CHAPTER 3: MATERIALS AND METHODS

3.1. Materials Used In This Study

One resin modified glass ionomer cement, one dentine adhesive system and two composite resins were used in the study. These materials were used according to the manufacturer's recommendations.

The materials that were used in this study are listed in the table 3.1.

Materials	Presentation	Manufacturer	Lot # and (Exp. date)
Vitrebond [™] resin modified glass ionomer liner/base.	Powder in bottle. Liquid in vial.		20060429 2009-02
Filtek [™] P60 packable composite.	Single paste in syringes.	3M ESPE Dental Products St. Paul, MN 55144-1000, USA.	5RG (8100B2) (2008-10)
Filtek [™] Z350 flowable nanocomposite.	Low viscosity single paste in syringes.		6AH (6031B2) (2008-01)
Adper [™] Scotchbond [™] multi-purpose Plus: Scotchbond [™] Etchant Rely X [™] ceramic primer Adper [™] Adhesive	Single liquid Single liquid Single liquid		7545S 6KG 2009-01 5BB 2008-12 6PK 2008-12

Table 3.1. List of materials,	presentation.	manufacturer	, lot number	r and ex	piry date
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These materials are illustrated in Figures 3.29, 3.39, 3.40 and 3.44.

The materials main contents and manufacturer's recommendations were followed in this

study are listed in table 3.2.

Materials	Main contents	Manufacturer's recommendations	
Vitrebond [™] resin modified glass ionomer liner/base. Powder in bottle and liquid in vial.	Powder: ion-leachable fluoroalumino- silicate glass powder with SiO2, AlF3, ZnO, SrO, cryolite, NH4F, MgO and P2O5. Liquid: modified polyacrylic acid methacrylate groups, HEMA (2 hydroxyethylmethacrylate), water and photoinitiator.	First layer 0.5 mm thickness or less, need 30 sec curing with 400mW/cm ² . second layer up to 1mm, need 30 sec curing with 400mW/cm ² .	
Filtek [™] P60 packable composite, Syringe, Shade B2.	61% vol. 83% wt. inorganic filler (zirconia/ silica) (without silane treatment) (0.01-3.5 μm) size. Bis- GMA, UDMA and Bis-EMA resins.	1-2.5 mm thickness in increment, need 20 sec curing with 400mW/cm ² .	
Filtek [™] Z350 flowable nanocomposite, Syringe, Shade B2	55% vol., 65% wt. inorganic filler (zirconia nanofiller/ silica nanofiller) (0.06-1.4 microns) size. Bis-GMA, TEGDMA and Bis-EMA resins.	0.5-2.00 mm thickness need 20 sec curing with 400mW/cm ²	
Adper [™] Scotchbond [™] multi-purpose Plus: Scotchbond [™] Etchant Rely X [™] ceramic primer Adper [™] Adhesive	Aqueous solution of 35% Phosphoric acid (H ₃ PO ₄). HEMA, light-cured polymer. Bis-GMA (60-70%), HEMA (30- 40%), dimethacrylates, polyalkenoic acid copolymer and photoinitiator.	15 sec etching, 15 sec rinsing, 2 sec drying, 5 sec ceramic priming with 5 sec drying, 10 sec curing of adhesive layer with 400mW/cm ² .	

Table 3.2. Main contents of materials and manufacturer's recommendations.

3.2. Methodology

3.2.1. Steps in Specimens Fabrication:

Eighty-eight specimens (simulated PFM crowns with access openings) were fabricated in the following manner:

3.2.1.1. Wax Preparation:

A standardized custom washer (Figure 3.1) measuring 19 mm in diameter with a standardized centre hole (5 mm in diameter to simulate the access openings) was manufactured in the ceramic laboratory (Faculty of Dentistry, University of Malaya, Malaysia) from metal (Collon Buim Type I[®], Japan) to produce the standardized wax disc patterns (Figure 3.2) from calibrated green sheet wax 0.4 mm in thickness (Dentaurum[®], Germany). A separating medium fluid (Dentaurum[®], Germany) was used to isolate and prevent the sheet wax from adhering to both the glass slab and the standardized custom washer during the fabrication of the wax disc patterns by using a scalpel and sharp surgical blade #11. However, a small projection of wax (1 mm in length and 1 mm in width) was created attached to the outer border of the standardized wax disc pattern. It was made to facilitate grasping when reproduced in metal during buildup of porcelain layers. The dimension of the access cavities in the PFM specimens was pre-established to approximate clinical conditions (Figure 3.3).

