ORIGINAL RESEARCH

Portraiture and Double Bond Conversion of a Monomethacrylate-based Oral Prosthetic Resin Substituted with a Novel Tri(azine-acrylate) Cross-linker

Vikraman Rakshagan[1], Ranganathan Ajay[2], Thillaigovindan Ranjani[3], Arul Queenalice[4], Sengottaiyan Vinothkumar[5], Paulpandian Saravanadinesh[6]

Abstract

Aim: To formulate, design, and chemically characterize a novel denture base resin (DBR) copolymer containing triazine-based antimicrobial comonomer and also to evaluate the double bond conversion (DC) in the copolymer with various concentrations of the comonomer by fourier transform infrared (FTIR) spectroscopy.

Materials and methods: The study groups comprise a control group G0 in which the specimens ($n = 10$) were polymerized without the triazine comonomer and trial groups G10 and G20 where the polymerized specimens ($n = 10$ each) contained 10 and 20% triazine comonomer, respectively. FTIR was employed to ascertain and evaluate copolymerization (CP) and DC. The obtained DC values were subjected to statistical analysis.

Results: A new denture base copolymer containing antimicrobial triazine comonomer was formed with ascertained copolymerization and higher DC than the control group. Twenty percent triazine comonomer in the copolymer exhibited the maximum DC.

Conclusion: Incorporation of the antimicrobial comonomer copolymerized with DBR to form a novel denture base copolymer exhibiting high DC.

Clinical significance: The novel denture base copolymer may prevent the microbial adhesion on the denture surface thereby preventing denture-induced stomatitis in the edentulous patients. Nonetheless, this novel copolymer may enhance the other necessary properties of the DBR and would ameliorate the living quality of the senile geriatric population with good *in vivo* serviceability.

Keywords: Antimicrobial, Comonomer, Conversion, Denture base, Triazine.

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Introduction

The lost teeth and the associated structures in completely/partially edentulous patients are often rehabilitated and restored with acrylic polymers. Poly(methyl methacrylate) [P(MMA)] is a widely employed polymeric material because of its palatable physico-mechanical and biological properties that satisfy the requirements to be an intraoral prosthetic material.[1] However, P(MMA) DBR get colonized by multifarious microbes that contribute to local/oral and systemic infections.[2,3] The surface porosities due to improper processing and surface roughness due to inadequate polishing are the two main reasons for the microbial adhesion on the denture surface that eventually lead to not only caries, malodor, and stomatitis[4,5] but also aspiration pneumonia, pulmonary candidosis, and infectious endocarditis.[6–9] Most geriatric hospitalized patients present with compromised denture hygiene due to their debilitating physical or mental health conditions.[10] Approximately, 11–67% of geriatric denture users are affected by chronic atrophic candidosis.[11–13] Nevertheless, the use of denture cleansers is not only futile in preventing denture micro-colonizers but also deteriorates dentures.[14] Antimicrobial resistance is a common problem encountered upon long-term use of systemic or local antimicrobial agents to prevent or eliminate the denture microcolonizers.[15–17]

These concerns suggest the need for a novel modified denture material, especially the ones with antimicrobial activity. The antimicrobial activity shall be integrated with DBR by employing biocidal agents videlicet silver nanoparticles, nano-dioxides of titanium/silicon, cationic bisguanides, and organic comonomers.[2,3,15,16,18–25] The organic comonomers include ethylene glycol methacrylate phosphate, methacryloyloxyundecyl

[1]Saveetha Dental College and Hospital, Saveetha Institute of Medical and Technical Sciences, Chennai, Tamil Nadu, India

[2]Department of Prosthodontics and Crown and Bridge, Vivekanandha Dental College for Women, Tiruchengode, Tamil Nadu, India

[3]Department of Prosthodontics and Crown and Bridge, Thai Moogambigai Dental College and Hospital, Chennai, Tamil Nadu, India

