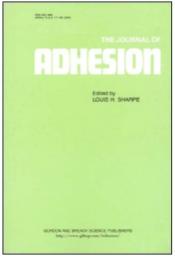
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# Tensile Bond Strength of a Highly Cross-Linked Denture Tooth to the Compression-Molded and Injection-Molded Denture Base Polymers

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## Tensile Bond Strength of a Highly Cross-Linked Denture Tooth to the Compression-Molded and Injection-Molded Denture Base Polymers

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This study compared tensile bond strengths between conventional compressionmolded heat (HC)-, auto (AP)-, and microwave-polymerized (MC) poly(methyl methacrylate)-based denture resins and a relatively new injection-molded, microwave-polymerized polyurethane based resin (MI) bonded to a highly cross-linked denture tooth. In the first part of the experiments, denture teeth were used as received. In the second part, they were treated with dichloromethane to see its effect on bonding of conventional denture bases (HCS and APS). Bond strength was tested in tension according to ADA specification No.15. The results showed that the HC group failed cohesively because of higher interface bonding (49.95 MPa) compared with those of the others (AP: 25.41 MPa; MC: 22.06 MPa; MI: 20.02 MPa). The application of dichloromethane improved bond strengths of HCS and APS groups (60.61 and 32.03 MPa, respectively). It was suggested that dichloromethane could be applied on the denture teeth ridge lap area prior to denture base processing to enhance adhesion between the tooth/resin.

**Keywords:** Cross-linked denture teeth; Denture base resins; Interpenetrating network (IPN); Tensile bond strength

#### INTRODUCTION

The loss of natural dentition is a general problem and the replacement of teeth by prostheses is of paramount importance in achieving the

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Address correspondence to Orhan Murat Dogan, Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas 58140, Turkey. E-mail: mdogan@ cumhuriyet.edu.tr masticatory function. The most preferred material for denture fabrication is poly (methyl methacrylate) (PMMA) resin polymer because of its good optical properties, excellent resistance to environmental degradation, and because it is one of the hardest thermoplastics available [1].

The artificial teeth become an integral part of the prosthesis and increase the stiffness and strength of the entire denture. The conventional composition of acrylic teeth is essentially cross-linked PMMA. However, co-monomer cross-linking is not evenly distrubuted in the denture tooth and the gingival ridge lap area (bonding surface of teeth) may not be as highly cross-linked as the incisal area of the tooth. The lowered cross-link density allows better chemical bonding between the acrylic tooth and the acrylic denture base [2]. However, acrylic teeth are prone to wear, delamination, and chipping of the incisal edges. In order to improve the wear resistance of acrylic teeth, the hardness and fracture toughness must be improved [1]. Recently, an interpenetrating polymer network (IPN) (Bioblend IPN<sup>®</sup>) was introduced as an abrasion-resistant tooth material (Dentsply International Inc., York, PA, USA). IPN teeth are produced by blending two or more existing polymers with compatible networks, which form permanent entanglements and owe their improved properties to micro-mechanical interlocking of the different network structures [3–6]. Although in most instances the bonding of artificial teeth to the denture base seems satisfactory, failures can be found in practice. It has been reported that fracture/debonding damage accounts for 20 to 33% of all denture repairs [7–11]. This is significant considering the total amount of time and money spent on denture teeth repairs.

It has been shown that in dentures subjected to bending deformation, tensile stresses are encountered with the area lingual to the most heavily stressed. Eventual failure at the tooth/denture base interface will occur when cracks originating from the high stress areas propagate. Such problems may result from heavy and uneven masticatory forces, unbalanced occlusion, and/or parafunctional habits where increased force is directed to prosthetic components [8].

Apart from intraoral conditions, debonding may be related to manipulative factors including faulty boil-out procedures which permit traces of wax to remain on ridge-laps of the teeth or to the careless application of tin-foil substitutes to the teeth, which can prevent chemical bonding [9,12–14]. Spratley [14] has reported that the single most common denture repair made in his clinics involved the replacement of detached acrylic resin teeth and it has been concluded that residual wax on the ridge lap surfaces appeared to be the chief cause of this type of failure.