3.2.1.2. Spruing:

Evey 10 wax disc patterns were attached to their minor sprues by using a wax wire 3 mm in thickness (Dentaurum[®], Germany) (Figure 3.4).

3.2.1.3. Investing:

Surface tension reducing agent debubblizer (Kerr[®], USA) was applied to the wax patterns. Ten samples were invested together using casting ring size C which was lined

with a single layer of asbestos free ring liner (Kera-Vlies, Dentaurum[®], Germany) after wetting it with water and adapted to the inner surface of the ring. Phosphate bonded investment powder and liquid (Jelenko[®], NY, USA) were used to invest the samples (Figure 3.5). The investment material was mixed manually and also mechanically by using a mixing vaccum machine (Dentaurum[®], Germany). Investing was carried out according to the manufacturer's recommendations (Figure 3.6).

3.2.1.4. Burnout:

The wax disc patterns were burned out by using an electrical furnace (Jelenko[®], accthermII, XL-M, N.Y., USA) according to the manufacturer's recommendations regarding temperature and timing.

3.2.1.5. Casting:

The wax disc patterns were cast in an induction casting machine (Bego[®], Germany) with a nickel-chromium alloy (Collon Bium Type I[®], Ni-Cr alloy, Japan) (Figure 3.7) melted in a ceramic crucible (Bego[®], Germany). Casting technique was also carried out according to the manufacturer's recommendations.

3.2.1.6. Preparation and finishing of the metal specimens to receive porcelain:

All residual investment was removed from each metal casting by sandblasting with nonrecycled pure aluminum oxide particles size 50 μ m (Dentaurum[®], Germany) (Figure 3.8).

3.2.1.6.1. Removing the sprue (de-spruing):

After the casting had been inspected and found to be satisfactory, the sprue from each completed casting was removed by using a thin carborundum disc (Dentaurum[®],

Germany) mounted on a low-speed handpiece controlled at 30,000 rpm (Kavo[®], NM-4000, Germany). Each disc was used for five specimens only.

3.2.1.6.2. Fitting the samples:

Each specimen was checked for any bending or deformity. If there was any defect, the sample would be discarded. All specimens were laid fully and completely on a flat surface without bending (Figure 3.9).

3.2.1.6.3. Recontouring the sprue area:

As the specimens were confirmed flat without bending, the areas where remnants of sprues remained were reduced carefully by using a coarse ceramic abrasive (Dentaurum[®], Germany). A properly finished porcelain-bearing surface was achieved with a fine ceramic stone followed by white aluminum oxide stone bur (Dentaurum[®], Germany) moved in one direction to reduce the surface irregularities and to remove debris. Each bur was used for five specimens only.

3.2.1.7. Metal Oxidation:

After finishing the metal specimens, they were cleaned thoroughly. Firstly, the porcelain-bearing surfaces of all the specimens were sandblasted with nonrecycled pure aluminum oxide particle size 50 μ m (Dentaurum[®], Germany) at a maximum pressure of 3 bars for 10 seconds to remove the excess oxide layer. They were further steam cleaned (Triton, Bego[®], Germany) for 30 seconds to remove any debris (Figure 3.10) and then heat-treated in a porcelain furnace (Vita Vacumat[®] 40 T, Vivadent[®], Germany) to degas for 5 minutes at 1000 °C under vacuum (Figure 3.11).

3.2.1.8. Porcelain Buildup:

Vita VKM[®] 68 feldspathic porcelain opaque, body and enamel (shade B2) (VITA Zahnfabrik, Bad Säckingen, Germany) was used in this study because it was indicated for fabricating PFM restorations (Figure 3.12).

