CHAPTER 2

MATERIALS AND METHODS

MATERIALS AND METHODS

2.1 MATERIALS

Three denture base polymers were selected for this study; light-polymerized, heat-polymerized and chemically-polymerized denture base polymers.

Materials	Type	Manufacturer	Batch No.
Eclipse	Light-polymerized urethane dimethacrylate polymer	Dentsply,USA	030204
Meliodent	Heat-polymerized PMMA	Bayer Dental, Newbury,UK	A1397B-2
Probase Cold	Chemically-polymerized PMMA	Ivoclar Vivadent Ltd., Liechtenstein	D53289

2.1.1 Light-polymerized urethane dimethacrylate based polymer

Eclipse which is light-polymerized denture base polymer and was selected for this study. It contains a blend of urethane dimethacrylate monomer, submicron particles of silica, polymethacrylate beads and camphoroquinone. The polymerization process was activated by placing the resin on a rotating table, in a light processing unit and exposing it to high intensity visible light of 400 to 500 nm.

Figure 2.1: Light-polymerized urethane dimethacrylate denture base polymer

Figure 2.2: Light-polymerized urethane dimethacrylate denture base polymer material supplied in arch form

2.1.2 Heat-polymerized PMMA

In this study, Meliodent heat-polymerized PMMA was selected (Figure 2.3). It is supplied in a powder and liquid form. The powder contains poly(methyl methacrylate) polymer and the liquid is methyl methacrylate monomer.

2.1.3 Chemical-polymerized PMMA

Probase Cold material which is a chemically-polymerized denture base polymer and was selected for this study (Figure 2.4). It is supplied in a powder and liquid form. The powder contains poly(methyl methacrylate) polymer and the liquid is methyl methacrylate monomer.

Figure 2.3: Heat-polymerized denture base polymer

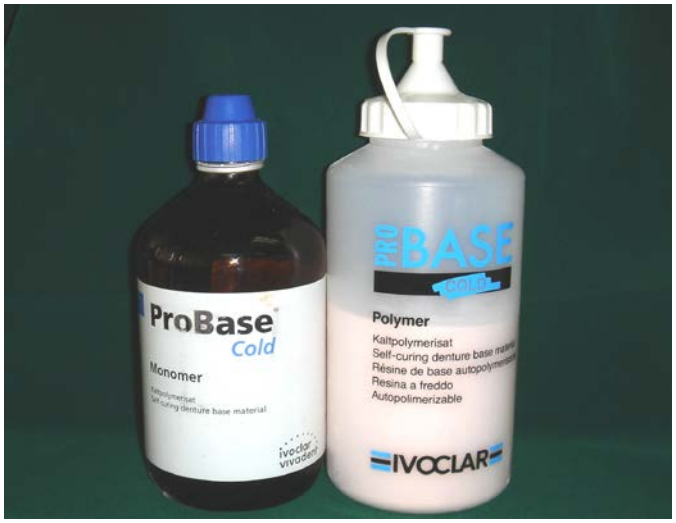

Figure 2.4: Chemically-polymerized denture base polymer

2.2 METHOD

The methods used for preparing specimens, and the tests for the microhardness and three-point flexural bending are explained below.

2.2.1 Surface hardness of light-polymerized denture base polymer (Eclipse)

- A pilot study.

2.2.1.1 Specimen preparation of Eclipse

The specimens were divided into 6 groups and each group was polymerized with one of the following polymerization times; 4 min, 6 min, 8 min, 10 min, 12 min and 14 min. There were 15 specimens in each group. The specimens were prepared by first investing a Perspex block of size 70 x 50 x 3mm (Figure 2.5), to make a stone mould by using a conventional metal flask (Figure 2.6). The mould was preheated in a special oven (Figure 2.7) at 55˚ C for 2 minutes before the material was adapted to the mould. Separating agent was applied beforehand onto the mould and the resin was adapted onto the mould by using finger pressure. Air barrier coating agent was applied on the resin surface, to prevent inhibition of polymerization by oxygen, before the materials were polymerized in the processing unit. To allow uniform thickness of the specimens, a glass slab was pressed onto the material after finger adaptation.

The polymerization process was activated by placing the resin on a rotating table, in a light processing unit and exposing it to high intensity visible light of 400 to 500 nm (Figure 2.8 and Figure 2.9). Each specimen was exposed to the light for various lengths of time of 4 min, 6 min, 8 min, 10 min, 12 min and 14 min. The polymerized specimens were removed from the mould. The upper surface (irradiated surface) was labelled as surface A, and the lower surface (non-irradiated surface) was labelled as surface B.