A presumed advantage of acrylic denture teeth is their chemical bond to the denture base, yet dislodgements are still frequent,

especially with auto-polymerizing denture bases [9,15]. As an alternative to compression-molded, heat-, or auto-polymerizing resins, it has been proposed that microwave-polymerized resins exhibited good performance with clinically acceptable physical and mechanical properties and superior adaptation to master casts compared with these two conventional denture base resins [16–18]. One processing method that has been recently used for the microwaveable resins is a continuous-pressure-injection technique. With this technique, less polymerization shrinkage occurs and more accurate dentures are produced compared with those made by the compression-molding methods [19]. A polyurethane-based polymer, Microbase<sup>®</sup>, when used with the injection technique, is claimed by the manufacturer to have high biologic compatibility. Because its chemical structure is different from the other PMMA-based polymers, especially with respect to the degree of cross-linking, it might be expected to exhibit different mechanical properties. However, there exists no data related to bond strength of acrylic teeth/denture bases made by injection-molding and microwave polymerization.

Most attempts to improve the bond strength of denture teeth to an acrylic resin denture base have tried chemical treatment or mechanical modification of the ridge-lap portions of the denture teeth. The effect of mechanical preparation of teeth is well-documented and conflicting results with the removal of the denture tooth glaze and/or the placement of diatorics have been reported [9,12–14,20,21].

One example of chemical treatment include the application of methyl methacrylate monomer (MMA) [20,22]. On a theoretical basis, the polymerizable monomer plasticizes the surface of denture teeth and diffuses into the denture teeth acrylic resin upon polymerization, and an interwoven network of polymer chains that unites the denture base to the resin tooth is formed [5]. However, the lack of an effect of the polymerizable monomer on the bond strength has been partially attributed to the highly cross-linked denture teeth used in a previous study [20]. The denture resin has also been used with a nonpolymerizable solvent such as dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) or trichloromethane (chloroform, CHCl<sub>3</sub>) with the anticipation that the solvent would enhance the monomer diffusion and polymer network formation [5,23–25]. No distinct advantage of a monomer-chloroform mixture over plain monomer has been shown [23], whereas a significant improvement in bond strength with an equal amount of monomer and dichloromethane with or without the addition of 10% PMMA has been reported [26]. The non-polymerizable solvent may facilitate the swelling of the tooth polymer which, if cross-linked, would be slow with MMA alone, and thus MMA diffusion [23]. This presumably

might improve the extent and quality of the IPN when polymerization is completed.

The aim of this *in vitro* study is to compare tensile bond strength of a high-impact acrylic tooth bonded with a new injection-molded, microwave-polymerized, polyurethane-based polymer with three different compression molded, heat-, auto-, and microwave-polymerized PMMA-based polymers. In addition, the effect of treatment with dichloromethane of teeth bonding surfaces on the interface strength was investigated for assessing an adequate bond of conventional heat-cured and auto-polymerizing denture bases.

#### MATERIALS AND METHODS

#### Materials and Experimental Groups

The materials used in this study are shown in Table 1. Representative makes of Portrait<sup>®</sup> IPN cross-linked resin denture teeth (Dentsply Trubyte, York, PA, USA) were tested for tensile bonding with four types of denture base resins: An auto-polymerizing resin (Probase<sup>®</sup> Cold), a heat-cured resin (Probase<sup>®</sup> Hot), a microwave-polymerized, cross-linked resin (Acron<sup>®</sup> MC)], and an injection-molded and microwave-polymerized, polyurethane-based resin (Microbase). Denture tooth molds were chosen that had a >9.5 mm width buccal to lingual and mesial to distal when measured at the occlusal or cervical region. The tooth chosen was a mandibular first molar. A total of 60 teeth were used in the study.

Six experimental groups including an equal number of teeth (n = 10) were established. In order to make a comparison between different types of denture base resins and tooth bonding, in the first four groups the ridge lap surfaces of denture teeth were untreated (Group AP, for auto-polymerizing; Group HC, for heat-curing; Group MC, for microwave-polymerizing, compression-molded resins; and Group MI for microwave-curing, injection-molded resin). Two additional groups (n = 10), each of the auto-polymerized (Group APS) and heat-cured acrylic resins (Group HCS) were also tested to see the effect of chemical treatment of ridge lap surfaces on bonding.

