Development of a novel resin for provisional prostheses using hyperbranched polyurethane acrylate and triethylene glycol dimethacrylate - An in vitro study

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Purpose: To develop a novel resin for provisional prostheses using hyperbranched polyurethane acrylate (HBPUA) and triethylene glycol dimethacrylate (TEGDMA) with promising mechanical properties and low volumetric shrinkage.

Methods: Four groups including TIH3-0 (100 wt% TEGDMA), TIH3-30 (30 wt% HBPUA + 70 wt% TEGDMA), TIH3-60 (60 wt% HBPUA + 40 wt% TEGDMA), and TB-60 (60 wt% bisphenol A-glycidyl dimethacrylate + 40 wt% TEGDMA) were prepared and commercial Luxatemp (DMG) was used for comparison. Fourier transform infrared spectroscopy and gel permeation chromatography were used for material characterization. Mechanical properties including microhardness, flexural strength, flexural modulus, and load energy were measured before and after water immersion. Physical properties measurement included weight changes, solubility, water absorption, surface hydrophobicity, and volumetric shrinkage. Finally, biocompatibility was evaluated using a 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide assay.

Results: The number- and weight-average molecular weights of the HBPUA were approximately 870 and 1480, respectively. The addition of HBPUA to TEGDMA increased the mechanical strength considerably. Although the weight changes and water absorption of TIH3-60 were higher than those of Luxatemp, the microhardness, flexural strength, flexural modulus, load energy, solubility, shrinkage, and biocompatibility of TIH3-60 were either comparable or superior to those of Luxatemp.

Conclusion: Based on the findings of the present study, TIH3-60 has potential for development as a new provisional material.

Keywords: Mechanical characteristics, Hyperbranched polyurethane acrylates, Triethylene glycol dimethacrylate, Provisional material, Micro-hardness

1. Introduction

Provisional prostheses are designed to enhance esthetics, stabilization, and/or function for a limited period of time, after which they are replaced with definitive dental prostheses[1]. Fabricating an ideal fixed provisional prosthesis is crucial for gingival health and protection of the pulp, for prosthetically guided tissue healing to achieve an acceptable emergency profile, for reduction of dental abutment migration, and for assessment of the prospective form and function of a definitive prosthesis[2]. Perfect adaption should feature lower polymerization shrinkage and should remain functional during thermal and mechanical cycling[3]. Additionally, a provisional prosthesis is subjected to masticatory forces in the oral environment, and therefore requires specific mechanical properties, such as rigidity, flexural strength, and long-term dimensional stability[4].

