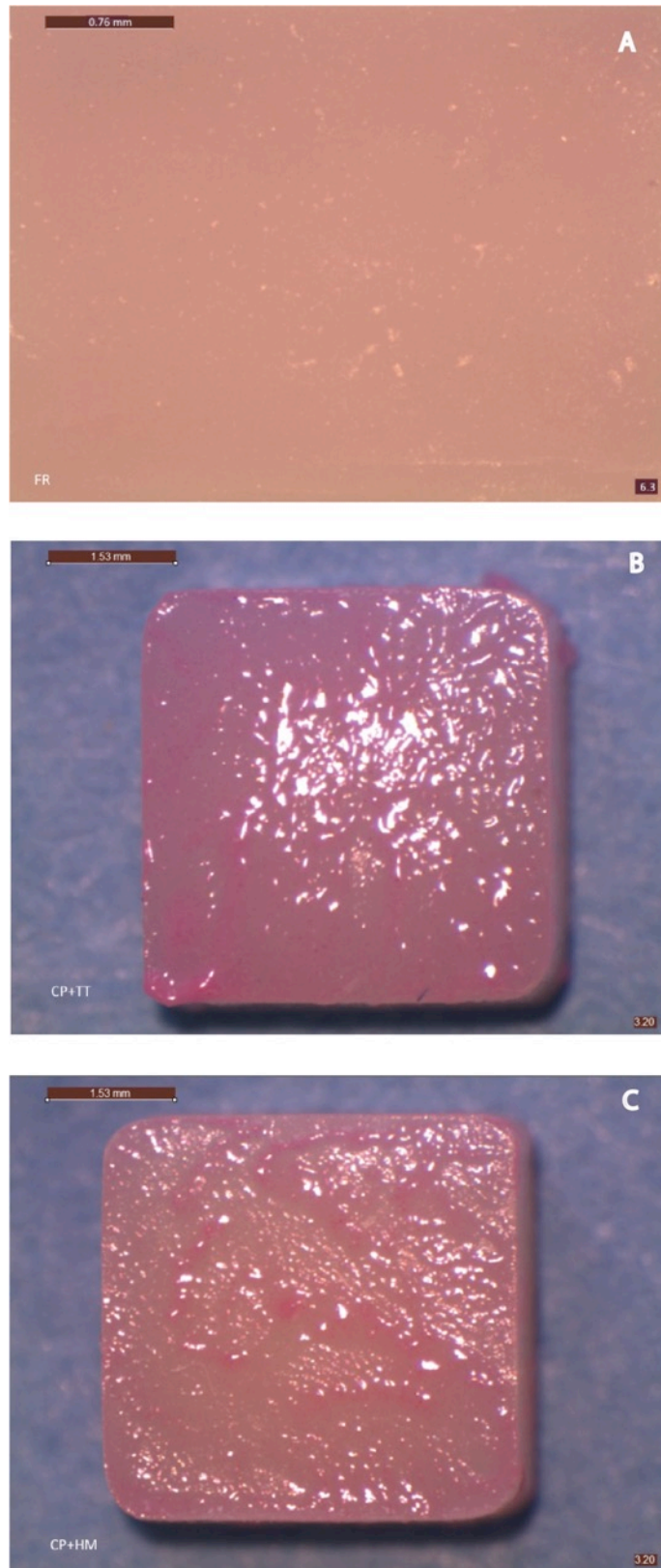


FIGURE 24. A) FR FCR before the interaction with the impression material at 6.3 magnification; B) TT IFPM after contacting CP PVS impression material at 3.2 magnification; C) HM FCR after contacting CP PVS impression material at 3.2 magnification.