[4]Private Practitioner, Chennai, Tamil Nadu, India

[5]Department of Prosthodontics, Faculty of Dentistry, Tishk International University, Erbil, Iraq

[6]Department of Orthodontics, Saveetha Dental College and Hospital, Saveetha Institute of Medical and Technical Sciences, Chennai, Tamil Nadu, India

Corresponding Author: Ranganathan Ajay, Department of Prosthodontics and Crown and Bridge, Vivekanandha Dental College for Women, Tiruchengode, Tamil Nadu, India, Phone: +91 8754120490, e-mail: jrangclassiq@gmail.com

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pyridinium bromide, and dimethylamino ethyl methacrylate, which ultimately copolymerize with the base polymer. Fluorinated acrylate monomers were also copolymerized with the propriety denture base resins which elicited the conspicuous contact-kill phenomenon. 1,3,5-Triazine derivatives were investigated for their antimicrobial properties, terminating primarily Gram-positive bacteria through severing the membrane continuity.[26–28] 1,3,5-Triacryloylhexahydro-1,3,5-triazine (TATA) was used in the orthodontic adhesive which conspicuously decreased bacterial growth.[29] TATA is an antibacterial acrylate monomer containing three polymerizable aliphatic carbon-carbon double bonds (C=C). However, there is no research on copolymerizing TATA with P(MMA) DBR and its effects on the DC. Hence, the present research aimed to formulate, design, and chemically characterize a novel DBR copolymer containing TATA antimicrobial comonomer and also to evaluate the DC with the addition of TATA at different concentrations. The null hypothesis was that the TATA incorporation in the DBR does not influence DC upon evident copolymerization (CP).

Materials and Methods

Study Groups

The study groups were divided according to the TATA's concentration and composition (Table 1). The present *in vitro* research consists of three groups videlicet a control group G0 [P(MMA) powder + 2% dibenzoyl peroxide] and two trial groups G10 and G20 with TATA comonomer additionally at 10 and 20% concentration, respectively. The liquid composition was uniform for all the groups.

Preparation of the Powder and Liquid Monomer

The research was conducted in the Central Electrochemical Research Institute, Karaikudi. Poly(methyl methacrylate) [P(MMA)] powder (Molecular weight ~ 3,50,000), dibenzoyl peroxide (DBPO), methyl methacrylate (MMA) with ≤30 ppm mequinol as an inhibitor, *n*-butyl methacrylate (BMA), tricyclodecanedimethanol diacrylate (TCDDMDA), and 1,3,5-triacryloylhexahydro-1,3,5-triazine (TATA) were acquired from Sigma-Aldrich (Sigma-Aldrich Co. St. Louis, USA) and employed in the research without further purifications. TATA and DBPO were mixed in P(MMA) powder in a ball miller for an hour at 25 (±1)°C. The liquid was prepared by adding BMA and TCDDMDA in MMA contained in a conical flask with a magnetic stirrer and stirred for 30 minutes at 25 (±1)°C. The prepared powder and liquid were stored in three identical labeled plastic and dark glass bottles, respectively. Random letter stickers (A, B, C) were randomly stuck over the labels for blinding and to avoid experimental bias. These stickers were removed after the completion of the statistical analysis.

Preparation of the Specimens

The polymerized specimens of ≈200 µm thickness for chemical characterization and DC were prepared in the mold space obtained by the glass-slide technique (Fig. 1) as described by Rodriguez et al.[30] and thermo-polymerized for 8 hours at 74°C with terminal boiling at 100°C for 30 minutes in an acrylizer. The unpolymerized specimens were prepared without incorporating DBPO thereby eschewing premature polymerization reactions. Ten polymerized and unpolymerized specimens ($n = 10$ per group) were prepared.