Provisional prosthesis restoration materials can be divided into two main groups according to their chemical composition: those based on monomethacrylates or acrylic resin (including poly-methylmethacrylate (PMMA) and polyethyl/butyl methacrylate), and those based on dimethacrylates or bis-acryl/composite resins such as bisphenol A-glycidyl dimethacrylate (Bis-GMA)[2,5]. The major disadvantages of the acrylic resin provisional materials are the dimensional stability after polymerization and low mechanical strengths[2,6]. A composite resin used as a provisional material comprises Bis-GMA and triethylene glycol dimethacrylate (TEGDMA), with the latter used as a diluent to improve the degree of conversion[7,8]. Mixtures are always prepared from Bis-GMA and TEGDMA at a ratio of 7:3 or 6:4. Although these compositions improve the rate of polymerization shrinkage, the shrinkage rates range from 2.01% to 4.6%[9].
Hyperbranched polymers have many peripheral, pending, and side functional groups. These highly branched structures give hyperbranched polymers distinctive characteristics such as low viscosity and high solubility, which differentiates them from linear polymers[14]. For example, hyperbranched polyester has been used to develop a new tissue conditioner to improve viscoelastic stability[15]. Polyurethane acrylates have been widely used as UV-curable coatings because of their favorable mechanical properties and chemical resistance[16]. In addition, polyurethane acrylates have been applied in wound dressings and medical implants and devices because of their excellent biocompatibility, good oxygen permeability, and barrier properties[17,18]. In this study, Bis-GMA was replaced with a hyperbranched polyurethane acrylate (HBPUA) polymer that was synthesized via the copolymerization of 1,1,1-tris(hydroxymethyl) propane (TMP), isophorone diisocyanate (IPDI), and 2-hydroxyethyl acrylate (HEA). A novel resin for provisional prostheses was developed using HBPUAs and TEGDMA. The aim of this study was to measure the mechanical properties including microhardness, flexural strength, flexural modulus, and load energy before and after water immersion. In addition, the aging effect of water immersion was evaluated, including weight change, solubility, and water absorption. Finally, the shrinkage and biocompatibility were investigated using Archimedes’ principle of buoyancy and a 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) assay, respectively. Using Fourier transform infrared (FTIR) spectroscopy (FT-720, Horiba, Kyoto, Japan), the molecular weights of the branched PUAs were measured using gel permeation chromatography (GPC) coupled to a column (PU-2080 PUMP/RI 2000, Jasco, Tokyo, Japan). The molar ratio of TMP/IPDI/HEA was 1:3:3. In a three-necked flask filled with acetone, TMP, IPDI, and HEA were dissolved and stirred by a magnet in a constant-temperature water tank at 45 °C for 5 h, followed by reduced pressure distillation (60 cm-Hg) for 30 min at 50 °C. The thin films of the reactant and product were analyzed using Fourier transform infrared (FTIR) spectroscopy (FT-720, Horiba, Kyoto, Japan). The molecular weights of the branched PUAs were determined using gel permeation chromatography (GPC) coupled to a column (PU-2080 PUMP/RI 2000, Jasco, Tokyo, Japan; KS-804L, Shodex, Kyoto, Japan) in which tetrahydrofuran (THF) was the carrier solvent, and the column was calibrated with polystyrene standards. Figure 1 shows the chemical structure of TMP, IPDI, HEA, and HBPUA.

### 2. Materials and methods

#### 2.1. Preparation of HBPUA

The HBPUA was prepared via the polymerization of trifunctional monomers: TMP (B3, Acros Organics, Geel, Belgium), IPDI (A2, Acros Organics), and HEA (BR, Tokyo Chemical Industry Co., Tokyo, Japan). The molar ratio of TMP/IPDI/HEA was 1:3:3. In a three-necked flask filled with acetone, TMP, IPDI, and HEA were dissolved and stirred by a magnet in a constant-temperature water tank at 45 °C for 5 h, followed by reduced pressure distillation (60 cm-Hg) for 30 min at 50 °C. The thin films of the reactant and product were analyzed using Fourier transform infrared (FTIR) spectroscopy (FT-720, Horiba, Kyoto, Japan). The molecular weights of the branched PUAs were determined using gel permeation chromatography (GPC) coupled to a column (PU-2080 PUMP/RI 2000, Jasco, Tokyo, Japan; KS-804L, Shodex, Kyoto, Japan) in which tetrahydrofuran (THF) was the carrier solvent, and the column was calibrated with polystyrene standards. Figure 1 shows the chemical structure of TMP, IPDI, HEA, and HBPUA.

#### 2.2. Microhardness, flexural strength, flexural modulus, and load energy measurement

Five groups were included in this study. Group 1 contained only TEGDMA (TCI, Tokyo Chemical Industry Co.) presented as THI3-0. Group 2 contained 30 wt% HBPUA and 70 wt% TEGDMA, presented as THI3-30. Group 3 contained 60 wt% HBPUA and 40 wt% TEGDMA, presented as THI3-60. Group 4 contained 60 wt% Bis-GMA (Sigma-Aldrich, St. Louis, MO, USA) and 40 wt% TEGDMA, presented as TB-60. Group 5 contained the commercial product Luxatemp Star for provisional prostheses. Groups 1–4 all had 0.1 mol (C=C) camphorquinone (CQ, Acros Organics) and 0.1 wt% ethyl 4-((dimethylamino) benzoate (EDMAB, Acros Organics). All photocuring procedures were conducted with a halogen lamp (Triad 2000, Dentsply Sirona, New York, USA) for 1 min at a distance of 1.5 cm with a light intensity of 2150 mW/cm² which was delivered perpendicularly to the specimen. Figure 2 shows the flow chart of methodology used in this study.

