CHAPTER 3

MATERIALS AND METHODS

3.1 MATERIALS

Two denture base polymers and three chairside reline materials selected in this study are shown in Table 3.1.

3.1.1 Denture Base Materials

3.1.1.1 Heat-and light-polymerized UDMA

Eclipse material is based on urethane dimethacrylate chemistry. It is supplied in a light proof package and in arch shape form (Fig. 3.1). Separating agent (Al-Cote, Dentsply) (Fig. 3.2) is used for easy removal of the polymerized denture base from the working model. A layer of air barrier coating (ABC, Dentsply) (Fig. 3.2) provides a barrier between the surface of the material and atmospheric oxygen during polymerization. Air inhibition of the surface layer polymerization is prevented by applying an air barrier coating before final polymerization. The material is polymerized in the processing unit (Dentsply Int, York, USA) which uses six halogen lamps of 44 Volt each. The light triggers the polymerization process, but both heat and light are needed to polymerize the material. According to the manufacturer the temperature within the processing unit reaches a maximum of 129°C during polymerization. As claimed by the manufacturer, Eclipse uses different initiator species from Triad Denture Resins to ensure high strength.

3.1.1.2 Heat-polymerized PMMA

Meliodent heat-polymerized PMMA is supplied in a powder and liquid form (Fig. 3.3). The powder contains approximately 97% poly(methyl methacrylate) polymer and benzoyl peroxide as an initiator. The liquid is methyl methacrylate monomer with 6% ethylene glycol dimethacrylate as the cross linking agent. The manufacturer recommended its applications for dentures, denture repairs, relining, extending of dentures, for completing metal dentures and for orthodontics use.

Table 3.1. Materials used in this study.

Brand name	Material type	Main composition	Manufacturer	Batch number
Eclipse Base Plate	Heat- and light-polymerized UDMA	Single paste component Matrix: urethane dimethacrylate Filler: silica and polymethacrylate beads	Dentsply Int, York, USA.	Lot 030909
Meliodent	Heat-polymerized PMMA	Powder: polymethyl methacrylate Liquid: methyl methacrylate, ethylene glycol dimethacrylate	Heraeus Kulzer, Hanau, Germany.	Powder: Lot A1397B-2 Liquid: Lot 012155
Meliodent RR	Laboratory-processed auto-polymerized PMMA reline material	Powder: polymethyl methacrylate Liquid: methyl methacrylate, ethylene glycol dimethacrylate, ethylene glycol p-toluidine	Heraeus Kulzer, Hanau, Germany.	Powder: Lot RB136B-14 Liquid: Lot 013029
Secure	Intra-oral auto-polymerized reline material (HEMA-based)	Two paste component: Base: hydroxyethyl methacrylate Catalyst: benzoyl peroxide	Imtec Corp., Ardmore, USA.	Lot 137872019
Kooliner	Intra-oral auto-polymerized reline material (PEMA-based)	Powder: polyethyl methacrylate Liquid: iso-butyl methacrylate, dimethyl-p-toluidine	GC America, Alsip, USA.	Lot 0406021
Secure	Bonding Agent	Dichloromethane	Imtec Corp., Ardmore, USA.	Lot 137872019
ABC	Air barrier coating	Not known	Dentsply Int, York, USA.	Lot 050209
Al-Cote	Separating Agent	Not known	Dentsply Int, York, USA.	Lot 050414

3.1.2 Reline Materials

3.1.2.1 Auto-polymerized PMMA laboratory reline material (Meliodent RR)

Meliodent RR, an acrylic resin commonly used for repairs or relining dentures, is a laboratory-processed material (Fig. 3.4). It is supplied in a powder and liquid form. The powder consists of PMMA and benzoyl peroxide as the initiator. The monomer liquid consists of MMA with the additional of cross linking agent and ethylene glycol p-toluidine (1%) as an activator. The polymerization is carried out in a pressure pot at a temperature of 55° C, at 2 bar pressure for 10 minutes.