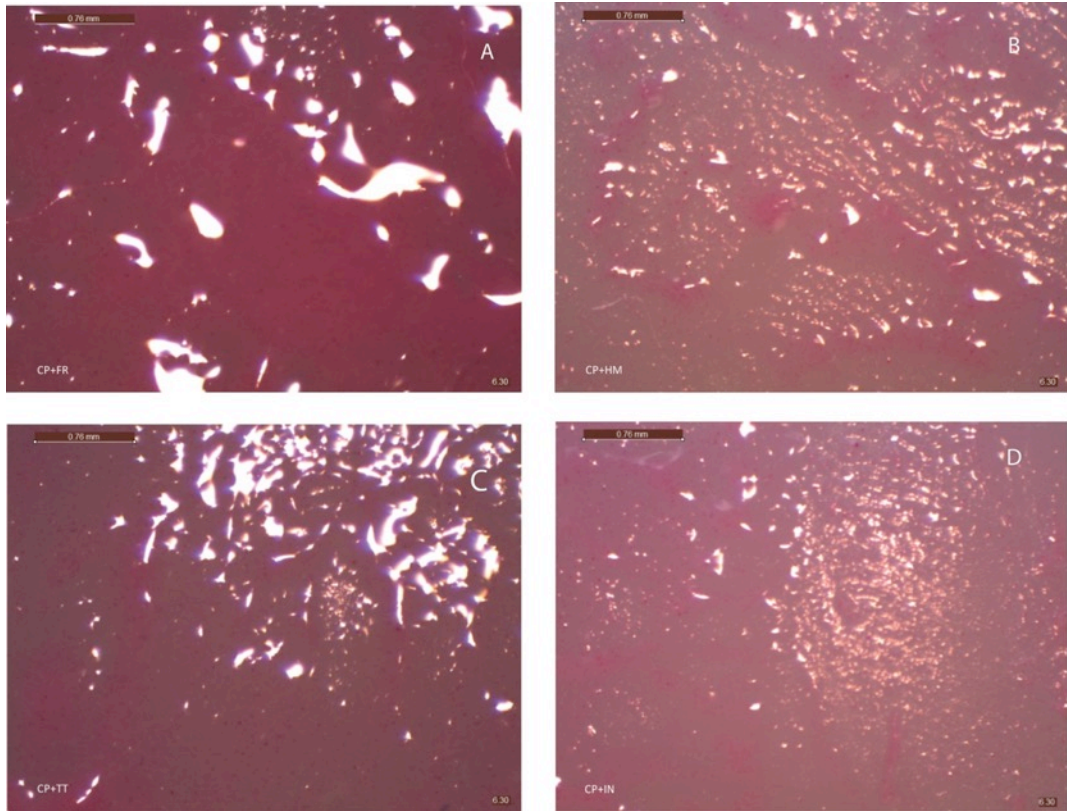


FIGURE 25. CP PVS impression material after contact with A) FR FRC; B) HM FRC; C) TT IFPM; D) IN IFPM at 6.3 magnifications.

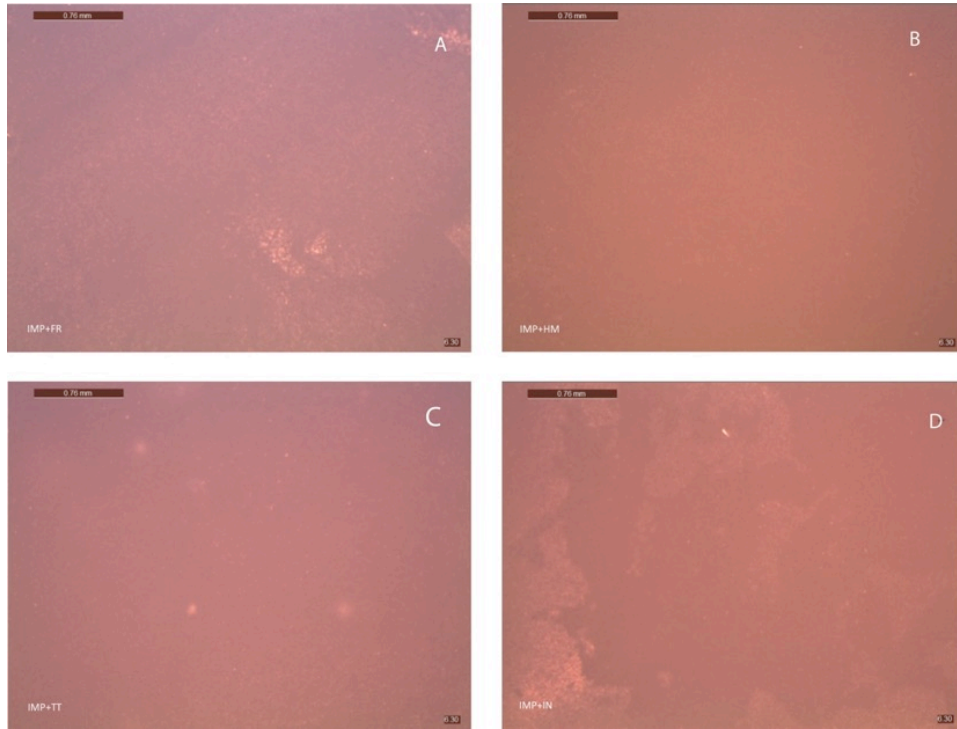


FIGURE 26. VIT PVS impression material after contact with A) FR FRC; B) HM FRC; C). TT IFPM; D) IN IFPM at 6.3 magnifications.

