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Original article

Effect of novel cycloaliphatic comonomer on the flexural and impact strength of heat-cure denture base resin

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Abstract: This study aims to evaluate the impact of adding a novel tricyclodecane dimethanol diacrylate comonomer on the flexural strength (FS) and impact strength (IS) of heat-cure denture base resin at 10% and 20% (v/v) concentrations. To test the FS and IS, a sum total of 150 bar-shaped specimens were prepared according to standard specifications. For the FS, the specimens were subjected to the three-point bend test with a span length of 50 mm. For the IS, Charpy's test was executed with a span length of 60 mm. The FS was measured in MPa, and the IS was measured in kJ/m^2. There was a statistically significant difference ($P < 0.001$) between the control and experimental groups in that both the FS and IS were increased in the experimental groups relative to the control group.

Keywords; cross-linker, cycloaliphatic monomer, flexural strength, impact strength, monomer

Introduction

Using dental prosthesis is indispensable in facilitating the functional and esthetic rehabilitation of edentulous patients. As such, denture prostheses should therefore simulate, as closely as possible, the natural structures or tissues that have been lost. The introduction of an acrylic denture base material occurred when Dr. Walter Wright in 1937 released his experimental results of methyl methacrylate (MMA) resin. Poly methyl methacrylate (PMMA) has numerous advantages, particularly its color, ease of processing, accurate adaptation, stability in the oral environment, good color stability, superior esthetics, and easy pairing with inexpensive equipment. However, it also has certain compromised mechanical properties [1]. Denture base resin is subjected to various stresses during function, including compressive, tensile, and shear stresses. The mechanical properties of denture base materials are a major concern because clinical surveys have revealed a higher incidence rate of in-service denture fracture [2]. Hence, considering the comparative mechanical properties of PMMA is important in selecting or developing proper denture base materials.

Denture fractures may appear following the occurrence of flexural fatigue with accompanying midline fracture. Flexural strength (FS) is a material property, defined as the maximum bending stress that can be applied to a material before it yields or fractures in a flexure test. Impact fracture, on the other hand, may result from accidental dropping of the dentures by their owners [3]. Impact strength (IS) can be defined as the capacity of a material to withstand a suddenly applied load or stress. These flexural fatigue or impact forces lead to the concentration of stress around micro-cracks formed within the denture base material because of the repeated submission of *in vivo* forces. Continuous force leads to crack propagation, which, in turn, reduces the strength of the denture base and finally results in a midline fracture [4].

The incidence rate of denture fractures is relatively high: within three years of denture fabrication, 68% of device fail, with partial dentures at even greater risk relative to complete dentures [5]. Researchers have also reported that 33% of denture repairs are made as a result of de-bonded denture teeth, while 29% of fractures occur in the denture base midline, observed more frequently in maxillary than mandibular dentures. The remaining 38% of fractures occur as a result of miscellaneous types of failures [6,7].

Various modifications have been suggested to overcome these issues, which can be summarized by the following two approaches: enhancing the polymer strength of dentures by adding a polyfunctional cross-linking monomer or reinforcing the denture base polymer with fibers, whiskers, metal wires, or metal nets [8]. In the past two decades, a dramatic increase in the use of fiber-reinforced denture materials has been seen, with different fibers such as glass, aramid, carbon, nylon, whiskers, or ultrahigh-modulus polyethylene fibers incorporated into the acrylic resin [9].

In recent years, monomeric modifications have been executed to improvise the properties of denture base resins. MMA monomer modification was conducted via the addition of fluoromonomers, phosphate monomers, methacrylic acid monomer, itaconate monomers, nitromonomers, or nonspecific monomers or solutions [10]. While these modifications have emphasized the need to improve the mechanical properties of denture polymers, most studies to date have not reported on the copolymerization of the modifiers with PMMA, since copolymerization dictates and determines the mechanical properties [10].

Cycloaliphatic monomers are a new class of monomers adopted into the field of restorative dentistry. These are also known as tricyclodecane (TCD)-based monomers. The existence of residual unpolymerized monomer can significantly affect the mechanical properties of denture base resins. The involvement of a TCD tri-ring central group imparts a steric hindrance effect that slows the rate of polymerization and facilitates monomeric conversion to polymer, thereby reducing the degree of residual unpolymerized monomer (C=C double bond) content in the final polymerized specimens. Also, TCD tri-ring monomers are classified under the category of cross-linking monomers. Tricyclodecane dimethanol diacrylate (TCDDMDA) is a cycloaliphatic monomer that appeared noncytotoxic to murine fibroblasts [11] when copolymerized with denture base resins [12]. However, its effects on FS and IS have not been yet documented in the dental literature. As such, this *in vitro* study aims to evaluate the effect of the addition of TCDDMDA comonomer on the FS and IS of heat-cure denture base resin at 10% and 20% (v/v) concentrations. The null hypothesis was that the addition of TCDDMDA comonomer would have no effect on both the FS and IS of the tested acrylic resin.

