Original Article

Effect of Methyl Formate-methyl Acetate Treatment on the Tensile Bond Strength between Denture Teeth and Denture Base Resin

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Abstract

The aim of this study was to examine the tensile bond strength between two types of acrylic denture teeth and a heat-polymerized acrylic denture base after being treated with acrylic denture base liquid or methyl formate-methyl acetate (MF-MA) solutions. Conventional denture teeth and highly cross-linked denture teeth were polished at the ridge lap surface and then randomly divided into 12 groups. Groups 1 and 7 were control groups. Groups 2 and 8 were treated with acrylic denture base liquid (MMA) and the remaining groups were treated with MF-MA solutions at various concentrations (25:75, 40:60, 55:45, and 70:30 % v/v). Heat-polymerized acrylic resin was bonded to the denture teeth. Small dumbbell-shaped specimens were prepared and tensile bond strength testing was performed. The data was analyzed using two way ANOVA and one way ANOVA where significant differences in the groups were found, individual means were compared with the Tukey test at a 95 % confidence level. The surface treated groups demonstrated higher tensile bond strengths than the untreated groups ($p<0.05$), except for the 70:30 % v/v group. Within each surface treatment group, the type of denture teeth had no effect on bond strength ($p>0.05$). This study suggests the application of acrylic denture base liquid or a MF-MA solution (25:75, 40:60, or 55:45 v/v) before packing the acrylic resin can increase the bond strength between denture teeth and the denture base.

Keywords: Acrylic denture teeth, Acrylic denture base, Tensile bond strength, Surface treatment

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Introduction

Removable dentures are widely used by edentulous individuals. However, common removable denture problems include debonding of the denture teeth and denture fracture.[1] Denture tooth debonding may occur because of contamination of the tooth or denture surface, or differences in the structures of the two components.[2] There are two major methods for improving the bond strength between acrylic denture teeth and the denture base. Mechanical methods include grinding the tooth ridge lap surface[3], preparing a retention groove[4], or sandblasting.[5] Alternatively, chemical methods employ a chemical solution applied at the ridge lap surface. Previous studies have shown that MMA[6-8], MMA-based bonding agent[9], 4-META (4-methacryloxyethyl trimellitate anhydride)[10], dichloromethane (CH_2Cl_2)[11,12], methyl acetate (MA) based experimental bonding agent[13], or tribochemical silica coating and silanization[14] could increase bond strength. However, some studies indicated that MMA did not enhance the bond strength.[15-17]

The use of a chemical agent such as methyl formate (MF), MA or MF-MA solution has been suggested for repairing acrylic denture base resin. These agents reduced adhesive failure compared with the use of MMA.[18] A previous study showed that MF-MA solution enhanced the shear bond strength between reline resins and denture base resin.[19] A comparative study of the effect of these chemical agents on the bond strength between denture teeth and denture base has not yet been reported.

The objectives of the present study were to: 1) evaluate the effect of MMA and MF-MA solutions as surface treatments on the tensile bond strength between acrylic denture teeth and a heat-polymerized acrylic denture base, and 2) compare the bond strength between conventional denture teeth and highly cross-linked denture teeth. The first null hypothesis was that the tensile bond strength of the treated surface denture teeth and denture base resin were not significantly different from that of the untreated surface group. The second null hypothesis was that the tensile bond strength of the conventional acrylic denture teeth groups also were not significantly different from that of the highly cross-linked acrylic denture teeth groups.