Microhardness—Materials were placed in a mold (5-mm diameter and 1.5-mm thickness) and light cured for 1 min. Five indentations were made on the top surface of each specimen using a microhardness tester (HMV-2, Shimadzu, Kyoto, Japan) and the average Vickers hardness number (VHN) was obtained (n = 12). A diamond indenter with a load of 0.98 N and a dwell time of 15 s was used to make indentations.

Flexural strength, flexural modulus, load energy—Materials were placed in a mold (25 × 2 × 2 mm) and light cured for 1 min to produce rectangular specimens according to the ISO 4049 standard. Three-point bending of the specimens was tested using a universal testing machine (Instron 5566, Instron Corp., Norwood, USA) at a crosshead speed of 1.0 mm/min. The flexural strength, flexural modulus, and load energy were calculated (n = 12) using the following equations[19]:



**Fig. 1.** Chemical structures of TMP, IPDI, HEA, and HBPUA.
Flexural modulus: $\frac{PL^3}{4BD^3}M$

Load energy: $\frac{P}{2}$

where $P$ is load at fracture, $L$ is span (20 mm), $B$ is specimen width (2 mm), $D$ is specimen thickness (2 mm), $M$ is the deflection corresponding to load $P$ at a point in the straight-line portion, and $l$ is maximum deflection.

2.3. Weight change, solubility, and water absorption measurement

Specimens were prepared as disks with a diameter of 6 mm and thickness of 1.5 mm ($n = 12$). The procedures and instruments for measuring weight change, solubility, and water absorption were the same as previously described[15]. In addition, the microhardness, flexural strength, flexural modulus, and load energy of specimens after water immersion for 28 days were measured.

2.4. Contact angle measurement

Six light-cured specimens in each group were prepared using a mold (5 mm in diameter and 1.5 mm in thickness). The procedures and instruments for measuring the contact angle were the same as previously described[20].

2.5. Volumetric shrinkage measurement

A balance with an accuracy of ±0.1 mg coupled with a density determination kit (Densimeter SD-220L, Qualittest, Bridgewater Township, USA) was used to measure shrinkage according to Archimedes’ principle of buoyancy[21]. Briefly, six specimens in each group were weighed in air before polymerization to determine the uncured resin density ($D_1$). The cured resin density ($D_2$) was also measured after polymerization. All measurements were performed at room temperature (25 ± 1 °C). The shrinkage of the materials was calculated using the following equation:

$$\frac{D_2 - D_1}{D_2} \times 100\%$$

where $D_2$ is the cured density and $D_1$ is the uncured density.

2.6. MTT assay

An MTT assay was used to evaluate the cytotoxicity of the materials. Human gingival tissues were collected with the approval of the Research Ethics Committee of National Taiwan University Hospital (April 22, 2020; Case No. 202002055RIND. All participants provided informed consent. Human gingival fibroblasts (HGFs) with passage numbers of four to nine were used. Six light-cured specimens in each group were prepared, with diameters of 6 mm and thicknesses of 1 mm. The other procedures and instruments for the MTT assay were the same as described previously[15].

2.7. Statistical analysis

The SPSS software (SPSS 22.0, IBM, New York, USA) was used to perform a one-way analysis of variance for analyzing differences between values. The level of significance was set at 0.05 based on Tukey–Kramer multiple comparison tests.