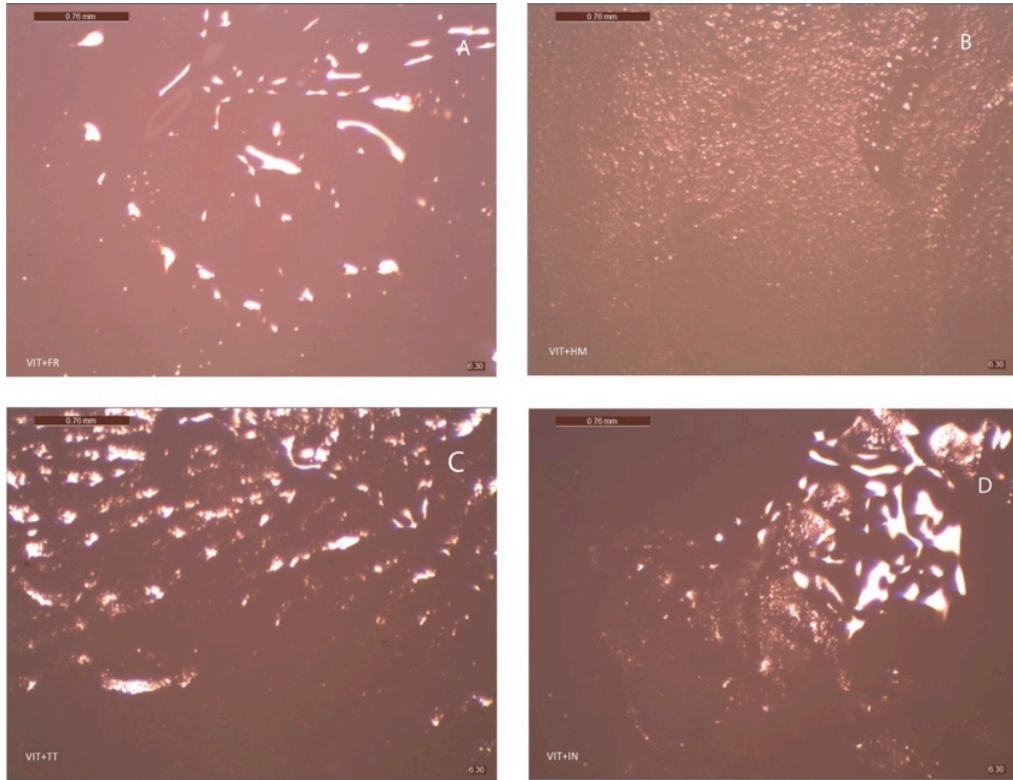


FIGURE 27. IMP polyether impression material after contact with A) FR FRC; B) HM FRC; C) TT IFPM; D) IN IFPM at 6.3 magnifications.