3.1.2.2 Auto-polymerized HEMA-based intra-oral reline material (Secure)

Secure is a direct reline material and it is specially indicated for picking-up metal caps of the O- ball abutment of mini dental implants (IMTEC Sendax MDI, USA) into the fitting surface of an implant overdenture. The base material is hydroxethylmethacrylate (HEMA) and benzoyl peroxide is the catalyst. It is supplied in a cartridge in two paste forms and it is dispensed using a gun extruder (Fig. 3.5). The adhesive liquid provided in the set contains dichloromethane. It is claimed by the manufacturer that it release low heat during polymerization therefore it can be safely used directly in the mouth.

3.1.2.3 Auto-polymerized PEMA-based intra-oral reline material (Kooliner)

Kooliner is a hard chairside reline material (Fig. 3.6). The powder contains polyethyl methacrylate as polymer constituent and the liquid contains iso-butyl methacrylate as monomer constituent and dimethyl-p-toluidine as the activator. No cross-linking agent or plasticizer is present in the liquid. Particle size distribution of the powder components is mainly in the range of 50-100 um (Arima et al., 1996). Kooliner material has a low peak polymerization temperature, approximately 43°C at 7 minutes after mixing, which is considered suitable for intra-oral use.

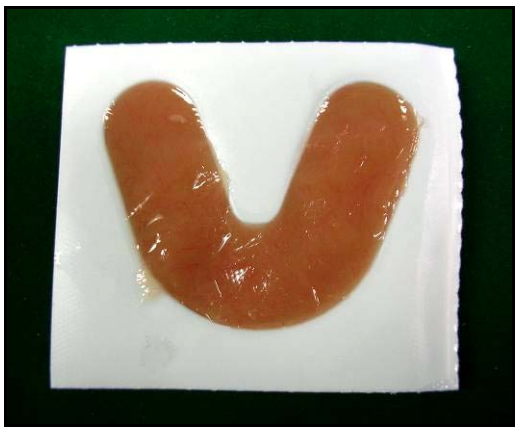

Fig 3.1. Eclipse base plate material.

Fig. 3.2. Eclipse base plate material with
Air Barrier Coating and Separating Agent.

Fig. 3.3. Powder and liquid of Meliodent
heat-polymerized PMMA denture base material.

Fig. 3.4. Powder and liquid of Meliodent RR auto-polymerized reline material.

Fig. 3.5. Secure intra-oral reline material with the cartridge dispenser, mixing tip and its adhesive.

Fig.3.6. Powder and liquid of Kooliner intra-oral reline material.

3.2 METHODS

The methods used for specimen preparation and the relining procedure are explained below. Table 3.2 shows the processing method employed for denture base specimen fabrication and relining procedures.

3.2.1 Preparation of denture base specimens

Specimens of both heat-polymerized PMMA (Meliodent) and light-polymerized UDMA (Eclipse) were prepared by investing brass columns measuring 15 mm in diameter and 4 mm in height in stone. The powder: liquid ratio of 100 g of stone to 30 ml of water was used to prepare gypsum moulds. Gypsum mould for Meliodent denture base specimens was prepared by investing multiple brass columns in a metal flask (Fig. 3.7). For Eclipse specimens, the mould was prepared by investing multiple brass columns in stone, which was sandwiched between two circular Perspex blanks of a diameter of 11 mm (Fig. 3.8). The size of the mould was selected to fit on the rotating table of the polymerization oven.

3.2.1.1 Heat-polymerized PMMA specimens

The recommended powder: liquid ratio of 35g: 10 ml of Meliodent was used in the preparation of the specimens. Cold mould seal was applied beforehand onto the mould as a separating media. The mixture was packed into the moulds when it reached the dough stage, then the flask was kept under pressure in a spring clamp for 15 minutes. The polymerization was carried out in a water bath (Acrydig 10, Manfredi) using the curing cycle of 7 hours at 70ºC followed by 1 hour at 100ºC. A total of 30 specimens were prepared.