3. Results

3.1. Characteristics of HBPUA

The HBPUA (THI-3) was synthesized via the reaction of isocyanates on IPDI with hydroxyl groups on TMP or HEA, thus forming urethane groups. The IR spectrum of the initial reactant mixture in Figure 3 shows intensity bands at 2267 and 3394 cm$^{-1}$, indicating the N=C=O and OH groups, respectively. The typical IR absorbance wavenumbers of the generated urethane linkage of the product increased at 1716 cm$^{-1}$ (C=O), 1241 cm$^{-1}$ (C-O), and 1535 cm$^{-1}$ (N-H). A smaller peak was observed at 2267 cm$^{-1}$ for the product compared with the reactant as a result of the consumption of isocyanate functional groups during polymerization. The conversion of isocyanate groups determined by the decreasing intensity of absorption at 2267 cm$^{-1}$ in the FTIR spectra was higher than 95%, and the refractive index (RI) signal intensities of monomers in the GPC of the products
almost completely disappeared after the reaction. This implied that only a few unreacted monomers remained in the products. The number- and weight-average molecular weights of the HBPUA determined using GPC were approximately 870 and 1480, respectively.

### 3.2. Microhardness, flexural strength, flexural modulus, and load energy

The microhardness of five groups is shown in Table 1. Before water immersion, Luxatemp exhibited the highest microhardness (23.41±1.51) followed by TIH3-60 (21.32±0.79), TIH3-30 (18.99±0.54), TIH3-0 (16.86±0.72), and TB-60 (15.68±0.68). After water immersion, the microhardness of all groups decreased, with TIH3-60 exhibiting the highest microhardness. However, no significant differences were found among the five groups.

Before water immersion, TIH3-60 and TB-60 exhibited significantly higher flexural strength than Luxatemp (Table 1). In addition, the flexural strength of TIH3-30 and TIH3-60 was significantly higher than that of TIH3-0. The flexural strength of the five groups also decreased after water immersion. The results of flexural modulus (Table 1) were similar to flexural strength in that TIH3-60 and TB-60 exhibited significantly higher flexural modulus than Luxatemp before water immersion. Moreover, TIH3-30 and TIH3-60 exhibited significantly higher flexural modulus than TIH3-0. After water immersion, the flexural modulus of the five groups did not exhibit prominent changes and no significant differences were found compared with the flexural strength before water immersion. TIH3-30, TIH3-60, TB-60, and Luxatemp exhibited similar load energy before water immersion and the load energy of all groups decreased after water immersion (Table 1).

### 3.3. Weight change, solubility, and water absorption

The weight change of Luxatemp was the lowest and the most stable among the five groups during 28 days of water immersion (Table 2). By contrast, TIH3-0 exhibited the highest weight change, and the weight change of TIH3-0, TIH3-30, TIH3-60, and TB-60 gradually increased as the duration of water immersion increased. About the results of solubility, no significant differences were found among the five groups (Table 2). The results of water absorption showed that TIH3-0 exhibited the highest water absorption, followed by TIH3-30, TB-60, TIH3-30, TIH3-60, and Luxatemp.
TB-60, and TIH3-60. The water absorption of Luxatemp was significantly lower than that of the other four groups.

3.4. Contact angle, volumetric shrinkage, and MTT assay

The contact angle results showed that Luxatemp was the most hydrophobic and TIH3-0 was the most hydrophilic (Fig. 4). The addition of HBPUA increased the contact angle and TIH3-60 was more hydrophobic than TIH3-30. TIH3-60 and Luxatemp exhibited the lowest volumetric shrinkage, followed by TIH3-30, TB-60, and TIH3-0 (Fig. 5). In addition, the shrinkage of TIH3-0 was significantly lower than the other 4 groups \((p < .05)\). The results of the MTT assay showed that the five experimental groups exhibited values similar to the control, indicating that none of the groups caused significant cytotoxicity (Fig. 6).