#### **Preparation of Denture Teeth**

The teeth were ground axially so that they would appear cylindirical and have a maximum diameter of approximately 8.5 mm. The next series of steps involved the removal of the highly cross-linked occlusal portion, which is necessary to bond these surfaces to the top section of the denture base material, and to facilitate bond testing of the ridge-lap of the

		2		
Material (Groups)	Manufacturer	Chemical composition	Processing method	Polymerization mode
Probase Cold (AP/APS)	Ivoclar Vivadent AG, FL-9494, Schaan, Liechtenstein	Powder: PMMA, benzoyl peroxide, pigments, plasticiser Liquid: 90–95% MMA, <5% butandiole dimethacrylate, catalysts	Compression	40°C/2.2 bar under pressure 15 min
Probase Hot (HC/HCS)	Ivoclar Vivadent AG, FL-9494, Schaan, Liechtenstein	Powder: PMMA, benzoyl peroxide, pigments, plasticiser Liquid: 85–95% MMA, 5–10% EGDMA, catalysts	Compression	Water bath 73°C/ 90 min 100°C/ 30 min
Acron MC (MC)	GC-Dental Indust. Corp. Tokyo, Japan	Powder: PMMA almost 100%, barbituric acide derivative Liquid: MMA 95%, difunctional MMA 5%, QAC trace	Compression	Microwave oven 3 min at 500 W
Microbase (MI)	Dentsply DeTrey GmbH, Dreieich, Germany	Polyurethane 44%, polymer beads 54%, initiator 2%, silicone dioxide, glass powder, fillers	Injection	Microwave oven 7 min at 750 W
*According to PMIMA = poly chloride.	According to manufacturers' data sheet. ?MMA = poly(methyl methacrylate); MMA = loride.	*According to manufacturers' data sheet. PMMA = poly(methyl methacrylate); MMA = methyl methacrylate; EGDMA = ethylene glycol dimethacrylate; QAC = quartenary ammonium doride.	e glycol dimethacrylate; QAC	i=quartenary ammonium

**TABLE 1** Details of the Denture Base Materials and Polymerization Modes<sup>\*</sup>

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denture tooth. Thus, the occlusal surfaces were ground to form a flat surface greater than 2 mm in width perpendicular to the mesio-distal aspect and convex facio-lingually. Slightly curved ridge-lap surfaces of the teeth were untouched and not subjected to any grinding. The teeth were then scrubbed in a detergent solution, flushed with clean boiling water, and then allowed to cool and air dry for at least 30 min. In Groups AP, HC, MC, and MI, the ridge-lap portions of the teeth were used in as-received condition; the teeth in Group APS and HCS were also prepared with the same procedure as mentioned above, except for the application of dichloromethane solution (LabScan Ltd., Dublin, Ireland) onto the ridge-lap surfaces of these teeth with a brush for  $5 \, \text{s}$ . The denture teeth from the first part of the experiment did not undergo any surface treatment; thus, they served as internal control samples. After the preparation of teeth, they were directly inserted into the centre of a cylindirical shaft of uncured resin in a flask as described below. In order to evaluate the changes of surface topography of the denture teeth prior to and after the application of dichloromethane, the specimens were examined at  $\times 1000$  and  $\times 5000$  magnifications in a scanning electron microscope (SEM) (QUANTA 400, FEI Company, Eindhoven, Netherlands).

#### Preparation of the Molds

For tensile test specimens, a brass pattern was prepared in the manner described by ADA Specification no. 15, Section 4.3.7.1 [27], except that the specimens were obtained individually in final form. Diagrammatic representation of a specimen and its final form prepared for testing are given in Figs. 1 and 2.

Denture flasks were used for the preparation of the specimen molds. For the compression-molded method, Probase Hot and Probase Cold specimens were prepared in a conventional metal denture flask, the specimens of Acron MC in a fiber-reinforced plastic flask (FRP Flasks, GC America Inc., Alsip, IL, USA), and Microbase specimens in special fiber-reinforced flasks, with the injection unit (Dentsply/De Trey GmbH, Dreieich, Germany).