Determination of Copolymerization and Double Bond Conversion

The CP and DC were determined by FTIR-attenuated total reflection (FTIR-ATR) spectroscopy (Tensor 27 model; Bruker Optik, GmbH, Germany) at transmittance and absorbance (Abs) mode, respectively. All the spectra were acquired from 64 scans and standardized at 4 cm^{-1} spectral resolution. In the IR spectra, two Abs peaks appeared corresponding to aliphatic C=C and carboxylic ester carbonyl (C=O) stretches. An appropriate baseline was drawn, and these two Abs peaks were separated into two Lorentzian curves. The intensities of absorption were determined by calculating the areas of the peaks.[31] The DC was calculated using the following equation:

$$DC\% = \left[1 - \frac{\left(\frac{\text{Abs C=C}}{\text{Abs C=O}}\right)\text{Polymerized}}{\left(\frac{\text{Abs C=C}}{\text{Abs C=O}}\right)\text{Unpolymerized}}\right] \times 100.$$

Statistical Analysis

The obtained data were subjected to statistical analysis and analyzed using SPSS software Version 21.0. The normality of the data was tested using the Kolmogorov-Smirnov test and found to be normally distributed ($p > 0.05$). Descriptive statistics were executed. The mean, standard deviation (SD), standard error, upper bound, and lower bound were calculated. One-way analysis of variance (ANOVA) and *post hoc* Bonferroni and Holm multiple comparison tests were used to compare the DC between the groups. $p < 0.05$ was considered for statistical significance.

Table 1: Groups and their compositions

		Composition	
Group		*Powder*	*Liquid*
Control	G0	Polymeric powder of P(MMA), 2% dibenzoyl peroxide as initiator.	MMA with ≤30 ppm mequinol as an inhibitor, 10% *n*-butyl methacrylate as a plasticizer, and 10% tricyclodecane dimethanol diacrylate as a cross-linker.
Trial	G10	Polymeric powder of P(MMA), 2% dibenzoyl peroxide as initiator, and 10% TATA.	
	G20	Polymeric powder of P(MMA), 2% dibenzoyl peroxide as initiator, and 20% TATA.	