3.2.1.8.1. Opaque Application:

Vita VKM[®] 68 opaque powder was mixed with Vita opaque liquid in a clean glass slab by using a glass rod. This mixing was done on a vibrator to disperse air bubbles according to the manufacturer's instructions. First, a thin wash layer was applied to the porcelain-bearing surface by using a brush (Figure 3.13) and fired according to the manufacturer's instructions. Second, a thin creamy consistency was applied to the sample around the perforation with a brush, and then the hemostat was tapped lightly with a serrated lecron in a vibrating motion to condense the opaque porcelain. Excess opaquing liquid rising to the surface was removed with thin absorbant tissue paper placed against an edge of the moist opaque porcelain. Excess liquid was blotted away from as many directions as possible until the opaque material took on a dull appearance. Third, the end of the hemostat holding the specimen was exposed to the heat radiating from the porcelain furnace muffle to dry the opaque surface. Then any dried opaque from non porcelain-bearing areas, such as the lower surface and borders of the metal specimen, was removed using a small stiff-bristle brush. Fourth, the opaqued porcelain samples were put on a saggar tray and transferred to the middle of the muffle stand for firing according to the manufacturer's instructions. A second opaque application would completely mask the metal oxide colour.

Finally, the opaque layer around the access openings was determined to be 0.3 mm \pm 0.1mm in thickness by a metal caliper device (Figure 3.14) from four random positions and the average was taken.

3.2.1.8.2. Body and Enamel Porcelain Applications:

The technique for mixing Vita VKM[®] 68 body powder with Vita modelling liquid and its application was virtually the same as that used to mix opaque porcelain. It was then fired according to the manufacturer's instructions (Figure 3.15). If a second layer of body porcelain was required, it was applied in the same manner as the first. The mixing of enamel porcelain application and firing was done according to the manufacturer's instructions. The total thickness of both body and enamel porcelain layers was 1.5 ± 0.2 mm. Adjusting and finishing were carried out by using a sintered diamond bur (Dentaurum[®], Germany) mounted on a controlled low-speed micromotor handpiece until the thickness of the specimen was 2 ± 0.5 mm as measured by both metal caliper and digimatic micrometer devices. Finally, a thin layer of glaze was applied and fired according to the manufacturer's instructions. The porcelain furnace (Vita Vacumat[®] 40 Vivdent[®], Germany) was programmed according T. the manufacturer's recommendations for all firing cycles of each applied layer of porcelain of five specimens together each time.

The fabrication of the 88 metal discs and dental porcelain build-up were carried out by one operator according to procedures in the actual construction of porcelain-fused to metal-crowns and were standardized as much as possible (ISO/TS 6872, 1999; Rosenstiel et al., 2001) following the manufacturer's recommendations.

The final thickness and shape of the PFM samples to be tested were measured to be 2 ± 0.5 mm after wet grinding using a diamond disc (Beuhler[®] IL, USA) and grinder (Metaserver[®] 2000, Beuhler[®] IL, USA) (Figure 3.16) to ensure the elimination of irregular surfaces particularly around the access openings. They were placed in a container of normal physiological saline (0.9% w/v Sodium Chloride solution, B/Braun[®], Germany) inside an incubator (Memmert B-400, GmbH Corporation, Germany) at 37°C for one week until ready to be restored (Figure 3.17).

To simulate clinical conditions, the diameter of the simulated access opening was confirmed to be 5 mm (Figure 3.18) and the total thickness of the PFM sample was 2 ± 0.5 mm (Metal + 2 layers of opaque + 1 layer of body + 1 layer of enamel + a thin layer of glazed porcelain) (Figures 3.19 and 3.20).

3.2.1.9. Treatment of the inner surfaces of the PFM access openings:

The inner surfaces of all the PFM access openings were treated with super coarse taper diamond burs (ISO No.: 806 314 546 016, FG 546G016, Diamant, Horico[®], Germany) by using a high speed turbine topair at 300,000 rpm (No. TS1-14771, Semince[®], Germany) under heavy water spray and mounted on a dental surveyor (AF30, Switzerland) (Figures 3.21 and 3.22). A total of five access cavities were treated successively by one bur and the time was recorded to the nearest 10 s (Stokes and Tidmarsh, 1988). A total of 88 specimens were examined with a laboratory light-microscope (x 7 magnification) to ensure that they were free from porcelain cracking or chipping. Eight specimens were discarded to provide 80 specimens for the investigation.