Materials and Methods

Heat-cure denture base acrylic resin (DPI Heat Cure; Dental Products of India, Mumbai, India) and the cycloaliphatic monomer TCDDMDA (Sigma-Aldrich, St. Louis, MO, USA) were chosen for this research (Table 1). The control (C) PMMA specimens for FS and IS experiments were prepared from 100% MMA monomer and the control groups were designated

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Table 1 Materials used in this study

Material	Manufacturer	Batch/lot number	Composition
Heat-cure denture base acrylic resin	DPI Heat Cure, Dental Products of India; Mumbai, India	Polymer: 1184 Monomer: 1186	Powder: includes prepolymerized beads of poly(methyl methacrylate), benzoyl peroxide as the initiator, dibutyl phthalate as plasticizer and pigments Monomer: includes methyl methacrylate, dibutyl phthalate, ethylene glycol dimethacrylate as the cross-linker, and hydroquinone as the inhibitor
Tricyclodecane dimethanol diacrylate (TCDDMDA)	Sigma-Aldrich, St. Louis, MO, USA	MKCG3853 CAS no. 42594-17-2	Monomer: bis(acryloyloxymethyl) tricyclo[5.2.1.$0^{2,6}$]decane Empirical formula: $C_{18}H_{24}O_4$ Molecular weight: 304.38 g/mol

Table 2 Specimen distribution

Flexural strength: (n = 75)	Impact strength: (n = 75)
Control groups: PMMA specimens with 100% MMA	
Group-FSC (n = 25)	Group-ISC (n = 25)
Experimental groups: PMMA specimens with 10% and 20% TCDDMDA	
Group-FS10 (n = 25)	Group-IS10 (n = 25)
Group-FS20 (n = 25)	Group-IS20 (n = 25)

Table 3 Comparison of median (IQR) flexural strength among groups

Group	Flexural strength (MPa)	Test value	*P*-value
FSC	36.0 (12.0)	66.826	0.000*
FS10	72.0 (12.0)		
FS20	108.0 (6.0)		

*Asymptotic significances are displayed. The significance level is 0.05.

Table 4 Comparison of the median (IQR) impact strength among groups

Group	Impact strength (kJ/m^2)	Test value	*P*-value
ISC	25.0 (12.5)	63.757	0.000*
IS10	50.0 (12.5)		
IS20	62.5 (12.5)		

*Asymptotic significances are displayed. The significance level is 0.05.

as FSC and ISC respectively. The experimental P(MMA-*Co*-TCDDMDA) copolymeric specimens for FS and IS experiments were prepared from a monomer mixture containing 10% (v/v) TCDDMDA comonomer in MMA monomer. The experimental groups of FS and IS experiments were designated as FS10 and IS10 respectively. Likewise, the experimental groups FS20 and IS20 consist of copolymeric specimens prepared from a monomer mixture containing 20% (v/v) TCDDMDA comonomer in MMA monomer. Table 2 describes the distribution of specimens in the groups for FS and IS. The pre-prepared monomers were stored in three individual identical dark glass containers and labeled with notes about the appropriate concentrations. Author blinding was accomplished by concealing the labels with opaque stickers with random numbers from one to three (M1, M2, and M3) to avoid an expectation bias.

Seventy-five PMMA specimens (65 × 10 × 2.5 mm^3) [13] for FS and seventy-five specimens (80 × 10 × 4 mm^3) [14-17] for IS were prepared out of dies, laser-cut from commercially available acrylic sheets. Dies were impressed into putty impression material (Photosil soft putty; DPI, Mumbai, India) within the dental flask to obtain a mold space. According to the manufacturer's instructions, the polymer was mixed with the monomer in 3:1 ratio and packed into mold space in the dough stage at a packing pressure of 3,500 psi via a mechanical press (Sirio Dental Srl, Meldola, Italy) for 10 min. The dough-forming time was 8 min for the control group and 15 min for the experimental groups. The heat-cure cycle included exposure at 74°C for 8 h, followed by terminal boiling treatment at 100°C for 1 h in an acrylizer (Unident Instruments Pvt. Ltd., New Delhi, India). After 30 min of bench-cooling and deflasking, the processed specimens were retrieved and finished with a tungsten carbide bur at 45,000 rpm, polished using pumice for 90 s at a rate of 2,800 rpm (Unident Instruments Pvt. Ltd.), and then underwent additional soft leather polishing using a wheel for 90 s at a rate of 6,500 rpm (Unident Instruments Pvt. Ltd.) [9]. All specimens were prepared by a single investigator. The final thickness of the specimens was reassured with the help of a digital Vernier caliper (Mitutoyo 500-197-20, Mitutoyo South Asia Pvt. Ltd., Bangalore, India). All specimens were stored in distilled water at 37ºC for 50 ± 2 h prior to testing [18].