4. Discussion

According to the results of the present study, the null hypothesis was rejected. HBPUA addition affected the mechanical properties, water absorption, and volumetric shrinkage, causing significant difference among different groups.

Provisional prostheses are often used for several weeks or months and must withstand masticatory forces during chewing in the oral environment. Therefore, favorable mechanical properties such as hardness, flexural strength, flexural modulus, and load energy are required[5]. Hardness represents the resistance to permanent surface indentation or resistance to plastic deformation. Bis-GMA is used as the principal base monomer in most dental resins. However, the viscosity of Bis-GMA is high because intermolecular hydrogen bonds form at the hydroxyl groups of Bis-GMA[22]. Therefore, Bis-GMA requires a diluent monomer with low viscosity, such as TEGDMA, for homogeneous mixing[8]. In this study, TB-60 contained 60 wt% Bis-GMA and 40 wt% TEGDMA but the microhardness of TB-60 before water immersion was lower than that of TIH3-0, which consisted of only TEGDMA (Table 1). This may have been because Bis-GMA exhibited a lower degree of C=C bond conversion after polymerization compared with TEGDMA[23]. The addition of both 30 wt% and 60 wt% HBPUA to TEGDMA prominently increased the microhardness, and the microhardness of TIH3-60 was significantly higher than that of TIH3-30. Many dendritic units, \(b_3\), were observed in the HBPUA, as shown in Figure 1. Each dendritic unit contained three urethane groups, which were formed by one core monomer TMP (\(b_3\)) that reacted with three bifunctional monomers IPDI (\(a_2\)). Those dendritic units became effective nodes in the polymer network after the acrylic curing process. Moreover, hydrogen bonds can occur between urethane N-H and C=O groups. Therefore, a highly crosslinked polymer with a rigid structure could be obtained via the addition of synthesized HBPUA[24,25]. These characteristics are effective in enhancing microhardness and present HBPUA as a potential replacement for Bis-GMA.

The material traditionally used for provisional prostheses is PMMA resins, which is relatively inexpensive and easy to operate and polish. However, PMMA resins exhibit low wear resistance, high po-
lymerization shrinkage, and exothermic polymerization[26]. About
the composite based materials, Luxatemp is one of the products
which is used widely for fabrication of provisional prosthesis[6,27].
It was developed using Bis-GMA and inorganic fillers to overcome
the drawbacks of PMMA. Therefore, Luxatemp was considered as
the standard for comparison. The toughness, impact strength, wear
resistance, polymerization shrinkage, and exothermic reaction of
Luxatemp are superior to those of PMMA[28]. The microhardness
of Luxatemp was significantly higher than that of TIH3-60 (Table 1),
likely because no filler was incorporated into TIH3-60.

The flexural strength is the maximum stress measured in a
3-point bending test. The addition of HBPUA to TEGDMA significantly
increased the flexural strength and flexural modulus. In addition,
the flexural strength and flexural modulus of TIH3-60 before water
immersion was significantly higher than even those of Luxatemp
(Table 1). Although Luxatemp contains fillers, flexural strength is
related to the filler ratio and the shape and size of the fillers[29]. A pre-
vious study showed that the flexural strength of Luxatemp was low
because of larger interparticle spacing and wider filler size distribu-
tion than Tempsmart (GC Co., Tokyo, Japan) and Integrity MultiCure
(Dentsply Sirona)[30]. The larger interparticle spacing impedes the
pinning effect in crack propagation. Notably, the flexural strength
and flexural modulus of TB-60 approximated those of TIH3-60.
We observed that TB-60 endured larger deformation until fracture
during the 3-point bending test, indicating that TIH3-60 was more
brittle than TB-60. No significant differences were observed in the
load energy among TIH3-30, TIH3-60, TB-60, and Luxatemp (Table 1),
although the inclusion of filler particles in Luxatemp is generally
considered to produce a pinning effect in crack propagation and in-
creased fracture energy[31].