3.2.2. The Surface Roughness Test:

A surface profilometer (Surftester[®], Mitutoyo[®], Japan) (Figure 3.23), with sleeve removed from stylus (Figure 3.24), was used to measure and determine the mean surface roughness (Ra, μ m) of the inner surface of the PFM after treatment with the super coarse taper diamond. Randomly, twenty PFM specimens were selected and measured at 5 areas each in various locations at the margins of the inner surface of the PFM perforations (curve surface) with a distance from each other of approximately 1.0 mm. The mean of the five measurements determined the surface roughness (Ra- μ m). All profilometer recordings were made parallel to a line connecting the index notches at coordinated positions giving a mean Ra = 1.11 μ m. (Appendix C).

3.2.3. Construction of Standardized Epoxy Mould with a centre cavity:

A silicone impression material (Optosil[®] P Plus and Activator universal Optosil-Xantopren[®], Heraeus Kulzer, Germany) was used together with a clear cold curing epoxy resin (Mirapox[®] A and B, Miracon[®], Malyasia) was used to construct the 80 standardized epoxy moulds.

First, a calibrated pink sheet wax (Dentaurum[®], Germany) was used to construct a standard box (30 mm in length x 26 mm in width x 4 mm in thickness) (Figure 3.25) which was placed over a clean glass slab and fixed to it by molten wax. A standardized wax disc measuring 7.5 mm in diameter and 1.5 mm in thickness was punched with a standardized plastic band 7.5 mm in diameter and 30 mm in length. It was then placed in the centre of the wax box and fixed to the base of the box by using molten wax. Second, mixed green stone was poured into the wax box and left for 30 minutes to completely set.

Third, the standard stone mould was removed from the wax box (Figure 3.26), and then silicone impression material was adapted with gentle pressure into the stone mould. From this, a standardized silicone mould was obtained to produce a standardized epoxy mould with its own cavity (Figure 3.27). Each standardized stone mould was used to produce 20 standardized silicone moulds and each standardized silicone mould was used to produce four standardized epoxy moulds. The epoxy resin was left for 24 hours to completely polymerize and set (Figure 3.28).

Fourth, the standardized epoxy mould was removed from the silicone and then all 80 standardized epoxy moulds with their cavities were placed under hot running water for 30 minutes to get rid of any debris.

After that, all the surfaces of the epoxy moulds were ground on 180-grit silicon carbide paper (Buehler, USA) to remove excessive epoxy resin under water coolant. A grinder and polisher (Metaserver[®] 2000, Buehler[®] IL, USA) was used to remove any irregular borders and to smoothen the surfaces. Each standardized epoxy mould has a standardized cavity in the centre measuring 7.5 mm in diameter and a depth of 1.5 mm \pm 0.2, which was confirmed by a digmatic indicator (Mitutoyo[®], Japan) from 4 different points. The overall dimensions of each standardized epoxy mould was measured by a digmatic micrometer and digmatic vernier caliper (Mitutoyo[®], Japan) to obtain a length of 30 mm and 26 mm in width.

Finally, the standardized simulated cavity in each epoxy mould was filled with Vitrebond[™] resin modified glass ionomer liner/base (3M ESPE, USA) (Figure 3.29) according to the manufacturer's recommendations. The filled simulated cavities were ground on 180-grit silicon carbide paper (Buehler, USA) to remove excess Vitrebond[™] cement from the filled cavity surfaces under water coolant by Grinder and Polisher (Metaserver[®] 2000, Beuhler[®] IL, USA) (Figure 3.30) to ensure all surfaces were smooth and flat. After this, each PFM was fixed to the filled cavities in the epoxy moulds by using epoxy resin. This was done by carefully adding a thin layer of a cyanoacrylate glue (Supa Glue, Selleys[®], Australia) (Figure 3.31) on the clean lower metal surface of the PFM specimen. Care was taken to ensure that no cavity surfaces were contaminated by the glue and epoxy resin (Figure 3.32). The PFM specimen was centred on the simulated filled cavity in the epoxy mould and gentle pressure applied on it for 10 seconds. It was allowed to set up to 12 hours and then a thin layer of the mixed epoxy resin was added all around the outer borders of the PFM sample and the epoxy mould (Figure 3.33) and left for 24 hours to completely polymerize and set to ensure a good seal and to prevent any penetration around the borders during immersion in blue ink.