FS was measured in the groups using a three-point bending test in a universal testing machine at a 5 mm/min cross-head speed. The FS in MPa was calculated using the following formula: FS (σ) = 3WL / 2 bd^2, where W is the maximum load before fracture, L is the span length of 50 mm, b is the specimen width, and d is the specimen thickness [19]. The IS of the groups were tested using Charpy's impact tester (Enkay Enterprises, New Delhi, India). The unnotched specimens [14,17,20] were supported 10 mm from the edges on each side and flatwise so that the impact span length was 60 mm [16]. The pendulum of the testing machine used in this research has an impact capacity of 164 J and a striking velocity of 5.6 m/s. Initially, the pendulum was dropped from a specific height to record the zero reading (zeroing). Then, the specimens were placed flatwise, and the pendulum was dropped again to impact the specimen. The energy absorbed by the material was obtained by the difference between these reading values (corrected reading) [20]. Charpy's IS was measured in kJ/m^2 by applying the following equation: IS = CR / A, where CR is the corrected reading in kJ and A is the test specimen area (breadth × thickness) in m^2.

Statistical analysis

All analysis was conducted using the Statistical Package for the Social Sciences (SPSS) version 20.0 software program (SPSS Inc., Chicago, IL, USA). Descriptive statistics were performed. Lilliefors significance correction by the Kolmogorov-Smirnov test of normality showed that there was a statistically significant difference apparent within the data distribution among each group of both FS and IS. Based on data distribution, continuous data were presented as median and quartiles. Separately, the nonparametric Kruskal-Wallis test was carried out to analyze differences in the mechanical properties between the control and test specimens. *Post hoc* multiple pairwise comparisons were conducted by Dunn's test (α = 0.05). A *P*-value of less than 0.05 was considered to indicate statistical significance.

Results

Tables 3 and 4 show the median (± interquartile range [IQR]) FS and IS of the compared groups, respectively. Group FS20 (108.0 ± 6 MPa) possessed a greater FS when compared with FS10 (72.0 ± 12 MPa). The FS was smallest in the control group FSC (36.0 ± 12 MPa). Group IS20 (62.5 ± 12.5 kJ/m^2) possessed a greater IS than that of IS10 (50.0 ± 12.5 kJ/m^2). The IS was smallest in the control group ISC (25.0 ± 12.5 kJ/m^2). There exists a statistically significant difference ($P < 0.001$) among the groups. Dunn's multiple comparison tests (Tables 5 and 6) revealed that a statistically significant difference existed between the groups of both FS and IS ($P < 0.05$). Greater FS and IS values were found among the experimental specimens when compared with the control specimens. Both the FS and IS were increased by the addition of TCDDMDA comonomer to MMA.

Discussion

Monomer modifications of denture base acrylic resins are not uncommon. Numerous researches have explored the use of monomer modifications to improvise the physicomechanical properties of denture base resins [10]. TCDDMDA is a novel cross-linking, difunctional, dual-reactive, cycloaliphatic acrylic monomer. In their report, Ajay et al. [11,12] suggested that TCDDMDA comonomer can easily copolymerize with PMMA and is noncytotoxic to the mouse fibroblast. However, there appear to be no studies that have assessed the influence of this novel monomer on FS and IS. In the present study, the addition of TCDDMDA comonomer to MMA increased both the FS and IS of denture base resin. Hence, the null hypoth-

Table 5 *Post hoc* Dunn's test of FS

Group comparison	Test statistic	Standard error	Standard test statistic	*P*-value
FSC-FS10	−25.000	6.116	−4.087	0.000**
FSC-FS20	−50.000	6.116	−8.175	0.000**
FS10-FS20	−25.000	6.116	−4.087	0.000**

**Asymptotic significances (two-sided tests) are displayed. The significance level is 0.05.

Table 6 *Post hoc* Dunn's test of IS

Group comparison	Test statistic	Standard error	Standard test statistic	*P*-value
ISC-IS10	−27.080	6.018	−4.500	0.000**
ISC-IS20	−47.920	6.018	−7.962	0.000**
IS10-IS20	−20.840	6.018	−3.463	0.001**

**Asymptotic significances (two-sided tests) are displayed. The significance level is 0.05.

esis was rejected.

The observed increase in FS can be attributed to reactive moieties. The acrylic moieties of diacrylates are generally more reactive than the methacrylic moieties of dimethacrylates and are thus relied upon when faster reaction kinetics are desired. In the context of dimethacrylate cross-linkers, after the first methacrylate group has reacted, the second pendant methacrylate group is estimated to be less reactive by 7.5- to 10-fold. The final conversion for copolymerization of MMA with ethylene glycol dimethacrylate (EGDMA) decreases together with the increasing content of this cross-linking agent. Such pendant dimethacrylate groups in control specimens acted as plasticizers at low deformation rates and were responsible for the lower FS than that found in the experimental groups. This plasticizing effect is due to the pendant methacrylate groups that separate the main chain segments [21].