Type III dental stone (Moldano<sup>®</sup>, Bayer Dental, Leverkusen, Germany) was used to invest the specimen patterns. All of the internal surfaces of the flasks were lubricated with petroleum jelly. Both sides of the pattern were lightly lubricated. The lower part of the flask was filled with a mix of Type III dental stone. The pattern was placed into the unset stone with gentle pressure until one half of the pattern was submerged in stone.

When the stone was set, the surface was made smooth and coated with a thin layer of petroleum jelly. The upper part of the flask was

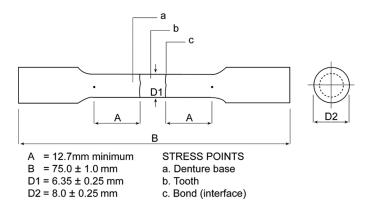


FIGURE 1 Diagrammatic representation of tensile test specimen.

put in place and filled with a vacuum-spatulated stone. The top plate was placed on the flask and sealed. When the stone was set, the two halves of the flask were separated and the pattern was removed, leaving an impression mold in which the tooth and denture base material could be placed. This procedure was followed for all molds.

All the stone surfaces were painted with one coat of an undiluted alginate separating medium (Separating Fluid, Ivoclar, Vivadent AG, Schaan, Liechtenstein). The flasks were conditioned for 1 h at  $23 \pm 2^{\circ}$ C before denture base materials were packaged. The base resins were mixed in a clean, dry jar using the recommended powder-to-liquid ratios defined as follows: 22.5 g to 10 mL for Probase Hot and 15 g to 10 mL for Acron MC and for Probase Cold resins. The acrylic resin was handled with latex examination gloves to

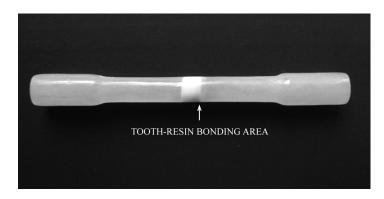


FIGURE 2 Completed test specimen.

avoid contamination of the resin with skin oils. Microbase resin was supplied in a single paste form in a plastic cartridge.

Each half of the mold in the flask was filled with denture base polymer dough and covered with a polyethylene sheet. The filled flask halves were clamped and then pressed together just short of full closure. The flask was then opened and the polyethylene sheets removed. A prepared tooth was pressed into the raw polymer dough in each specimen mold, half in the lower half of the flask so that it would be centered and the long axis would be parallel to the length of the acrylic mold. After packing, the flask halves were then clamped together and specimens of different base polymers were cured as follows:

For the compression-molded method, the assembled flask including Probase Hot was placed in a hydraulic press for 10 min, and the clamped assembly was immersed in a 73°C water bath for 90 min and then transferred to boiling water for 30 min. The polymerization of Probase Cold specimens was carried out in a pressure device (Ivomat<sup>®</sup>, Ivoclar Vivadent AG, Schaan, Liechtenstein) at 40°C and at 2.2 bar (770 kPa) pressure for 15 min. The specimens of Acron MC were microwave-irradiated for 3 min at 500 W in a microwave oven (Vestel Goldstar ER 535 MT, Manisa, Turkey).

For the injection-molded method, Microbase specimens were prepared maintaining a pressure of 550 kPa for 20 min during injection to allow complete outflow of the material into the molds. Polymerization was carried out in a microwave oven (Vestel Goldstar ER 535 MT) using the recommended curing mode of 7 min of irradiation at 750 W.

After curing and bench-cooling to room temperature, the central area of each specimen in which the tooth was placed was machined on a lathe to establish a gauge area of undefined length and of a diameter  $(6.35 \pm 0.25 \text{ mm})$  (Fig. 1) having the appearance shown in Fig. 2. Thus, 60 complete cylinders of tooth material and processed resin were produced. The specimens were then immersed for 1 d in distilled water at  $37 \pm 0.1^{\circ}$ C prior to testing.