The depth of the access cavities in the PFM was determined by using a digmatic indicator (Figure 3.34) (Mitutoyo[®], Minato-Ku, Tokyo 108, Japan) at 4 random positions to confirm that it was 2 ± 0.5 mm. The spindle with its attached gauge was lifted and the fixed PFM-epoxy mould was placed in contact with the platform beneath the lifted gauge and then the tip of the gauge was lowered to a point at the top of the access opening margin and the measurement was taken (Figure 3.35). Then the tip of the gauge was lowered at the same point into the access cavity onto the surface of the Vitrebond filled epoxy mould (Figure 3.36). The depth of the PFM access opening was determined by the difference between the two measurements at the same point. This method was repeated four times at four different points and the mean was taken. The depth of PFM access openings for all 80 specimens was determined to be 2 ± 0.5 mm.

3.2.4. Sample Grouping:

The 80 standardized PFM-epoxy moulds were randomly divided into 2 equal groups according to the types of the filling materials, and then the 40 specimens in each group were further subdivided into 4 equal sub-groups according to water storage periods and thermocycling (Figures 3.37 and 3.38). The PFM-epoxy moulds were coded numerically using a permanent pen according to the filling material group, water storage periods and thermocycling.



Fig. 3.38. Diagram showing samples grouping and subgrouping.

1: Sub-group1: A1 and B1: 1 day distilled water storage without thermocycling.

2: Sub-group2: A2 and B2: thermocycling (500 cycles) after one day distilled water

storage.

3: Sub-group3: A3 and B3: 7 days distilled water storage without thermocycling.

4: Sub-group4: A4 and B4: 7 days distilled water storage and thermocycling

(504 cycles within 7 days).

3.2.5. Sample Filling:

3.2.5.1. Group A (Control Group):

The access cavities of group A were restored with packable composite (FiltekTM P60, Shade B2, 3M ESPE, USA) with AdperTM ScotchbondTM multi-purpose plus adhesive system (3M ESPE, USA) (Figure 3.39). The restorative procedures were done according to the manufacturers' recommendations as follows:

First, the walls of the access cavities were treated with ScotchbondTM Etchant (35% phosphoric acid, 3M ESPE, USA) applied with a disposable brush (Microbrush, 3M ESPE, USA) for 15 seconds. Subsequently, the etchant was rinsed off with a copious amount of water for 5 seconds and then dried with oil-free air for 2 seconds.

Second, Rely X[™] ceramic primer (silane primer) (3M ESPE, USA) was applied to the walls of the access cavities using a fully saturated disposable brush. Then, the walls were gently air dried for 5 seconds to leave a shiny surface and AdperTM ScotchbondTM multi-purpose plus adhesive was applied to the treated walls of the access cavities using a fully saturated 3M disposable brush by gentle brushing of the adhesive on the walls for 10 seconds and then thinning by using oil-free air spray until movement of the liquid was no longer visible. The adhesive was light-cured for 10 seconds by using a lightcuring unit with a light intensity of 400 mW/cm² (Spectrum 800, Dentsply[®] Caulk, USA). Third, Filtek[™] P60 was applied incrementally into the access cavities of PFM (Figure 3.40). Fourth, the restorative material was adapted using plastic instruments (Figure 3.41) to adapt the Filtek[™] P60 (Ash No. 6 and 49) (Carig and Powers, 2002). A Mylar strip (3M ESPE, USA) was placed on the restored cavity with a gentle passive digital pressure and finally adapted by a 5-kg static load for 5 minutes using a custom made loading device (Figure 3.42). Following adaptation, any excess at the margins was carefully removed by using a surgical blade #11 in the direction from restoration to access cavity margin (Neiva et al., 1998), and then covered with a Mylar strip and light cured for 20 seconds by holding the light emission window as close as possible to the Mylar strip (Figure 3.43).

3.2.5.2. Group B (Experimental Group):

The access cavities of group B had in addition the flowable nanocomposite (FiltekTM Z350, Shade B2, 3M ESPE, USA) as a liner under the FiltekTM P60 (Figure 3.44). The

restorative procedures were carried out according to the manufacturers' recommendations as follows:

First, the applications of ScotchbondTM Etchant (35% phosphoric acid), Rely XTM ceramic primer and then the AdperTM ScotchbondTM multi-purpose plus adhesive to the walls of the access cavities were the same as in group A. Second, FiltekTM Z350 was applied as a liner by using a needle syringe to approximate 0.5 mm thickness at the metal-opaque porcelain junction. This thickness was judged by two methods; the metal-porcelain junction became invisible and by a groove placed on the delivery needle (Figure 3.45). The FiltekTM Z350 lining material was light cured for 20 seconds. The application of FiltekTM P60, adaptation and light curing were done as for group A (Figure 3.46).