3.2.1.2 Light-polymerized UDMA specimens

The stone mould and Eclipse base plate resin were preheated in the conditioning oven to 55°C for 2 minutes. This was to allow the mould to dry and for Eclipse resin to be flowable for easier adaptation of the material. Separating agent was applied beforehand onto the mould and the warmed resin was adapted into the mould using finger pressure (Fig. 3.9). Air barrier coating was applied on to the exposed surface to prevent inhibition of polymerization by oxygen. Polymerization was carried out by placing the mould in the centre of the rotating table of the light-curing unit (Fig. 3.10 and 3.11) and exposing it to visible light of 400-500nm for 10 minutes using Menu no. 1 as in the manufacturer's instruction for baseplate cure. Six halogen lamps of 41 Volts each within the unit were required for polymerization. A total of 40 specimens were prepared.

Both heat-polymerized and light-polymerized denture base specimens were trimmed with an acrylic bur to remove excess material. The denture base surface was wet grounded using a grinding and polishing machine (Metaserv® 2000) on 600 grit silicone carbide paper before mounting.

3.2.2 Mounting of denture base specimens

Denture base specimens (Fig. 3.12) were then embedded in clear self-cure epoxy resin (Miracon ®) with the surface to be relined exposed. This was performed so that the embedded specimens could be mounted to the testing machine. A circular mounting cup (Buehler®) was used to hold the specimen in place before the epoxy resin was poured in (Fig. 3.13 & 3.14). The internal surface of the cup was coated with vaseline. Following overnight curing of the resin, the mounted specimen was removed (Fig. 3.15) and washed under running water. The exposed end of the denture base surface was wet grounded using a grinding and polishing machine (Metaserv® 2000) on 600 grit

silicone carbide paper (Fig. 3.16 and 3.17). All the specimens were stored in water at 37ºC in an incubator chamber (Memmert Gmbh) for 30 days (Fig. 3.18) before relining.

3.2.3 Relining

For the relining procedure, a brass ring of 6 mm internal diameter and 2.5 mm height (Fig. 3.19) was used to confine the reline materials. It was placed at the centre of the exposed surface of the mounted denture base specimens (Fig. 3.20). Prior to relining, the specimen surface was cleaned with spirit and rinsed with distilled water. Surface moisture was removed with clean gauze and it was left to air dry for 30 seconds. After mixing, the reline material was poured into the brass ring cavity (Fig. 3.21).

3.2.3.1 Preparation of reline materials

3.2.3.1.1 Meliodent RR material

The reline resin was mixed according to the recommended powder to liquid ratio of 10 g powder: 7 ml liquid. Spatulation of the mixture was done for 30 seconds and the resin was poured onto the denture base surface in the brass ring. Polymerization was carried out for 10 minutes in a pressure pot at 55°C, with a 2 bar pressure to simulate laboratory reline procedure. After setting, the brass ring was removed (Fig. 3.22). The diameter of the reline material was confirmed with a digimatic micrometer (Mitutoyo), shown in Fig. 3.23.

3.2.3.1.2 Secure material

The material was supplied in a cartridge which can be dispensed using a gun. Two layers of adhesive were applied on to the exposed surface of the denture base specimens using a brush and the adhesive layer was let to dry in the open air for 30 seconds. Polymerization was carried out for 10 minutes in a temperature-controlled chamber

(Memmert Gmbh) which was set at 37°C. This was to simulate the temperature of the oral cavity.

3.2.3.1.3 Kooliner material

The powder and the liquid with a ratio of 15 ml powder to 6 ml liquid was mixed and stirred thoroughly for 30 seconds and applied onto the denture base surface. Polymerization was carried out for 10 minutes in a temperature-controlled chamber (Memmert Gmbh) which was set at 37°C.

3.2.3.1.4 Eclipse base plate resin used as relining material

For this additional group, the reline resin was warmed in the conditioning oven of 55°C for 2 minutes. The softened resin was then adapted onto the denture base surface within the brass ring. Air barrier coating was applied on its surface. Polymerization was performed for 6 minutes in the polymerization oven (Fig. 3.24) using menu No. 4 as advocated by the manufacturer for relining.

3.2.4 Shear Bond Strength Testing.

All bonded specimens were stored in distilled water at 37ºC for 24 hours as described in ISO specification 11405:2003 for short-term water storage. The shear bond test was carried out using an Instron Universal Testing machine (Instron Inc.,USA) (Fig. 3.25) at a crosshead speed of 1.0 mm/min. The specimen was fixed in the solid block and compressive load was applied with a knife-edged blade placed parallel to the material interface on the load testing machine (Fig. 3.26). The test was performed dry under uniform atmospheric conditions at a temperature of 23ºC.