Materials and methods

Sixty lower posterior first molar conventional acrylic denture teeth (Major Dent, Major Prodotti Dentari, Moncalieri, Italy) and sixty lower posterior first molar highly cross-linked acrylic denture teeth (Cosmo HXL, Dentsply Dental, Tianjin, China) were polished at the ridge lap surface using a polishing machine (Ecomet 250, Buehler, Illinois, USA) with 400, 800, and 1200-grit silicon carbide paper. Vaseline was applied to the internal surface of a tube (18 mm height, 15 mm diameter), and the tube was placed on a flat metal plate and filled with warmed modeling wax. The denture tooth was placed on surface of the wax, secured with the warmed modeling wax, and the tube/tooth specimen was immersed in cool water. After the wax hardened, the specimen was removed from the tube. Dental plaster was poured into the lower half of a flask. The specimen was placed in the plaster with the tooth and approximately 3 mm of the wax tube was exposed. Separating media was applied to the plaster surface. The upper part of flask was attached and filled with dental plaster. The flask was pressed (2,000 kgf) for 30 minutes. After the plaster set, the flask was placed in boiling water for 5 minutes. The flask was opened and the softened wax was removed by washing with boiling water and anionic detergent. Prior to denture base resin packing, the specimens were distributed into 12 groups (n=10 for each group). Groups 1 and 7 were control groups (no treatment). In the remaining groups, the ridge lap surfaces of the teeth were treated with a chemical agent for 15 seconds: groups 2 and 8 were treated with acrylic denture base liquid, the remaining groups were treated with a MF-MA solution at various

concentrations (25:75, 40:60, 55:45, 70:30 % v/v). The denture base resin (Meliodent, Heraeus Kulzer, Sanden, Germany) was bonded according to the manufacturers' recommendations within 5 minutes after surface treatment. The flasks were then pressed (2,000 kgf) for 1 hour. The specimens were polymerized at 74°C for 9 hours. Subsequently, the flasks were kept in a curing unit until the water reached room temperature.

Table 1 *Materials used in this study*

	Product name	Materials	Manufacturer
denture teeth	Major dent	Conventional polymethyl methacrylate (lot no.9072)	Major Prodotti Dentari, Italy
	Cosmo HXL	Highly crosslinked–IPN polymethyl methacrylate (lot no.20120525D)	Dentsply Dental ,Tianjin, China
denture base	Meliodent	Heat-polymerized acrylic resin (lot no.10NOV087)	Heraeus Kulzer, Sanden, Germany
chemical solutions	Meliodent (liquid)	Liquid of Heat- polymerized acrylic resin (mainly MMA) (lot no.10NOV087)	Heraeus Kulzer, Sanden, Germany
	Methyl formate	Methyl formate (lot no. s246689)	Merck Schuchardt OHG, Germany
	Methyl acetate	Methyl acetate (lot no.s6328911)	Merck Schuchardt OHG, Germany

After deflasking, the specimens (Fig. 1a) were sectioned longitudinally and perpendicular to the bonding interface to prepare 2 mm thick test specimens using a low speed cutting machine (Isomet 1000, Buehler, Illinois, USA) (Fig. 1b). Rectangular specimens (4X11 mm) were prepared with a carbide bur and placed in a metal jig (Fig. 1c). A steel fissure bur was used in a surveyor (Fig. 1d) to prepare the specimens to create small dumbbell-shaped specimens with a 2.5x2 mm bond area (Fig. 1e). The dumbbell shape was similar to the model of Nakabayashi *et al*[20], but smaller in size (Fig. 2).

The specimens were stored in distilled water at 37°C for 24 hours. The specimens were attached to a metal holder and were fixed with screws (Fig. 3). Tensile bond strength testing was performed using a testing machine (EZ-S, Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min. The tensile bond strength was calculated by dividing the failure force by the adhesion surface area.