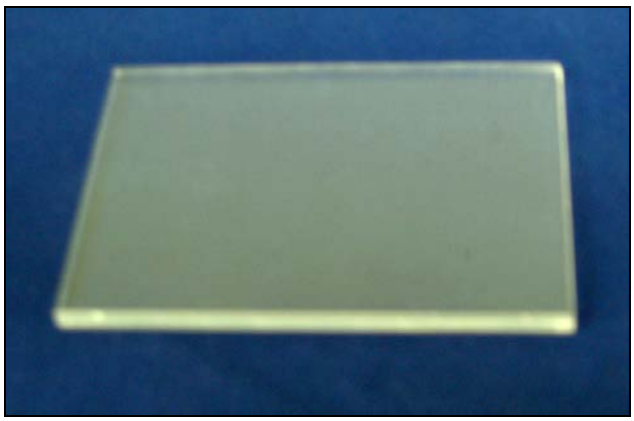

Figure 2.5: Perspex block

Figure 2.6: Investment of Perspex block in metal flask

Figure 2.7: Oven to preheat the model
for light-polymerized polymer

Figure 2.8: Eclipse processing unit

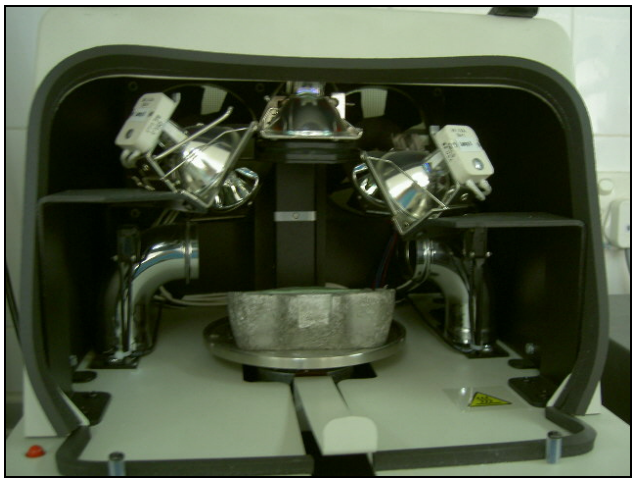

Figure 2.9: Flask on the rotating table in processing unit

The excess margins and thickness were trimmed on a grinding machine under irrigation. This procedure was followed by the manual cutting of each trimmed specimen block into 4 specimen strips by using a bandsaw to the size of 65 x 10 x 2.5 mm (Figure 2.10). The specimens were ground while wet and by using a polishing machine (Stuer Rotopole, Buehler Ltd., USA) with silicon carbide paper disks of grade 600 and 1,000 (Figure 2.11). Each specimen was individually measured to a final dimension by use of a micrometer (Mitutoyo Ltd., Tokyo, Japan) (Figure 2.12, 2.13 and 2.14). All specimens were immersed in water at 37°C for 30 days in an incubator prior to testing.

2.2.1.2 Hardness test of Eclipse

Specimens were tested for surface hardness immediately after removal from the water. Each specimen was held securely during the test by placing it on a table and locking it in position. Microhardness was measured in terms of depth of indentation of the pyramid diamond indenter (Vicker's indenter) by using a Shimadzu microhardness tester (Figure 2.15). The instrument measured the difference between the depth of penetration of an indenter into a specimen (Figure 2.16) under contacting load of 1± 0.2g and a penetrating load of 300 ±1g after 15 second of penetration. The surface hardness was measured on the irradiated surface and on the non-irradiated surface.

Figure 2.10: Final size of specimen

Figure 2.11: Polishing machine, Metaserv® 2000.

Figure 2.12: Length of specimen measured by micrometer

Figure 2.13: Width of specimen measured by micrometer

Figure 2.14: Thickness of specimen measured by micrometer

Figure 2.15: Shimadzu microhardness tester

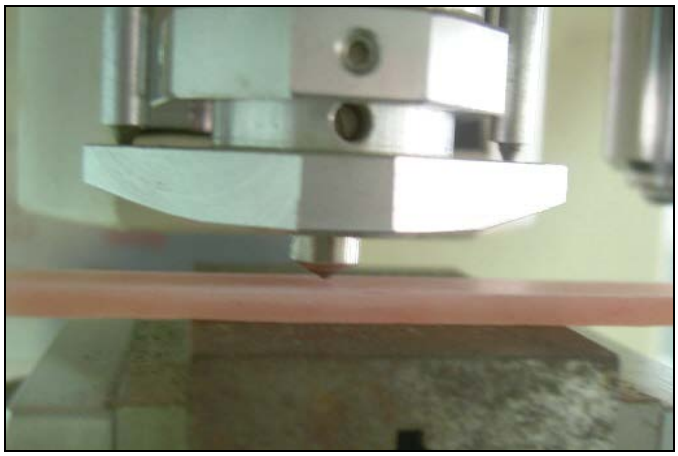

Figure 2.16: Close-up view of Shimadzu microhardness tester during the indentation of pyramid diamond indenter on the specimen

2.2.2 Hardness test of three denture base polymers

2.2.2.1 Specimen preparation

2.2.2.1.1 Eclipse

Specimen preparation followed the procedure described as in the pilot study, but a 10 minute polymerization time was selected to polymerize the material because it was found to be the optimal polymerization time as well as the length of time recommended by the manufacturer when constructing dentures.