The value of the bond strength was computed from the following equation.

$$F = \frac{N}{A}$$

Where F = shear bond strength (MPa)

N = maximum force exerted in specimen (in Newton)

A = size of the bonding area (mm^2)

3.2.5 Examination of Failure Modes

For all specimens, the interface where failure occurred was inspected using a stereo microscope (Kyowa SD-2PL) at a magnification of ×10 (Fig. 3.27). This allowed determination of the nature of the failure as adhesive, cohesive, or mixed. The following criteria as described by Sarac et al, (2005) were used in determining the type of failure at the denture base-reline interface.

i) Cohesive failure: There are more than 50% traces of denture reline material on the denture base surface or vice versa.

ii) An adhesive failure: No traces of denture reline material on the denture base surface or vice versa.

iii) Mixed failure: There are less than 50% traces of denture reline material on the denture base surface or vice versa.

3.2.6 Scanning Electron Microscope (SEM) evaluation

The UDMA and PMMA denture base specimen surfaces were examined under a field emission SEM (Quanta 200, FEI, Hillsboro, USA) using low vacuum imaging modes prior to relining. The effect of treating the PMMA and UDMA denture base surfaces with dichloromethane adhesive was also observed. A sample of bonding interface of UDMA denture base specimen that was relined using the same UDMA material and another sample of PMMA denture specimen that was relined with intra-oral HEMA-based material (Secure) were also selected for SEM evaluation.

3.2.7 Statistical Analysis

One-way analysis of variance (ANOVA) was used for comparison of shear bond strength amongst seven denture base-reline polymer combinations. To determine the interaction between denture base polymers and reline materials, a two-way ANOVA was used. For the two-way ANOVA, the result for UDMA denture base-UDMA Eclipse reline combination was excluded. Levene's test for equality of variance showed a significant departure from homogeneity ($p<0.05$). Hence, Post-hoc Dunnett T3 test was used to compare the significant difference between various relined specimens at 95% confidence interval.

Table 3.2: Processing method for various denture base and reline materials used in this study.

Brand name	Processing method
Eclipse base plate resin	Heat-and light-polymerized in the processing unit for 10 minutes (using Menu 1). Visible light of wavelength of 400-500nm using six halogen lamps of 41 Volts.
Meliodent	Compression-molding technique; heat processed at 70° C for 7 hours, followed by 100°C for 1 hour.
Meliodent RR	Auto-polymerized in pressure pot at a temperature of 55°C and pressured at 2 bars for 10 minutes.
Secure	Auto-polymerized at 37°C with polymerization completed in 10 minutes.
Kooliner	Auto-polymerized at 37°C with polymerization completed in 10 minutes.
Eclipse base plate resin for relining	Heat-and light-polymerized in the processing unit for 6 minutes (using Menu 4).

Fig. 3.7. Investments of brass columns in metal flask for Meliodent denture base specimens preparation.

Fig. 3.8. Mould for preparation of Eclipse denture base specimens.

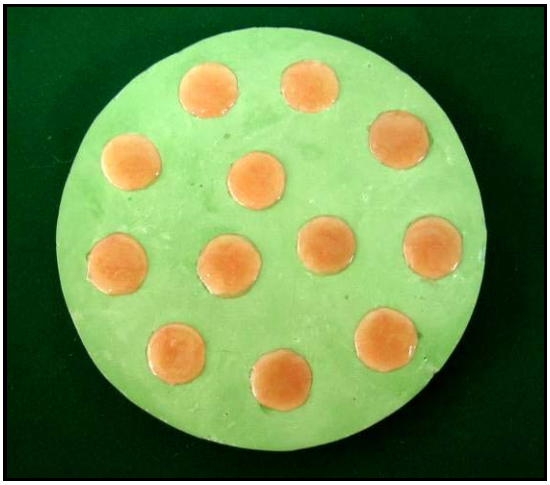

Fig. 3.9. Eclipse material after adaptation in the mould.