The powder-liquid ratio was 3:1 and kept constant for all the groups

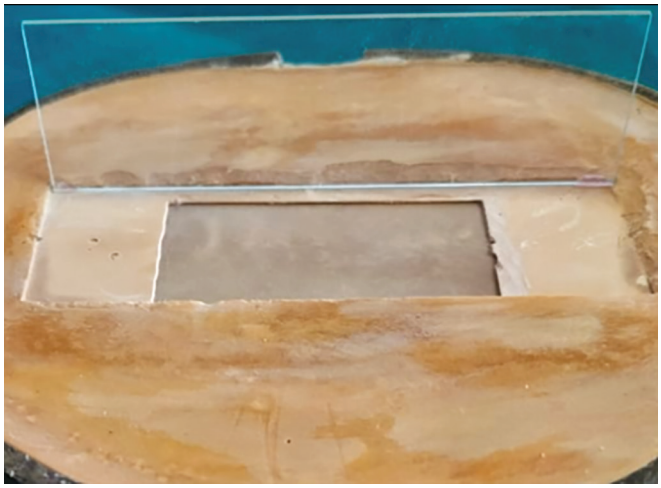

Fig. 1: Mold space for specimens obtained by the glass-slide technique

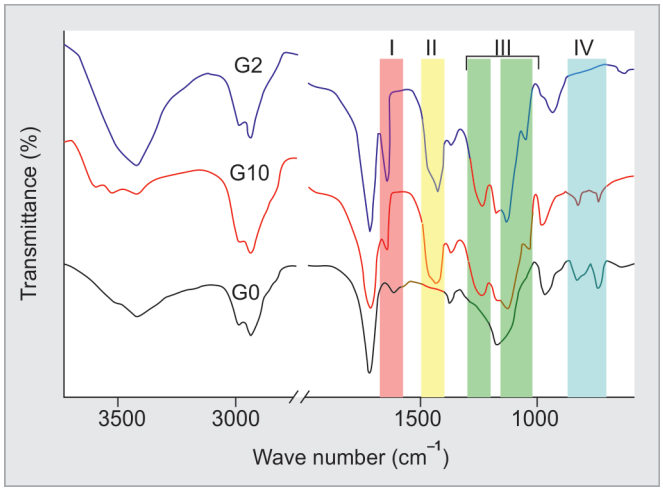

Fig. 2: Consolidated FTIR spectra with highlighted zones of spectral differences

Results

FTIR Spectroscopy and Copolymerization

The FTIR spectra of the trial groups G10 and G20 differed from the control group G0 in four distinct zones depicted in Figure 2. The peak corresponding to the aliphatic C=C stretch was evident in the G0 and was absent in the G10 and G20. The tertiary (3°) amide carbonyl stretch was spectacularly seen in the trial groups G10 and G20. This peak was dramatically absent in the G0 [Zone I]. The new peaks in G10 (1446.45 cm^{-1}) and G20 (1442.60 cm^{-1}) are ascribed to the triazine ring –CH_2– (C–H) stretch that was absent in the G0 [Zone II]. In Zone III, the 3° nitrogen C-N stretches related to >**(N-Ć)**=O and >**N-Ć**H_2 were observed in the G10 and G20 which was seldom seen in the G0. In Zone IV, the peak intensities corresponding to vinylidene (C=C) bend and *cis*-(C–H) wagging in the G0 diminished in the G10 and disappeared in the G20. The differences in the first three zones affirmed CP and novel denture copolymer P(MMA-*Co*-TATA) configuration. Table 2 tabulates the functional groups and their peak intensities of the groups.

Double Bond Conversion

The mean and standard deviations (SD) of the groups were tabulated in Table 3. A significant difference among the groups ($p = 0.000$) was observed. The DC of the G0 was lesser than the G10 and G20. Among the trial groups, G20 exhibited higher conversion than G10. Table 4 shows the multiple comparisons between the groups. All the comparisons exhibited statistically significant differences ($p = 0.000$). Figure 3 shows the IR graphs depicting the peak intensity differences between polymerized and unpolymerized specimens.

Discussion

Rendering the dentures with anti-microbial properties is of absolute necessity to prevent denture-induced stomatitis in the senile debilitating geriatric edentulous population. Several antimicrobial comonomers had experimented with the P(MMA) establishing their antimicrobial efficacy without ascertaining their CP with the P(MMA) and DC. Assessment of DC is important because it determines the polymerization quality and the polymer's properties. The distinct feature of this current research is that the TATA comonomer was substituted in the monomethacrylate-based denture base resin for the first time. In all the previous studies, TATA was substituted/added in the dimethacrylate-based orthodontic bonding agents and evaluated the antimicrobial property and bond strength without ascertaining the CP of TATA with other dimethacrylate resins videlicet *bis*-phenol-A dimethacrylate, triethylene glycol dimethacrylate, diurethane dimethacrylate, etc. However, in the present research, the CP and DC of the TATA comonomer with P(MMA) DBR were evaluated through FTIR technique as a primary step before evaluating the biomechanical properties.

Recently, monomeric modifications in DBRs are not uncommon.[32] In the present research, DBR was formulated by incorporating TATA comonomer at 10 and 20% concentrations. Three principle spectral differences were conspicuous among the groups in zones I, II, and III. The occurrence of new peaks designated to the 3° amide carbonyl stretch [>N-**(Ć=O)**], triazine ring –CH_2– (C–H) stretch, and 3° nitrogen C-N stretch [>(**N-Ć**)=O and >**N-Ć**H_2] only in the trial groups asseverated the CP and formation of a novel denture copolymer P(MMA-*Co*-TATA). The absence of these hallmark peaks in the G0 confirms the absence of TATA comonomer in the resultant polymer P(MMA). Stawski and Nowak also found a similar peak concerning 3° nitrogen in the FTIR spectrum for polydimethylaminoethyl methacrylate.[33]