2.2.2.1.2 Meliodent

Fifteen specimens were prepared by first investing a Perspex block of size 70 x 50 x 3mm, to make a stone mould by using a conventional metal flask. Cold mould seal (Bayer Dental Div., UK) was used as the separating media. The recommended powder:liquid ratio of 23.4 gm of powder to 10 ml of liquid was used in the preparation of the specimens for this and subsequent tests. The weight of the powder was measured by using an electronic balance (Tanaka scale warks Co., Ltd. Japan) (Figure 2.17).

The resin was packed into the flask when the mixture reached the dough stage. A thin layer of polythene sheet was used during trial packing. At final packing, the flasks were maintained under pressure for 15 minutes. The polymerisation cycle was accomplished in a water bath unit with a cycle of seven hours at 70°C followed by one hour at 100° C. The specimens were bench-cooled before deflasking.

2.2.2.1.3 Probase Cold

Fifteen specimens were prepared by first investing a Perspex block of size 70 x 50 x 3mm, to make a stone mould in a conventional metal flask. The stone surface was coated with two layers of separating agent (Ivoclar Vivadent Ltd., Liechtenstein) and

was allowed to dry completely. The recommended powder:liquid ratio of 20.5 gm of powder to 10 ml of liquid was used in the preparation of the specimens. The resin mixture was packed into the flask when it had reached the dough stage. The flasks were maintained under constant pressure at 80 bar for 30 minutes at room temperature (23ºC).

For all groups, the specimen blocks were removed from the mould and the excess margins and thickness were trimmed on a grinding machine (Buehler Ltd., USA). This procedure was followed by the manual cutting of each trimmed specimen block into 4 specimen strips using a bandsaw to the size of 65 x 10 x 2.5mm. They were wet ground using a polishing machine (Stuer Rotopole, Stuers) with silicon carbide paper disks of grade 600 and 1,000. Each specimen was individually measured to a final dimension by use of a micrometer. All specimens were immersed in water at 37°C for 30 days prior to testing.

2.2.2.2 Test procedure

The test procedure was conducted followed the same procedure as in the pilot study, except that for Eclipse, the specimens were tested for hardness on the irradiated surface only.

2.2.3 Three-point flexural bending test

2.2.3.1 Specimen preparation

The specimen preparation for the 3-point flexural bending test followed the same procedure as that used in the hardness test.

2.2.3.2 Test procedure.

The specimens were tested on the Instron machine (Figure 1.18) according to the ISO 1567 specification. They were symmetrically placed and centred on the testing rig at a span of 50 mm. A loading head that apply the load at a constant rate of 5 mm/min across the centre of the specimens was used for all the specimens.

The testing was carried out in water bath at 37°C. The values for the modulus of elasticity and flexural strength were recorded. The values of flexural strength, which is also referred to as the modulus of rupture, was computed from the following equation.

$$S = \frac{3NI}{2bd^2}$$

Where S = modulus of rupture (N/mm2)

N = maximum force exerted on specimen (N)

I = distance between supports (mm)

b = breadth of specimen (mm)

d = depth of specimen (mm)

The modulus of elasticity was computed from the following equation.

$$E = \frac{\text{Stress}}{\text{Strain}} = \frac{FI^3}{4ybd^3}$$

Where E = modulus of elasticity (N/mm2)

F = force at point p (N)

I = distance between the supports (mm)

b = breadth of specimen (mm)

d = depth of specimen (mm)

y = deflection at point p (mm)

the values obtained from the computation were expressed in MPa.

Figure 2.17: Electronic balance

Figure 2.18: Instron machine

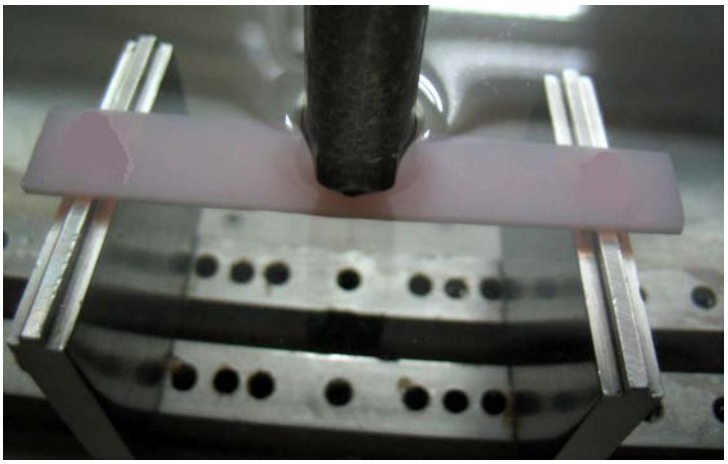

Figure 2.19: Close-up view of Instron machine during the loading of specimen

2.2.4 Statistical analysis

One-way analysis of variance (ANOVA) and t-test were used to analysed the result. Post-hoc analysis using Scheffe's test was carried out if there were significant differences among the groups from the ANOVA test. In this study, 'p' value was selected at $p = 0.05$.