TCDDMDA is a cycloaliphatic cross-linker possessing a tri-ring central group that imparts a steric hindrance effect that slows the rate of polymerization and facilitates the monomeric conversion to polymer, thereby reducing the residual monomer content. It is a dual-reactive monomer and has easily polymerizable carbon-carbon double bonds with highly reactive pendant acrylate group. The increased FS of the experimental groups (FS10 and FS20) can be attributed to low availability of the pendant acrylate groups after polymerization. Azevedo et al. [22] demonstrated an increased FS with MMA. Elsewhere, acrylamide monomer [23] and hydrophobic norbornyl/phenyl methacrylate [24] when added to MMA increased the FS of acrylic resin. However, several other experimental monomers adversely affected the FS of denture base resins. Fluoromonomers [25,26], though hydrophobic (water-shedding property), reduced the FS of denture base resins. Phosphate monomer [27] decreased the FS of acrylic resin, which was suggested to be the result of its hydrophilic nature; specifically, it caused increased water sorption, and the water acted as an inherent plasticizer. Nitro-monomers [19,28-30] also reduced the FS of acrylic resins.

In their investigation, Kawaguchi et al. [31] concluded that the addition of methacrylated dendrimer (DD1) as cross-linker in MMA did not improve the FS when compared with that of EGDMA. FS was high for both EGDMA and DD1 at lower concentrations. Arima et al. [32] concluded that the involvement of a cross-linking agent improved the mechanical properties of acrylic resin. However, a decrease in the mechanical properties was observed in correlation with very high concentrations of cross-linking agents. Caysik and Jagger [33] stated that an increase in the number of (CH_2-CH_2-O) units in cross-linkers promoted a greater level of flexibility in the materials that led to a decrease in FS. This reduction in FS was more marked with increasing concentrations of cross-linkers with long chains. However, this was not true in the case of TCDDMDA because it contains a central tri-ringed structure and no (CH_2-CH_2-O) units. Ruyter and Svendsen [21] suggested that differences in FS can be inferred by comparing the composition of monomer liquids. Monomers encompassing the cross-linking agent 1,4-butanediol dimethacrylate had a higher FS than that of EGDMA. Hence, from the above evidence, it is clear that the cross-linker's concentration, chain length, and type determine the FS of denture base resins.

With respect to IS, the addition of TCDDMDA comonomer to MMA increased the IS of denture base resin and was higher for IS10 and IS20 groups than the ISC group. This was in accordance with research performed by Al-Husayni and Hatoor [15], who substituted silver nitrate solution in MMA and concluded that silver nitrate at 60 ppm showed a higher IS than did the control. In contrast, the IS was decreased with the addition of dimethyl itaconate and di-n-butyl itaconate in MMA [16]. However, the addition of ethylene glycol methacrylate phosphate [34] and di-tetra hydrofurfuryl itaconate [35] in MMA did not adversely affect the IS of acrylic resin. As stated above, IS is also affected by the reactive moieties, concentration of the cross-linker, and type of the cross-linker used.

Notching the specimens is not uncommon and is normally performed in impact testing. However, the methods used to apply the notch can induce unique stresses and can be difficult, time-consuming, and nonreproducible to complete [36]. The currently used test specimens were unnotched because a better correlation was found between the IS and the energy absorbed in a flexural test with unnotched test specimens [37]. Al-Mulla et al. [38] compared the IS of notched and unnotched PMMA materials and observed the same ranking order, irrespective of specimen preparations. Hence, in the present *in vitro* study, unnotched specimens were tested for IS.

The present investigation was a triple-blinded study. The author, operator, and statistician were blinded by concealing the concentration of TCDDMDA comonomer substituted in MMA to avoid the creep of an expectation bias into the result. This study has limitations, and the results should be interpreted with caution. For example, although the specimens were immersed in water at 37°C to simulate the clinical situation, in adverse oral environment conditions, the materials are also submitted to thermal and loading stresses, and these aspects should be considered in future studies. This is the only research conducted concerning the FS and IS with TCDDMDA. Hence, the results must be carefully interpreted and extrapolated. Future investigations are necessary to evaluate the effect of TCDDMDA comonomer at a concentration of more than 20% v/v on the mechanical properties with mechanical loading and thermocycling.

In conclusion, within the limitations of this study, the addition of TCDDMDA comonomer to MMA increased both the FS and IS of heat-cure denture base resin. Meanwhile, more specifically, the addition of 20% TCDDMDA comonomer in MMA showed the highest FS and IS.

Conflict of interest

None.

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