In the present research, the resultant novel denture base P(MMA-*Co*-TATA) copolymer exhibited significantly higher DC than the control P(MMA) ($p < 0.05$). Therefore, the incorporation of triazine comonomer did not deteriorate the thermo-polymerization of the DBR. Hence, the null hypothesis was rejected. In a previous study based on orthodontic adhesive, the addition of a 5% triazine comonomer did not exhibit a significant difference in DC when compared to adhesive without the comonomer.[34] On the contrary, 20% TATA triazine comonomer increased the conversion significantly in the current research which is congruent with the previous studies where the addition of triazine comonomer improved the DC of the orthodontic adhesive.[29,35] Hence, from the above context, it is clear that high DC would result when the TATA comonomer's concentration is 10% and above.

Table 2: Functional groups and their peak intensities of the groups

	Peak intensity (cm^{-1})			
		Trial groups		
Functional groups	*G0 (control)*	*G10*	*G20*	*Zone*
CH_3 stretch (C–H)	2998.99	2997.06	2998.99	
Aliphatic chain $–CH_2–$ stretch (C–H)	2950.70	2952.70	2952.70	
Carboxylic ester carbonyl stretch [RO-(Ć=O)]	1731.89	1733.82	1733.82	
3° Amide carbonyl stretch [>N-(Ć=O)]	**Absent**	1660.53	1656.67	I
Aliphatic C=C stretch	1631.60	**Absent**	**Absent**	
Triazine ring $–CH_2–$ (C–H) stretch	**Absent**	1446.45	1442.60	II
CH_3 α-methyl bend (C-CH_3)	1390.53	1386.67	1384.74	
3° Nitrogen C-N stretch in >(N-Ć)=O	**Absent**	1253.59, 1143.66	1249.74, 1145.59	III
3° Nitrogen C-N stretch in >N-$ĆH_2$	**Absent**	1051.09	1062.66	
Aliphatic ether linkage I stretch (-C-O-C-)	1189.95	1188.02	1189.95	
Trans-(C–H) wagging	979.73	993.23	956.59	
Vinylidene (C=C) bend	840.87	842.80	**Absent**	IV
Cis-(C–H) wagging	754.08	752.15	**Absent**	

The bold "**absent**" signifies that the corresponding peaks are absent in the particular group

Table 3: One-way ANOVA of DC

Group	*Mean ± SD (%)*	*f-ratio*	*p value*
G0	77.87 ± 0.79		
G10	84.66 ± 1.16	262.55849	0.000
G20	86.67 ± 0.67		

Table 4: Bonferroni and Holm multiple comparisons of DC

Group (I)	*Group (J)*	*Mean difference (I-J)*	*t-statistic*	*Sig.*
G0	G10	−6.79*	16.8761	0.000
	G20	−8.80*	21.8632	0.000
G10	G20	−2.01*	4.9871	0.000