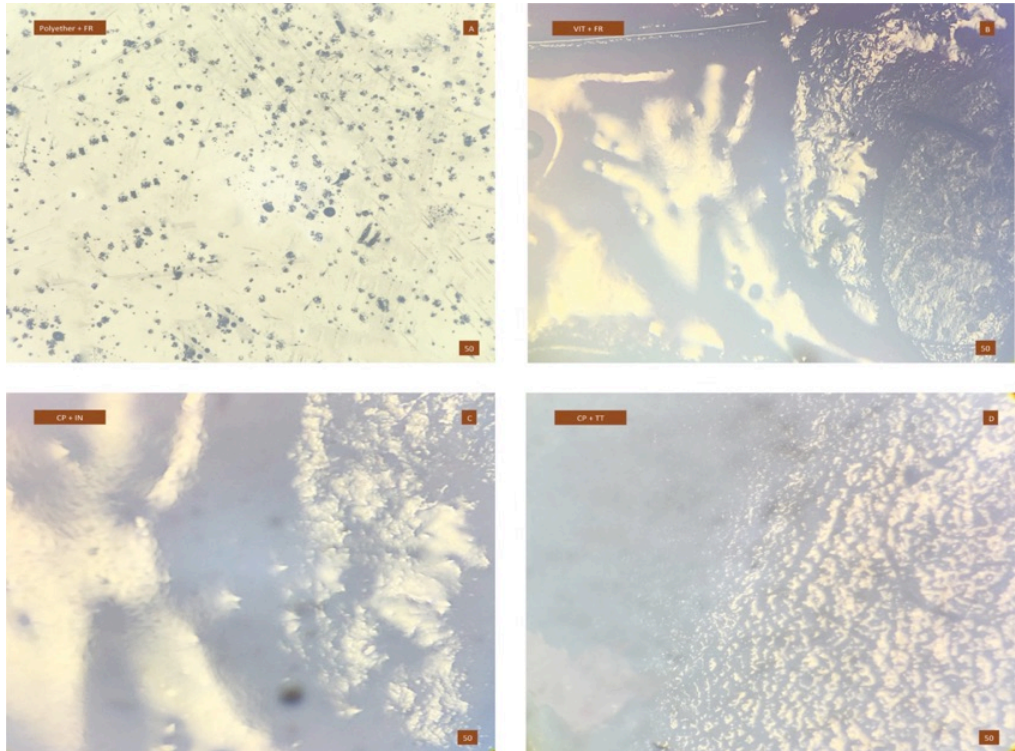


FIGURE 28. Metallographic images of (X50) of A) Completely polymerized IMP polyether filling the microporosites of FR FRC; B) Inhibited VIT PVS impression material on the surface of FR FRC; C) Inhibited CP PVS impression material on the surface of IN IFPM; D) Inhibited CP PVS impression material on the surface of TT IFPM.

DISCUSSION

This study assessed the inhibitory effect of certain types of interim fixed prosthodontic materials (IFPM) such as Tuff-Temp Plus (TT) and Integrity (IN) and flowable composite resin (FCR) materials such as Flows Rite (FR) and Heliomolar (HM) with polyvinyl siloxane impression materials Virtual (VIT) and Correct Plus (CP) as quantified by weight change and qualified by Fourier transform infrared (FTIR) spectroscopy.

According to the results of the present study, the null hypothesis was rejected. The results in Part I showed significant weight loss of the PVS impression materials when they have been in contact with FR and HM. FR showed the highest amount of PVS impression material inhibition. Two studies reported that the oxygen-inhibited layer was the primary reason for the inhibition of the PVS impression materials.^{64,65} However, later studies excluded the oxygen-inhibited layer as a factor in the inhibition of the PVS impression materials and the inhibition still existed after it was removed.^{19,20} This study was in agreement that the oxygen-inhibited layer was not the factor for PVS inhibition as the inhibition was noted significantly after removing the oxygen-inhibited layer.

A possible explanation for the high amount of inhibition with the FR is that they have a high percentage of methacrylate ester monomers (up to 65%). For HM FCR, there was significant inhibition; however, it was to a lesser degree than FR FCR because it has less urethane dimethacrylate monomer with only up to 5 percent. There was no obvious inhibition of the polyether impression material Impregum Garant (IMP) when in contact with either FR or HM. However, it was noted during the

removal of IMP from the experimental and control specimens that IMP adhered to the specimens and was difficult to remove from the specimens. Explanations for this may be related to polyether impression material having different properties than PVS impression material. They are hydrophilic and have less tear strength compared with PVS impression materials.^{66,67} In addition, the absence of the saliva in this study made it difficult to remove the polyether IMP impression material.

For Part II, results showed statistical difference in the P2/P1 ratio, and the FTIR-ATR was able to detect inhibited PVS impression materials on the surfaces of the experimental specimens. FTIR-ATR was able to locate the peaks of both silicon hydride (P1) groups Si-H and methylsilyl (P2) groups Si-CH₃. The hydride silicon group is very active and appeared to be small and unstable. However, the group Si-CH₃ was easy to locate on all the surfaces of the experimental specimens.

TT IFPM had similar monomer content to FR FCR, which would explain the inhibition of the PVS impression materials. In addition to that, IN is a chemically activated IFPM and caused significant amount of inhibition to the PVS impression material. IN composition contained sulfur compounds that potentially caused the inhibition of the PVS impression materials. Earlier studies of the interaction between latex gloves and the PVS impression material reported that the sulfur compound in latex gloves is the reason behind incomplete polymerization of PVS impression materials.^{9,56,59}

FTIR-ATR was able to locate polymerized IMP on the surface of the experimental and control groups and with no statistically significant difference. The peaks of IMP showed smaller ratios and reduced heights indicating the complete polymerization of the impression material.

Light microscopic evaluations were consistent with the results of weight loss

and the FTIR-ATR experiments. All FCR (FR, HM) and IFPM (TT, IN) caused significant amounts of inhibition of PVS impression material (VIT, CP). PVS impression material residue was seen on the top of the surfaces of the experimental specimens giving surface irregularities to the surface and reflected light and scattering of the light from the microscope. However, there was no evidence of inhibited polyether impression material (IMP) on the surfaces of the experimental groups. The surface of the experimental groups had a matte surface appearance after the removal of the polyether (IMP) impression materials.