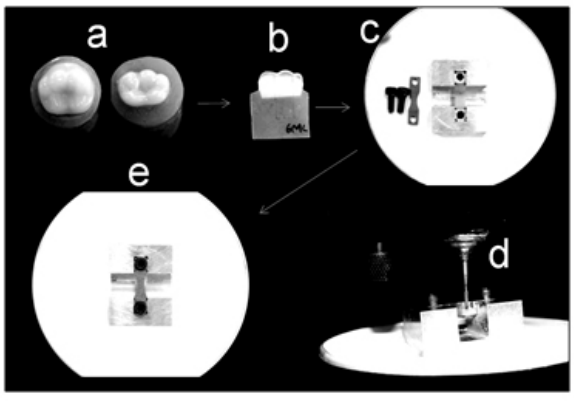

Figure 1 *Diagram of specimen preparation (a). the specimen after deflasking (b). 2 mm thick test specimens after cutting by low speed cutting machine (c). Rectangular specimen was placed in a metal jig for preparing dumb bell-shaped specimens (d).a steel fissure bur in a surveyor (e). dumbbell-shaped specimens*

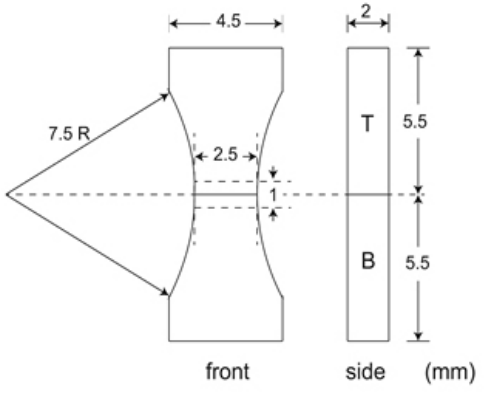

Figure 2 *Illustration of small dumbbell shaped specimen. T = denture teeth, B = denture base*

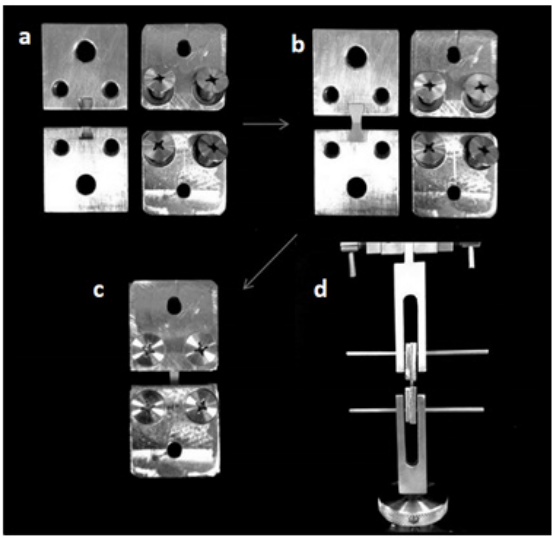

Figure 3 *Diagram of specimen fixation with metal holder and screws. (a) A metal holder and screws (b) A dumbbell-shaped specimen were placed in the metal holder (c) A dumbbell-shaped specimen were attached in the holder (d) A metal holder were attached with the universal testing machine*

To determine if the treated or untreated surfaces underwent any morphological changes, several denture teeth from each group were sputter-coated with gold and observed under a scanning electron microscope (JEOL-5410, JEOL Inc., Tokyo, Japan) at 15 kV. The fractured surfaces were examined using a stereo microscope (ML9300, Meiji Techno, Saitama, Japan) at 15X magnification and scanning electron microscopy (JEOL-5410, JEOL Inc., Tokyo, Japan) to determine the mode of failure. In the present study, the fractured surface was categorized as mixed primarily adhesive when the majority of the fracture was at the interface. If the majority of fracture occurred in the denture base, the specimen was categorized as having mixed primarily cohesive in denture base type failure. If a fracture occurred entirely in the denture base, the specimen was categorized as having cohesive failure of the denture base. There were no pure adhesive fractures because the SEM micrographs showed that in every fractured specimen, small fragments of denture base could be seen in the denture teeth.