*The mean difference is significant at the 0.05 level

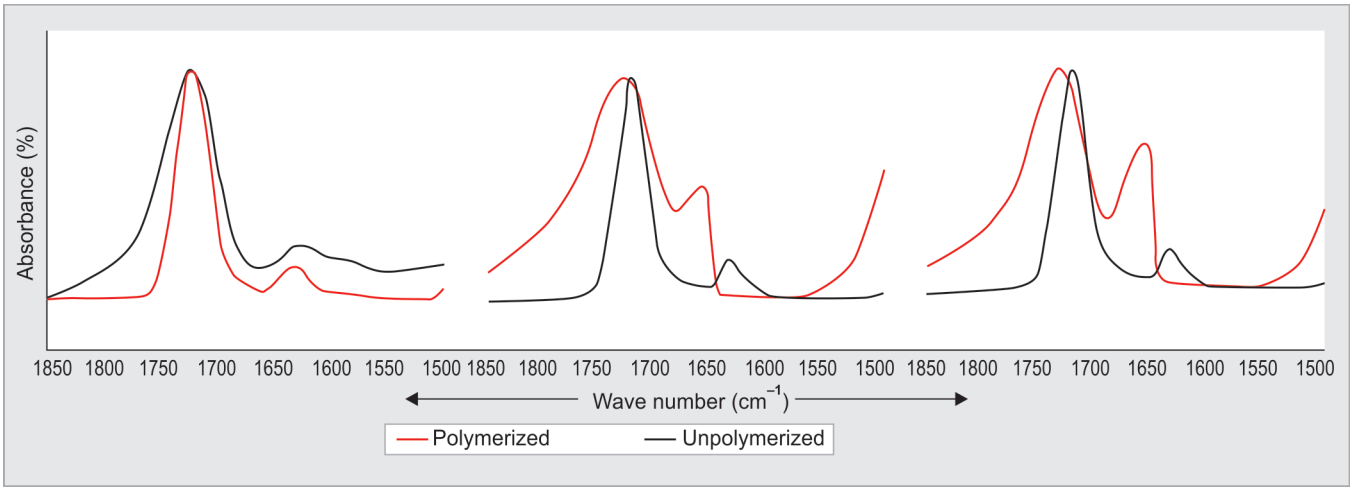

Fig. 3: IR graphs depicting the between polymerized and unpolymerized specimens

The DC of the orthodontic adhesives with TATA comonomer was reported between 57 and 71%. In the present research, the DC of DBR with TATA was 84 and 87%. This difference in the DC with the same triazine comonomer could be attributed to the reactivity ratios of dimethacrylate-based monomers in the former studies and monomethacrylate-based MMA in the latter with the

triazine comonomer. Cyclization of the dimethacrylates could yield a false high DC which is unlikely to happen with MMA-based DBR. TATA is a triacrylate monomer with three reactive terminals attached at the first, third, and fifth positions in its structure and its incorporation could enhance the C=C bond conversion.[29] Nevertheless, the increased DC in the current research can also be ascribed to the presence of acrylate moieties and their faster reaction kinetics, in contrast to the methacrylate moieties.[36] In a conventional DBR composition, ethylene glycol dimethacrylate as cross-linker and phthalates as a plasticizer were employed. However, in the present research, TCDDMDA was utilized as a cross-linker based on previous studies that focused on the prime importance of the reaction moieties causing high DC.[37,38] On the other hand, unlike phthalates, *n*-BMA participates in the polymerization reaction and would not leach from the denture during clinical service.[39]

The triazine structure yielded a highly branched and cross-linked copolymeric structure with orthodontic adhesives.[29,34,35] However, the cross-linking property of TATA in DBR is yet to determined. The DC is directly related to the mechanical properties,[40] inversely related to residual monomer content,[41] and polymeric deterioration.[42] Leaching out of the residual monomer causes allergic reactions.[40,43] All the above-mentioned parameters become theoretical assumptions when a novel comonomer is incorporated into a dental biomaterial. Hence, it is mandatory to ascertain each parameter through further research. TATA enhanced the DC in the trial groups containing 10 and 20% TATA comonomer. The copolymerizability of TATA comonomer with more than 20% concentration with DBR and its DC has not been determined yet. Assessment of the antimicrobial activity of the novel P(MMA-*Co*-TATA) copolymer is also open for future research. In this current research, no fillers were included in the resin matrix. Therefore, it is mandatory to incorporate the conventional inorganic fillers before analyzing the mechanical properties of the novel P(MMA-*Co*-TATA) copolymer. This is the lone-standing research executed with the triazine comonomer in DBR, and hence, the results obtained should be carefully interpreted.

Conclusion

Adhering to the research etiquettes and within the restraints, it is concluded that the incorporation of TATA comonomer copolymerized with P(MMA) to form a novel denture base copolymer P(MMA-*Co*-TATA) exhibiting higher DC than the control P(MMA). P(MMA-*Co*-TATA) with 20% TATA exhibited the highest DC.

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