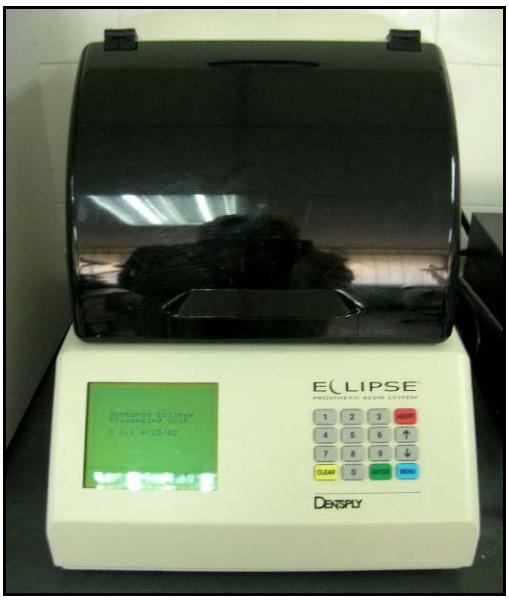

Fig. 3.10. Eclipse processing unit.

Fig. 3.11. Eclipse denture base specimens in the processing unit during polymerization

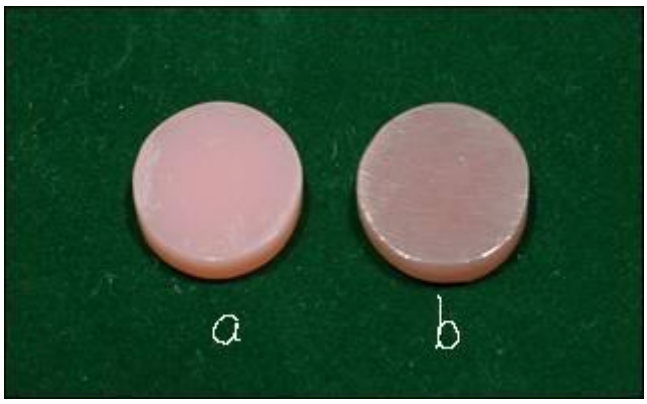

Fig. 3.12. An example of a) Meliodent and b)Eclipse denture base specimens.

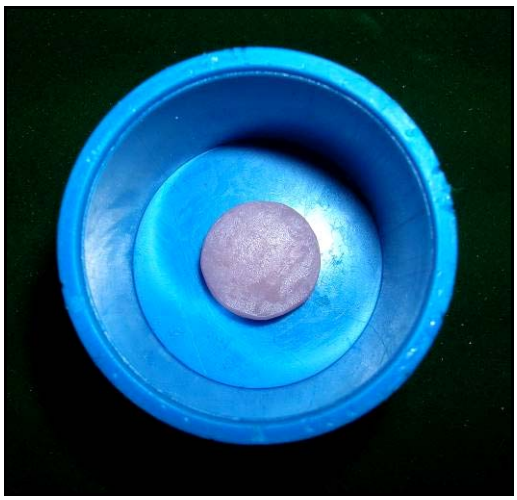

Fig. 3.13. Denture base specimen in the mounting cup.

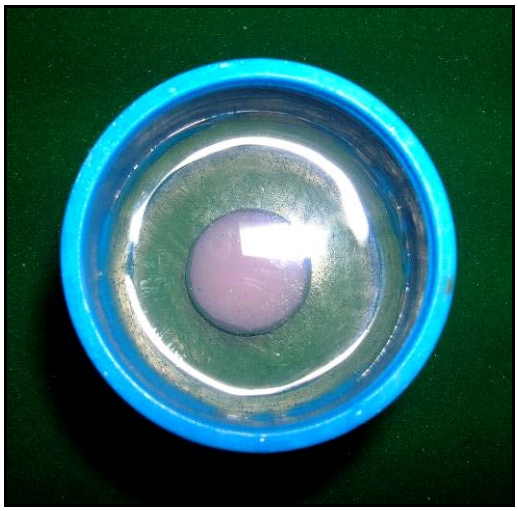

Fig. 3.14. Denture base specimen embedded within epoxy resin in the mounting cup.

Fig. 3.15. The mounted denture base specimen after setting.

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Fig. 3.16. Grinding and polishing machine (Metaserv 2000, Buehler).