The data were statistically analyzed using SPSS for Windows 17.0 (SPSS Inc., Chicago, IL, USA). The results were tested to determine the normality of distribution with the One-sample Kolmogorov-Smirnov test and the homogeneity of variance using the Levene's test. The data were normally distributed ($p>0.05$) and presented homogeneous variances ($p>0.05$), which indicated that a parametric analysis should be performed. The means and standard deviations for the tensile bond strength were calculated and statistically analyzed using two-way ANOVA and one-way ANOVA post hoc Tukey ($\alpha=0.05$). The modes of failure were analyzed using the Chi-square test.

Results

The results showed that surface treatment affected the bond strength between the denture teeth and the denture base. The surface treated groups demonstrated higher tensile bond strengths than the untreated groups ($p<0.05$), except for the 70:30 % v/v group (Table 2). Overall, the Major dent teeth groups exhibited significantly higher bond strength compared to the Cosmo HXL groups ($p<0.05$). Within each surface treatment group, there was no significant difference between the bond strength of conventional denture teeth and highly cross-linked denture teeth ($p>0.05$)

The mode of failure analysis (Table 3) revealed that the mode of failure was independent of the type of denture teeth and surface treatment ($p>0.05$). The negative control groups exhibited 100 % mixed, primarily adhesive failures. Chemical treated denture teeth with MMA or MF-MA resulted in an approximately 20 % decrease in the adhesive fracture (approximately 13 % mixed, primarily cohesive in denture base failure, and 8 % cohesive failure in denture base). Notably, Cosmo

HXL teeth treated with MF-MA at ratios of 40/60 or 70/30 exhibited a 40 % decrease in mixed, primarily adhesive failures (3/1 mixed, primarily cohesive in denture base failure/cohesive failure in denture base).

Table 2 *Mean and standard deviation (SD) of the tensile bond strength between denture teeth and denture base*

Chemical surface treatment	Denture teeth	
	Major dent	Cosmo HXL
Control	41.18 ± 6.12 [c, d]	37.51 ± 5.84 [d]
monomer (MMA)	56.10 ± 6.69 [a]	51.78 ± 6.39 [a, b]
MF 25 MA 75	56.66 ± 5.49 [a]	52.96 ± 7.51 [a, b]
MF 40 MA 60	53.50 ± 2.43 [a]	49.30 ± 6.46 [a, b, c]
MF 55 MA 45	55.79 ± 3.79 [a]	50.68 ± 6.87 [a,b]
MF 70 MA 30	49.25 ± 6.48 [a, b, c]	44.39 ± 4.62 [b, c, d]

MF = methyl formate, MA = methyl acetate, MMA = methyl methacrylate

**There was no significant difference (P>0.05) between groups denoted by the same letter.*

Table 3 *Mode of failure (n = 10 in each group).*

Chemical surface treatment	Denture teeth					
	Major dent			Cosmo HXL		
	mixed primarily adhesive	mixed primarily cohesive in denture base	cohesive in denture base	mixed primarily adhesive	mixed primarily cohesive in denture base	cohesive in denture base
Control	10	0	0	10	0	0
monomer (MMA)	9	0	1	9	0	1
MF 25 MA 75	8	1	1	9	1	0
MF 40 MA 60	7	1	2	6	3	1
MF 55 MA 45	8	1	1	8	2	0
MF 70 MA 30	9	1	0	6	3	1
Total	**51**	**4**	**5**	**48**	**9**	**3**

The SEM images of the untreated denture teeth showed a homogeneous surface with irregularities from grinding (Fig.4A, 5A). Treatment with acrylic denture base liquid created a blended and smoother surface (Fig.4B, 5B). Treatment with MF-MA solutions created surface pores (Fig.4C-F, 5C-F).

The top panel of Figure 6 shows an example of a mixed primarily cohesive in denture base failure specimen. The lower left panel shows the fracture at the interface area with a small fragment of the denture base attached to the denture tooth. The lower right panel shows the fracture in the denture base.