Tensile testing was performed in a universal testing machine (Lloyd NK5, Lloyd Instruments Ltd., Fareham, Hampshire, UK). The specimens were inserted into the grips, and tested in tension with a crosshead speed of 0.254 mm/min. The specimens were randomly picked and subjected to the tensile force until failure and the value of the bond strength was registered in MPa. The tensile bond strength was then calculated by the software using the equation below:

T = F/A,

where  $T = (N/mm^2)$ , F = maximum recorded force at failure (N), and  $A = original cross sectional area (mm^2)$ . Data were collected using Nexygen<sup>®</sup> software (Nexygen, Lloyd Instruments Ltd.).

#### **Statistical Analysis**

Data obtained from the test were analyzed with one-way analysis of variance (ANOVA), using SPSS statistical software (SPSS version 11.0 software, SPSS Inc., Chicago, IL, USA). The mean values and standard deviations were recorded for each group, and the LSD post-hoc test was used to determine significant differences between groups (p = 0.05).

#### RESULTS

Tensile bond strength data associated with failure types are listed in Table 2. In the groups including different types of resins bonded to untreated teeth, the greatest mean bond strength value was recorded with the compression-molded, heat-cured resin, Probase Hot  $(49.45 \pm 5.37 \text{ MPa})$ , and the weakest value was for the injection-molded, microwave-polymerized resin, Microbase  $(20.02 \pm 2.26 \text{ MPa})$ .

A one-way ANOVA test demonstrated significant differences in the tensile bond strength between the four groups of specimens at a level of significance of p < 0.01 (Table 2). On further analysis by the LSD test, it was found that the heat-cured resin specimens had a significantly higher bond strength compared with those of the Probase Cold,

				Failure mode (n)	
Group	Minimum	Maximum	Mean (SD)	Adhesive	Cohesive
AP	19.40	33.80	$25.41 (4.56)^{a}$	10	-
HC	41.80	59.50	49.45 (5.37)	2	8
MC	18.20	24.20	$22.06 (1.89)^{a,b}$	10	-
MI	16.70	24.00	$20.02 (2.26)^{\rm b}$	10	_
APS	21.60	40.80	32.03 (6.32)	8	2
HCS	53.00	70.80	60.61 (6.06)	-	10

**TABLE 2** Tensile Bond Strength Values (MPa) and Failure Mode of theGroups Tested

n = 10 specimens per experimental condition.

Standard deviations are shown in parentheses.

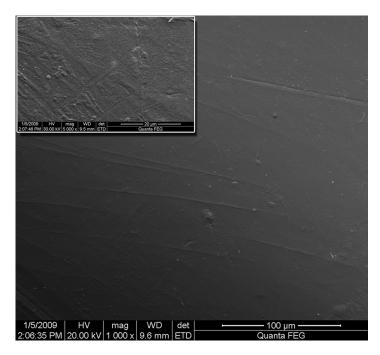
By the one-way ANOVA: F = 120.750, P = 0.000, p < 0.01.

Values with the same superscript letter are not statistically different at p > 0.05; others not superscripted differ significantly among themselves at p < 0.05 by LSD Test.

Acron MC, and Microbase resins, respectively (p < 0.01). Probase Cold specimens were also significantly different from Microbase specimens (p < 0.05). However, the bond strength of the specimens made of *Acron MC* was similar to those made of *Probase Cold* and *Microbase*, respectively (p > 0.05).

As far as the treated groups of teeth were concerned, that is for the heat- and cold-cured base resins, it was found that there was a significant difference between the tensile bond strengths. Results of mean comparisons using the LSD test showed that significantly higher strength was developed by bonding heat and cold resins to the surfaces that were previously treated with dichloromethane (p < 0.05). This treatment of teeth improved bond strength further to a mean value of  $32.03 \pm 6.32$  MPa in the auto-polymerized resin and  $60.61 \pm 6.06$  MPa in the heat-cured resin specimens thus, resulting in a significant increase in bond strength compared with the control ( $25.41 \pm 4.56$  and  $49.45 \pm 5.37$  MPa, respectively).

The modes of failure of all teeth are also depicted in Table 2. Most failures of the heat-cured material, whether the teeth were treated or



**FIGURE 3** SEM image of cross-linked denture tooth ridge lap surface before treatment. Scale marker of inset is  $2 \,\mu m$ .