After that, the restored access cavities were immediately stored in distilled water (Figure 3.47) for periods of 24 hours and 7 days. Finishing and polishing were carried out after 24 hours of distilled water storage. Finishing was carried out on #600 silicon carbide paper. Then, they were polished with Sof-LexTM polishing discs (3M ESPE, Dental Product, USA); one disc for 1 specimen by using a low-speed handpiece of 15,000 rpm (Simence[®], Germany) mounted on a dental surveyor under water spray (Figures 3.48-3.51). Polishing was conducted with a series of grit from coarse to superfine-grit in sequence of coarse (150), medium (360), fine (600) and superfine (1200). Uniform light pressure in a circular pattern for 10 seconds for each abrasive step was used to polish the specimens (Lui and Low, 1982; Lopes et al., 2002). All 80 specimens were polished using this method by one operator.

3.2.6. Water Storage, Thermocycling and Dye Application:

For the sub-group 1, after one day distilled water storage [ISO/TS (11405/2003)], the specimens were removed from the distilled water and allowed to dry for 2 hours at room temperature. After that, two coats of fingernail varnish were painted 1 mm from the

interfaces between the PFM and the restored access openings to prevent dye penetration from anywhere else other than at the PFM and filling material interfaces (Figure 3.53). After allowing 15 minutes for each coating of the fingernail varnish to set and air dry, the 10 specimens of each sub-group 1, were completely immersed in a container of blue ink (Parker[®] Quink[®], UK) (Table 3 Appendix A) (Figure 4.54) at 37° C for 24 hours. The pH of the ink was 7.4 as tested by a pH meter (HANNA Instrument, Singapore).

For the sub-group 2, following one day distilled water storage the specimens were placed in separate mesh bags and thermocycled between 5 °C \pm 2 °C and 55 °C \pm 2 [ISO/TS (11405/2003)] for 30 seconds in the cold and 30 seconds in the hot baths (Gale and Darvell, 1999) with a transfer time between baths of 10 seconds for 500 cycles [ISO/TS (11405/2003); Gale and Darvell, 1999] (Figure 3.52). After completing the thermocycling, the 10 specimens of each subgroup 2 were removed from the water bath and allowed to dry for 2 hours at room temperature. After that the same procedures for varnishing and immersing in a container of Parker[®] blue ink were carried out as for the specimens of sub-group 1.

For sub-group 3, after seven days of distilled water storage (Hirasawa et al., 1983; Øysæd and Ruyter, 1986; Sorensen et al., 1991; Momoi and McCabe, 1994), the samples were removed from the distilled water and allowed to dry for 2 hours at room temperature. After that the same procedures for varnishing and immersing in a container of Parker[®] blue ink were carried out as for the specimens of sub-group 1.

For sub-group 4, within the seven days of distilled water storage, the specimens were thermocycled between 5 °C \pm 2 °C and 55 °C \pm 2 [ISO/TS (11405/2003)] for 30 seconds in the cold and 30 seconds in the hot baths with a transfer time between baths of 10 seconds over seven days; 72 cycles per day giving a total of 504 cycles. After completing the thermocycling, the 10 specimens of each subgroup 4 were removed from

the water bath and allowed to dry for 2 hours at room temperature (Crim et al., 1985; Noguera and McDonald, 1990). After that, the same procedures for varnishing and immersing in a container of Parker[®] blue ink were carried out as for the specimens of sub-group 1.

Once the dye immersion was completed, all the specimens were rinsed in tap water for 1 minute to remove excess dye and then cleaned with a toothbrush and blot-dried to remove any excess water that could alter the concentration of the dye solution and also to remove any surface dye. The nail varnish was removed with a scalpel with #11 blades. Then, the specimens were allowed to dry for 2 hours at room temperature in order to allow dye fixation prior to embedding completely in clear epoxy resin (Mirapox[®] A and B, Miracon[®], Malaysia) (Figure 3.55).