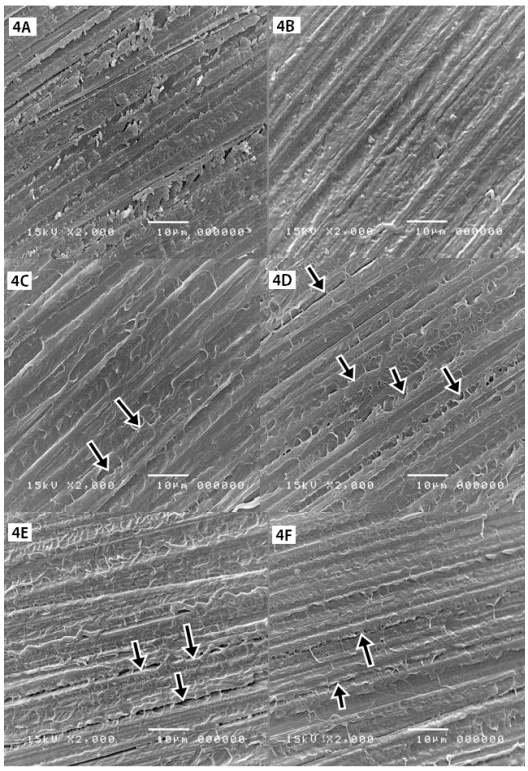

Figure 4 *SEM micrographs of the morphology of the untreated and treated surfaces of the Major dent denture teeth at 2000x magnificent level. (A) untreated (B) treated with MMA for 15 seconds. (C) treated with MF-MA solution (25:75 v/v). (D) treated with MF-MA solution (40:60 v/v). (E) treated with MF-MA solution (55:45 v/v). (F) treated with MF-MA solution (70:30 v/v). Black arrows indicate the pores on treated teeth surface*

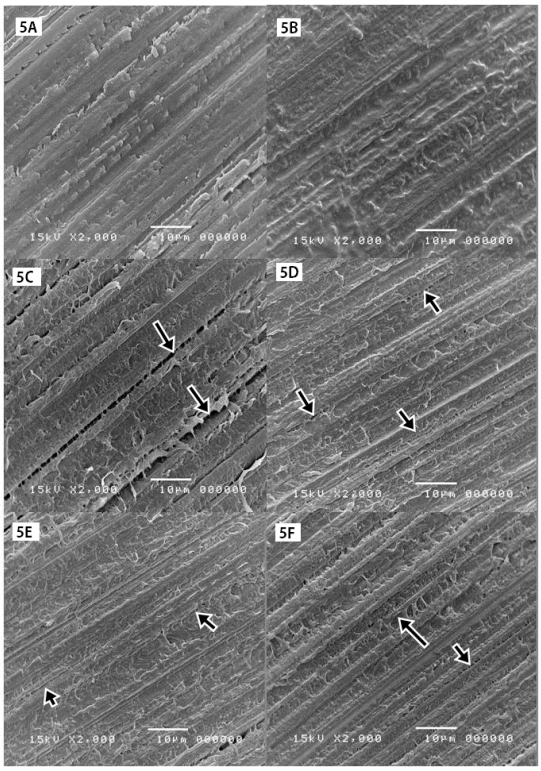

Figure 5 *SEM micrographs of the morphology of the untreated and treated surfaces of the Cosmo HXL denture teeth at 2000x magnificent level. (A) untreated (B) treated with MMA for 15 seconds (C) treated with MF-MA solution (25:75 v/v) (D) treated with MF-MA solution (40:60 v/v) (E) treated with MF-MA solution (55:45 v/v) (F) treated with MF-MA solution (70:30 v/v). Black arrows indicate the pores on treated teeth surface.*