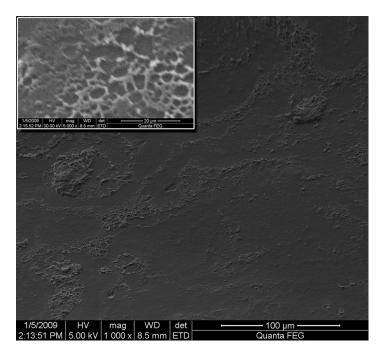


FIGURE 4 SEM image of cross-linked denture tooth ridge lap surface treated with dichloromethane. Scale marker of inset is  $20 \,\mu m$ .

not, were cohesive, occuring within the denture tooth resin close to the junction with the denture base resin. The site of bond fracture for Probase Cold, Acron MC, and Microbase resin systems demonstrated that adhesive bond failure occurred at the tooth/resin interface. Two specimens of Probase Cold, including teeth that had dichloromethane application, failed cohesively within the denture tooth.

SEM photomicrograph investigation of the ridge lap surface of a representative tooth prior to and after dichloromethane treatment are shown in Figs. 3 and 4, respectively. The non-treated group had a relatively smooth and glossy homogenous surface of the ridge lap portion (Fig. 3); dichloromethane treatment resulted in irregular texture and created some surface roughness with irregular distribution (Fig. 4). Dimensions of the pores and channels formed were a few micrometers and they appear to be interconnected. A representative SEM photomicrograph of the adhesively fractured surface of the AP group is given in Fig. 5. This was a typical MMA resin appearance, including no traces of tooth particles.

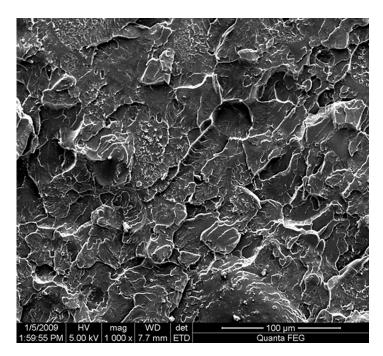


FIGURE 5 SEM image of the surface auto-polymerizing denture base resin that failed adhesively.

## DISCUSSION

The results of this study allow a comparison of the tensile bonding of a highly cross-linked tooth with the compression-molded heat-, auto-, and microwave-cured PMMA-based denture base polymers and the latest injection-molded and microwave-polymerized polyurethane-based material. For testing, the teeth were prepared according to ADA specification No. 15 [27] and inserted into the centre of the cylindirical shaft of uncured resin and then processed. The machining of the specimens, to establish the gauge area after processing, is considered to be indicative of the shortcomings of this type of testing, because cutting-induced stresses at the tooth/resin interface would have an adverse effect on the test results [28]. Moreover, during preparation of the teeth the ridge-lap surfaces were not changed. This was done on purpose because a circular cross-sectional area does not represent the true situation and, thus, it does not give the correct information about adhesion. The geometry of the curved ridge-lap surface of the teeth tested was highly complex; therefore, the stress distribution on the bond surface was also complex. Since the geometry of the bonding surface cannot be modelled simply by using flat surfaces, the true surfaces should be considered in experimental debonding tests.

When teeth were used as received, the highest tensile bond strength was obtained by heat-cured resin base material with a value of 49.45 MPa, which is higher than the acceptable bond strength limit value of 31 MPa, suggested by the ADA Spec. No 15. [27]. This finding is in agreement with the results of previous studies showing that the use of a heat-cured resin enhances the bond strength compared with autopolymerized [15,29-31] and microwave-polymerized [32] resins. It is presumed that the tensile bond strength value of heat-cured resin bonded to denture resin teeth reflects mainly complete polymerization. In other words, the lower tensile strength of other resins may be related to lower conversion in these materials than in heat-polymerized acrylic resin in a water bath. These data are confirmed by the difference in the mode of failure following the tensile test. When the fractured section surfaces were observed, failure had occured cohesively within the teeth bonded to the heat-cured resin while failure occured adhesively at the interfaces when using the others. The finding of cohesive failure within IPN teeth was in accordance with that of earlier studies testing the bonding of the same tooth material to heat-cured denture base resin [3,4,32].

As the cross-linking in a polymer increases, its bonding with the filler materials decreases. It could be likely that at high interface adhesion, failure can occur through IPN teeth where the bonding of the polymers to the filler material is expected to be low. The improvement of the strengthening of the matrix shifts the breaking surfaces from matrix to the more rigid filler materials, as in cement-sand concrete [33,34].