3.2.7. Sectioning:

Each specimen was sectioned into four equal quadrants by three sections (Sorensen et al., 1991; Tjan et al., 1992; Baldissara et al., 1998) by using a 0.5 mm thickness diamond blade isomet wafering (High-speed-2000, Isomet-Beuhler[®], USA) (Figures 3.56 and 3.57) in water coolant (lubricant).

The first section (longitudinal sectioning) was across the midline of the restored access cavities of the specimens into two equal halves. Each half section was photographed by a digital camera (Figures 3.58 and 3.59). The second section (perpendicular to the first) on the first half of the specimen was carried out across the center which produced two equal quadrants. The third section on the second half of the specimen was also cut across the midline to produce another two equal quadrants. Each specimen was therefore sectioned into four equal quadrants and each quadrant had two sides (Figures 3.60 and 3.61) totaling eight sides per specimen for evaluation of the dye penetration. These second and third sections (cuts) allowed the evaluator to see dye extension from

different directions. All 8 sides of each specimen were air dried and coded (blind technique) numerically using a permanent pen and stored in coded containers.

3.2.8. Measurement of Dye Penetration:

The coded sectioned specimens were first viewed under a stereomicroscope (Nikon type, Tokyo, Japan) at 15x magnification to ensure no evidence of leakage between the PFM and epoxy resin interface. Then, digital images of the dye penetration along the PFM-restoration interface for all the sections were evaluated with an image analyzing system (Fayyad and Shortall, 1987, 1989; Youngson et al., 1990). Digital images were obtained with a JVC camera (Tk. C1380-Colour Video Camera, Industrical optics, Video Lens-VSH300-39708, Japan) attached with a computer-image analyser software (Leica Qwin Lite T2-8, Serial No.: 3154, Leica Microsystems imaging solutions Ltd., U.K.) as shown in Figure 3.62. The sectioned specimens were all subsequently illuminated using a Schott 1500KL fibre optic unit and the dye penetration along the PFM-restoration interface was assessed at x 75 on-screen magnification using Leica Qwin Lite image analyzer; this allowed the measurement of the dye penetration along the PFM-restoration interface (1 pixel = 0.0109 mm). The depth of dye penetration between the PFM-restoration interfaces was assessed by linear measurement system by one examiner. Dye penetration was measured to the nearest 0.01 mm. The measurement was performed by a line drawn along the extension of the ink penetration at the PFMrestoration interfaces. The measurement of lengths was performed by using a light-pen on the screen of the computer-image analyzer and its software according to the linear leakage system. All the measurements were repeated by using the same protocol on three separate occasions with a three day interval between each evaluation without reference to the previous measurements by one evaluator (Darbyshire et al., 1988; Howdle et al., 2002). The measurements were tabulated and a final data set compiled.

3.2.9. Statistical analysis:

Data was subsequently transferred to a computer following breaking of the randomization code and statistical analysis was performed using SPSS for Windows statistical software (SPSS Inc., version 14.0, Chicago, IL, USA). The data were analyzed and statistical evaluation of the results was carried out in several stages:

The three linear measurements were taken for each eight sides of each specimen at three different occasions to determine the intra-rater reliability. An intra-rater reliability test was used to assess agreement in dye leakage level between the three linear measurements of the PFM-composite interfaces.

When intra-class correlation coefficient (intra-rater reliability) was calculated, there was an overall statistically significant agreement between measurements of dye leakage (mm). The intra-class correlation values are very high and the p-values are less than '0.001'. Thus, the measurements are highly reliable as shown in Table 3.3. The intraclass correlation between measurements (intra-rater reliability), a measure of rater reliability, was calculated to be '0.983'.

		95% Confidence					_
		Interval		F Test with True Value			
	Intra-class	Lower	Upper				p-
	Correlation	Bound	Bound	Value	df1	df2	value
Single	052	.945	.958	59.965	639.0	1278	.000
Measures	.932						
Average	083	.945	.985	59.965	639.0	1278	.000
Measures	.903						

 Table 3.3. Intra-class Correlation Coefficient.

The Three-way Analysis of Variance (ANOVA) is appropriate to determine the main effects and interaction of the restorative technique and water storage and thermocycling on the restored access cavities of PFM-composite resin interfaces. The two assumptions of concern are: (1) Normality of the residuals using histogram; and (2) Homogeneity of variance (Levene's test) between independent variables (p < 0.05).