Water immersion for 28 days significantly reduced the micro-
hardness, flexural strength, and load energy of specimens in the five
groups. In addition, no significant differences were found in these
mechanical properties between TIH3-60 and Luxatemp. Contact an-
gle measurement showed that Luxatemp was the most hydrophobic
(Fig. 4) and therefore had the least weight change and water absorp-
tion (Table 2). Nevertheless, water entrance into a polymer is known
to cause hydrolytic degradation of fillers or the bond between filler
and matrix, as well as between filler and silane[32], contributing to the
deterioration of mechanical properties. By contrast, the main
chain of TEGDMA contains many hydrophilic and flexible ethylene
oxide groups, thus TIH3-0 had the highest weight change and water
absorption. Another possible reason is that it is less homogeneous
and the larger space between the polymer clusters in TIH3-0 accom-
modates more water absorption than TB-60[33].

Archimedes’ principle of buoyancy was used to measure volu-
metric shrinkage in this study. This method is relatively inexpensive
and is unaffected by specimen geometry and size[34]. The weight
of materials in air is measured before polymerization to determine
the uncured resin density and cured resin density after polymeriza-
tion. The resins in the four groups (TIH3-0, TIH3-30, TIH3-60, TB-60)
were used to fill a fixed-volume test container without photocuring
to measure the weight before polymerization. However, Luxatemp
is a self-curing composite and materials in two preloaded cartridges
are mixed through an auto mixing tip. When Luxatemp was injected
into the test container, polymerization began before weight mea-
surement in air, despite the weight being measured as soon as pos-
sible. The measured uncured resin density (D_u) was slightly higher
than D_c without any polymerization, causing underestimation of ac-
tual shrinkage. Therefore, TIH3-60 exhibited the same approximate
shrinkage as Luxatemp (Fig. 5) but the shrinkage of TIH3-60 might
be lower than that of Luxatemp. The number of moles of acrylate
groups per unit mass of TEGDMA, acrylate concentration [acrylate],
was approximately 0.007 mol/g (TEGDMA). In contrast to TEGDMA,
the acrylate concentration of HBPUA was only 0.0026 mol/g (HBPUA),
which resulted from the intended fraction of HEA monomers added
in the synthesis of HBPUA with a high molecular weight. For the blend
with higher HBPUA content, fewer linkages could be formed during
the acrylic curing process, and therefore the volumetric shrinkage of
the cured sample decreased with increasing amounts of HBPUA in
the blend, as shown in Figure 5.

In this study, 0.1 wt% EDMAB was used because higher concen-
tration of EDMAB did not significantly increase microhardness in our
pilot study. A previous study showed that the ratio of CQ and tertiary
amine affected the efficiency of free radical generation[35]. Exces-
sive amounts of tertiary amine may terminate polymerization by
trapping sinitiating radicals[36].

This is the first study to report the use of HBPUA polymer for
replacing Bis-GMA. Although TIH3-60 exhibits promising mechanici-
properties and low volumetric shrinkage, its clinical application
in provisional prosthesis requires further experiments and evalu-
ations. Limitations of this study included that the novel resin TIH3-60
did not contained inorganic fillers. Further investigation is required
to evaluate the use of inorganic fillers on the mechanical and physi-
ical properties of materials. In addition, clinical trials are required to
prove its clinical efficacy.

5. Conclusion

TIH3-60 containing 60 wt% HBPUA and 40 wt% TEGDMA ex-
hibited promising mechanical properties, including microhardness,
flexural strength, flexural modulus, and load energy. In addition, the
volumetric shrinkage of TIH3-60 approximated that of Luxatemp
and the biocompatibility of TIH3-60 approximated that of the con-
trol. These results demonstrate that TIH3-60 has potential as a new
provisional material.

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Conflicts of interest

The authors declare that there are no conflicts of interest.

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