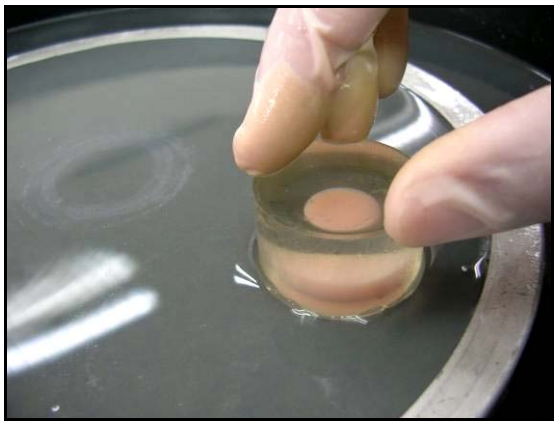

Fig. 3.17. Grinding of the denture base surface under water irrigation.

Fig. 3.18. Incubator chamber (Memmert Gmbh).

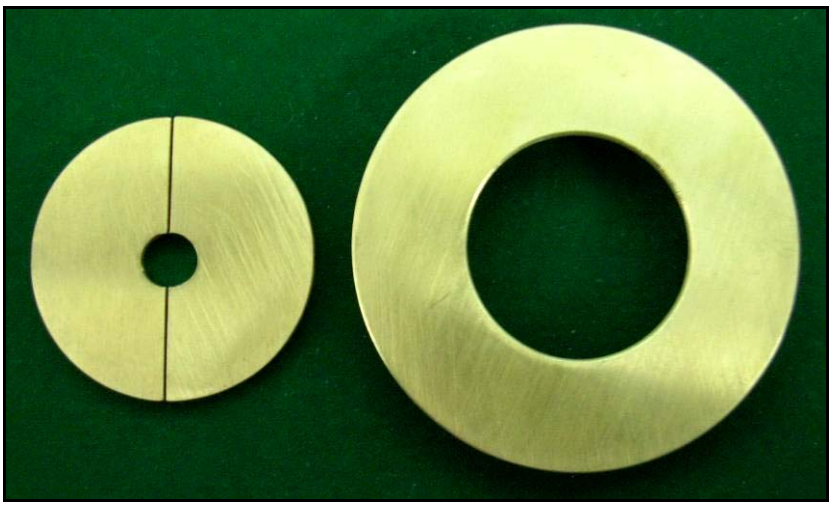

Fig. 3.19. The two parts of brass ring apparatus.

Fig. 3.20. Brass ring position on the mounted denture base specimen, with the denture base surface exposed in the centre of the ring.

Fig. 3.21. Reline material being applied onto the denture base surface, which was confined in the brass ring.

Fig 3.22. The relined denture base specimen mounted in epoxy resin.

Fig. 3.23. The diameter of specimen being measured using a digital micrometer (Mitutoyo Mfg.)

Fig. 3.24. Relined Eclipse denture base specimen in the polymerization unit.

Fig. 3.25. The Instron machine.

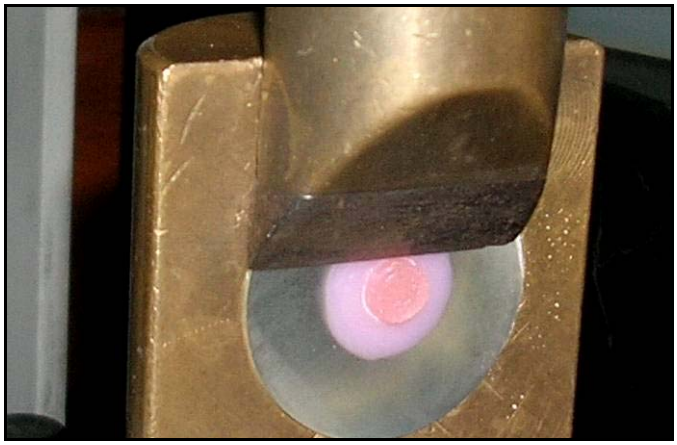

Fig. 3.26. Close-up view of the knife-edged blade during shear bond test.

Fig. 3.27. Stereomicroscope for examination of failure mode (KyowaSD-2PL).