To compare the means of two groups of an independent variable, the independent t test was used to compare means for two independent groups. Ideally, for this test, the subjects should be randomly assigned to two independent groups, so that any difference in response is due to the treatment (restorative technique, thermocycling or water storage) or no difference in response due to lack of treatment. Also, the nonparametric variant of pairwise comparison Mann-Whitney U test was used to identify similarities and differences between each two independent variables ($\alpha < 0.05$). For all the statistical tests, the level of significance was set at p < 0.05.



Fig. 3.1. Standard washer custom made. Fig. 3.2. Fabrication of wax discs patterns.



Fig. 3.3. PFM sample was fixed on the Filled Cavity-Epoxy-Mould.



Fig. 3.4. Spruing of wax discs.



Fig. 3.5. The investment materials.



Fig. 3.6. The sprued discs invested.



Fig. 3.7. Collon Bium Type I[®].



Fig. 3.8. The Casting was sandblasted.



Fig. 3.9. The flat metal discs.



Fig. 3.10. The metal discs after sandblasting



Fig. 3.11. The oxidation the metal.



Fig. 3.12. Vita VKM[®] 68 porcelain.



Fig. 3.13. Opaque porcelain application. Fig. 3.14. The thickness of opaque layer.



Fig. 3.15. The firing of body porcelain layer. Fig. 3.16. Grinder machine was used.



Fig. 3.17. PFMs were stored in saline.



Fig. 3.19. Thickness of PFM sample.



Fig. 3.18. Simulated PFM samples.



Fig. 3.20. Diameter of access opening.



Fig. 3.21. The dental surveyor was used to mount the turbine.



Fig. 3.22. The burs and turbine were used to treat the access openings.



Fig. 3.23. The Surftester[®] (Mitutoyo[®], Japan). Fig. 3.24. Roughness of inner surface was measured by a stylus.



0

Fig. 3.25. The standardized wax box made. Fig. 3.26. The standardized stone mould.







Fig. 3.28. The standardized cavity-epoxy-mould.





Fig. 3.29. VitrebondTM (3M ESPE, USA). **Fig. 3.30.** Filled cavities of epoxy moulds after grinding.



Fig. 3.31. Supa Glue (Selleys[®], Australia).



Fig. 3.32. PFM and filled cavity epoxy were glued.



Fig. 3.33. PFM and filled cavity fixed by epoxy resin.



Fig. 3.34. The digmatic indicator.



Fig. 3.35. Depth of access opening from surface.



Fig. 3.36. The depth of access opening from base.



Fig. 3.37. Ten sample for each sub-group.



Fig. 3.39. AdperTM ScotchbondTM Multi-Purpose Plus (3M, USA).



Fig. 3.40. Packable composite (Filtek[™] P60, 3M ESPE, USA).



Fig. 3.41. The application of P60.



Fig. 3.42. Applied 5 kg static load.



Fig. 3.43. Light-cure tip close to Mylar strip.



Fig. 3.44. Filtek[™] Z350 flowable nanocomposite (3M ESPE, USA).





Fig. 3.45. Filtek[™] Z350 was applied into access opening.

Fig. 3.46. Access opening of PFM was restored.



Fig. 3.47. Samples were stored in the distilled water.



Fig. 3.48. Restored PFM after grinding. Fig. 3.49. Sof-Lex[™] polishing discs (3M, USA).





Fig. 3.50. Sof-Lex[™] disc mounted on low-speed handpice.

Fig. 3.51. Restored access opening after finishing and polishing.



Fig. 3.52. The thermocycling machine used in this study.



Fig. 3.53. Varnished specimens were immersed in the blue ink.



Fig. 3.54. Parker[®] Quink[®] Blue Ink.



Fig. 3.55. Sample was embedded in epoxy resin.



Fig. 3.56. Isomet High-Speed was used.



Fig. 3.57. Sample sectioned by 0.5 mm diamond blade.



Fig. 3.58. Sample was sectioned into 2 equal halves.



Fig. 3.59. The one half section of the sample.



Fig. 3.60. Sample was sectioned into 4 quadrants.



Fig. 3.61. Quadrant section has 2 sites for evaluation.



Fig. 3.62. The digital Video Camera with Computerized Image-Analyzer System.