The achievement of a strong interfacial bond is possible by the diffusion and polymerization of MMA monomer across the tooth-denture base interface to form interpenetrating polymer networks. They may also be covalently interconnected by chain transfer reactions [35]. The important factors affecting such a bond are the rate at which MMA diffuses from the base resin mixture prior to curing and the time available for diffusion. The polymerization temperature has been reported to have an influence upon the MMA diffusion rate [23]. When using heat-cured resin, the time before polymerization commences is longer than with auto-polymerized materials; therefore, the potential for the penetration of the monomer into the tooth surface is greater. With auto-polymerized denture base materials, however, poor bonding to the teeth can result because of the shorter time of contact between teeth and base material before polymerization.

It has been stated that for bonding to occur, the monomer-polymer mix must either swell or dissolve the ridge-lap portions of the teeth [4,20,26]. In the dough stage, the acrylic monomers must be effective in swelling the surface region of the ridge-lap. This means that the more cross-linking agent used in processing the teeth, the less the swelling action by the available denture base monomers takes place. Thus, the degree of cross-linking and extent of copolymerization of the acrylic resin tooth and denture base are important for the strength of the bond achieved at the tooth/denture base interface; as the degree of crosslinking increases, the polymer swells less and bonding becomes more difficult [13]. The Acron MC, which consists of highly cross-linked polymers, has a higher solvent resistance (MMA) than the heat- and auto- polymerized resins which are composed primarily of linear polymers [36]. The different bond strength values of these base materials may be attributed to different chemical structures and also different temperature and time of polymerization protocols used. Because, during processing, Acron MC was in contact for significantly less time  $(i.e., 3 \min)$  with the microwave procedure than with the conventional procedure, it may be proposed that the less swelling of this product led to less interpenetration and, thus, reduced strength.

No previously published data related to tensile bond strength between IPN denture teeth with injection-molded microwave-polymerized denture base material were found. According to the manufacturer's information, this material consists of polyurethane, polymer beads, initiator, silicon dioxide, glass powder, and fillers. As for any dimethacrylate, polymerization of Microbase forms a highly cross-linked polymer matrix. In this study, among the tested base materials, the lowest tensile strength bond was observed by use of this material, with a value of 20.02 MPa. In an earlier study [19], it has been reported that this microwaveable injection-molded denture base material had inferior impact and flexural strengths compared with the conventional compressionmolded PMMA-based denture base polymers; however, in terms of rigidity, it has been found to be better than the conventional heatpolymerized and comparable with the microwave-polymerized PMMAbased polymer. The rigidity of polymers depends on the cohesion of chains and the rigidity is naturally expected to increase with the increase of cross-linking density as well as with the distribution of chain lengths between the cross-linking sites [37].

Pfeiffer and Rosenbauer [38] have compared the amount of water sorption of four denture base materials, including Microbase, after 7 d immersion in water. They have shown that the water sorption of this new material (3-mm thickness) was significantly higher than that of conventional heat-cured denture base (28.52 and 23.04 mg/ mm<sup>3</sup>, respectively). It is well known that depending on the thickness of the plastic specimens and the type of polymer, equilibrium of water sorption will be attained in 24 h [39] and that the sorption of water alters bond strength of denture base materials, which may lead to swelling or stress formation at interface [40,41]. In the current study, all specimens were kept in distilled water at 37°C for 1 d prior to testing. Although the effect of this parameter on the bond strength of the materials was not tested, based on data given by Pfeiffer and Rosenbauer [38], it could be proposed that the weak bonding of Microbase to teeth might be due to water action, at least to some extent. An advantage of using this new material is that it eliminates mixing and direct handling, as it is available in a cartridge in the form of a single paste. From the practical standpoint, if further improvement can be made to the present formulation to improve bond strengths, this material might have a great potential as a denture base.

The difference in failure type of the base materials could be correlated with their mean tensile strengths. As can be seen (Table 2), the groups with cohesive failure had higher tensile strength while others had a lower strength associated with brittle failure. Since the materials are different in composition it was difficult to make a decisive conclusion from the comparison of SEM pictures. Nevertheless, a representative picture of auto-polymerizing resin failing adhesively is given in Fig. 5 where there is no trace of tooth particles.