Compared to light microscopic evaluations, specimens that were examined under high magnification of the metallography revealed similar inhibition pattern of the PVS impression materials and surface irregularities of the experimental specimens. Moreover, IMP was found in the microporosities of the tested specimen surfaces left as a result of low tear strength compared to PVS impression materials.

Previous studies have evaluated using visual examination of the interactions of PVS impression materials to core build-up materials¹⁹ or interim fixed prosthodontics materials.²⁰ Other studies have reported the inhibition of PVS impression material with immediate sealing bonding using digital photography⁶⁵ or light microscopy alone.⁶⁴ Therefore, it appears that there was void in the literature to validate the inhibition of the PVS impression material with FCR and IFPM. There appears to be no research addressing this phenomena which used weight loss testing to quantify the amount of inhibited PVS impression material or FTIR-ATR and other supplementary evaluations such light microscopy and light microscopy.

A recent study by Chen et al. reported the effect of PVS impression materials on the polymerization of composite resins by assessing the Vickers microhardness and degree of conversion of composite resins. The scanning electron microscopy

evaluation showed that a 10- μm deep inhibited layer was found in the experimental group. Moreover, the Vickers microhardness and degree of conversion of composite resins showed statistically lower values for the experimental group than those of the control group.⁶³ It appears from this study that both PVS impression materials and resins composites affect each other when those material directly polymerize against each other.

Limitations of the present study include the absence of the oral cavity temperature for PVS impression material polymerization. Alternatively, the polymerization of each brand was studied in constant temperature room at 21°C. The specific time of the polymerization for each brand was determined with FTIR-ATR. The removal time of the PVS impression material was determined based on stability of the reacted silicon hydride groups (Si-H). When there was no longer change in height of silicon hydride groups (Si-H), the PVS impression material was considered to be completely polymerized. The absence of saliva during the experiment is another limitation to this study. It was noted that the removal of polyether (IMP) impression material was difficult and the lack of moisture might have contributed to IMP adherence to both experimental and control groups. Also, IMP low tear strength compared to PVS impression materials likely contributed to the result.

SUMMARY AND CONCLUSION

Within the limitations of this study it can be concluded that interim fixed prosthodontics materials (IFPM) and flowable composite resins (FCR) inhibit the polymerization of polyvinyl siloxane (PVS) impression material. A weight changes test showed a significant amount of PVS impression material was unpolymerized after contacting the experimental specimens. The Fourier transform infrared spectrophotometer with a diamond attenuated total reflectance (FTIR-ATR) was able to detect inhibited PVS impression material on the experimental specimens surfaces after they were in contact with each other. Light microscopy and reflective microscopies was detected that inhibited PVS impression material was left on the surfaces of IFPM and FRC. Such interaction may occur during dental treatment and clinicians must select the appropriate restorative and impression material to avoid any possible interaction. Polyether impression material did not exhibit any interaction with the experimental specimens, and it can be used as an alternative impression material.

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ABSTRACT

EFFECT OF INTERIM FIXED PROSTHODONTICS MATERIALS AND
FLOWABLE COMPOSITE RESINS ON POLYMERIZATION
OF POLYVINYL SILOXANE IMPRESSIONS

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Objective: The aim of this study was to test the inhibitory effect of interim fixed prosthodontic materials (IFPM) and flowable composite resins (FCR) materials with polyvinyl siloxane (PVS) impressions as quantified by weight change and qualified by Fourier transform infrared spectroscopy.

Materials and Methods: Specimens made of FCR Heliomolar Flow and Flows Rite were used. Baseline weights (S) for each specimen were determined before applying the PVS materials and then two brands of PVS materials were applied. The material loss of the PVS material caused by the inhibitory effect was quantified with a calibrated 0.0001-mg digital analytical balance. The difference between the pre-application weight of the PVS and post-application weight was considered as the amount of total inhibited PVS. (FTIR-ATR) was used to study changes in PVS materials when they came into contact with IFPM and FCR materials. Three types of

PVS impressions were tested after applying them to five different substrates, two IFPM, two FCR, and bovine enamel. One-way ANOVA was used to compare the FCR -PVS combinations for differences in weight change. Two-way ANOVA was used for differences in P2/P1 ratios of the impression materials.

Results: The amount of weight loss and the P2/P1 ratio of PVS impression materials showed statistical differences from the control group ($p > 0.05$). Light microscopy and reflective microscopy evaluation showed inhibited PVS impression materials on the surfaces of the experimental groups. One -way ANOVA was used for differences in weight change. Two-way ANOVA was used for differences in P2/P1 ratios of the PVS. A 5-percent significance level was used for all tests

Conclusion: Interim fixed prosthodontic materials and flowable composite resins inhibit the polymerization of the PVS impression materials.

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