As with any bonding process, the bonding strength of two different materials depends on their ability to establish intimate contact and form some kind of physical (electrostatic bonding), chemical (atomic or molecular bonding), or mechanical (material interlocking) bond at their interface [25]. Theories of bonding between polymeric materials have been discussed and the conditions necessary to effect bonding have been described, including an interweaving by diffusion of two polymeric materials that are similar enough to be mutually soluble or compatible [26]. In this case, the teeth to be bonded insoluble acrylic denture base resin were composed of highly cross-linked, insoluble acrylic resin polymer with an added filler of colloidal silica to add strength. Adding of cross-linking improves the strength and abrasion resistance of teeth, but also may result in a decreased chemical bond as compared with acrylic resin denture teeth without cross-linking. This is likely due to the fact that the diffusion and polymerization of MMA monomer is more difficult.

Monomers emanating from the polymerizing denture resin penetrate the denture tooth resin and swell its surface. The swelling is a result of the reaction of the monomer with the polymer beads and the PMMA matrix [24]. It has been reported that the thickness of the swelled layer was related to the strength of the denture resin-denture tooth resin bond [42]. The benefit of dichloromethane in improving the bond between denture teeth and heat- and auto-polymerized denture base resins is evident from the results of this study. Compared with untreated denture teeth, dichloromethane treatment improved the bond strength of denture teeth especially for heat-cured resin specimens up to 60.61 MPa. All the specimens in this group failed in cohesive mode. On the other hand, although auto-cured specimens failed predominantly in adhesive mode after treating of teeth with dichloromethane, the tensile bonding reached a value of 32.03 MPa which is higher than the acceptable limit suggested by the ADA. The results are in good agreement with other studies in which the efficacy of dichloromethane application in improving the bond strength of resin denture teeth has been well-established [5,24,26].

It seems that this non-polymerizable solvent facilitates the swelling of the denture tooth polymer and, thereby, enhances the diffusion of polymerizable materials, notably MMA, from the denture base resin, resulting in the formation of a more extensive interwoven polymer network [24,26]. Surface roughness present on dichloromethanetreated denture teeth also suggests that micro-mechanical retention as another mechanism that might play a role in improving the bond strength (Fig. 3). Based on the data, treating of teeth with dichloromethane prior to denture resin processing could be proposed, because its small quantity became an effective way in terms of interfacial strength improvement for conventional resins used.

Although dichloromethane is quite toxic, its toxicity can be tolerated by the human body. The response of oral tissues to dentures processed with this technique has not been tested previously. However, any tissue response is most unlikely because of the low toxicity to ingestion indicated by the minimum lethal dose (in dogs, it is 3.000 mg/kg) and the low amount of  $\text{CH}_2\text{Cl}_2$  (less than 20 mg) in any set of complete upper and lower dentures. Moreover, its high evaporation rate would noticeably reduce the free  $\text{CH}_2\text{Cl}_2$  before the dentures were delivered to a patient. However, if large quantities are being used, adequate ventilation is desirable, because excessive inhalation may cause toxic responses such as cardiac arrhythmia, nausea, and vomiting [26]. Therefore, in commercial applications, it is better to remove it under vacuum.

The initial objective of this research was to test the bond strength of denture base materials to IPN teeth. However, considering that most of the failures occurring clinically are due to the relative fatigue life of the systems, further studies are required to investigate the fatigue resistance of such systems.

## CONCLUSIONS

Under the present experimental conditions, the following conclusions can be drawn:

- 1. There were significant differences in the bond strengths between denture base materials and a highly cross-linked denture tooth.
- 2. The highest bond strength values were obtained using the compression-molded and heat-polymerized denture base materials, whereas the lowest values were of the injection-molded and microwave-polymerized resin.
- 3. In general, the heat-polymerized groups failed cohesively within the denture tooth; other groups failed adhesively at the ridge lap areas of the denture tooth.
- 4. Treating teeth surfaces with dichloromethane significantly improved the bond strengths of auto- and heat-polymerized resins.

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