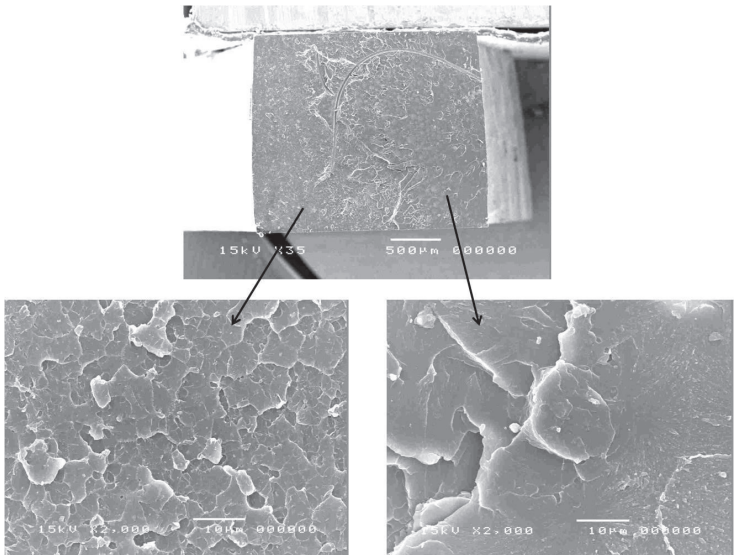

Figure 6 *SEM micrographs of a denture tooth after fracture at 35x magnificent level. The left arrow shows the fracture at the interface at 2000x magnificent level, the right arrow shows the fracture in denture base at 2000x magnificent level. This specimen was categorized as mixed primarily cohesive in denture base.*

Discussion

There are wide variations in the testing methods used for determining the bond strength between denture teeth and denture base.[21] ADA No.15[22] uses a tensile test but does not specify the bond area, while ISO 22112:2005[23] is only concerned with the mode of fracture. The present study used a tensile test and dumbbell shaped specimens according to the standard test for plastics (JIS K-691124, ISO527-125). The advantage of this design is that the tensile load was applied directly to the smallest part of the specimen, which was at the tooth-denture base interface.[20] A cross-section of 2.5x2.0 mm was prepared to maintain the correct dumbbell shape proportions based on the size of the denture teeth.

Based on these results, the first null hypothesis of surface treatment was rejected. The chemical surface treatments, used in this study, enhanced the bond strength between acrylic denture teeth and denture base. The swelling phenomenon occurs in acrylic denture teeth[26] when the monomer from the denture base polymer diffuses into the acrylic resin teeth during the packing process. In the present study, the increase in bond strength may have been caused by the chemical solution dissolving and swelling the denture tooth surface. This facilitates the diffusion of monomer from the denture base resin to form an interpenetrating polymer network with the denture teeth.

SEM images indicated that the denture teeth underwent morphological changes when treated with the chemical solutions. MMA created a blended and smoother surface on the denture teeth, while the MF-MA solutions created surface pores. These pores created space for the denture base resin and may have improved the bond strength due to micromechanical retention. However, there were no significant differences between the bond strengths of the MMA group and the MF-MA solution groups. This may result from the effect of the relatively high polymerization temperature, which can allow for greater monomer penetration.

Based on these results, the second null hypothesis of type of denture teeth was accepted. Conventional denture teeth demonstrated higher bond strength than the highly cross-linked denture teeth. However, there were no significant differences when compared by the same surface treatment. These results were confirmed by previous studies.[10,11] Conventional denture teeth have more unlinked polymer chains to develop an interwoven polymer network between the denture teeth and the denture base compared with the highly cross-linked denture teeth.[11] Differential scanning calorimetry has demonstrated that the glass transition temperature (T_g) of Major dent (115.1°C) is close to that of Cosmo HXL (117.8°C). The rate of monomer diffusion from the denture base resin is dependent on T_g.[27] This might be the reason why there was no significant difference in bond strength between conventional denture teeth and highly cross-linked denture teeth based on the surface treatment type. However, two-way ANOVA analysis indicated that the type of denture teeth had an effect on bond strength when comparing groups 1-6 with groups 7-12.

Vallitu[28] stated that an important factor for bond strength is that the bonding surface of the acrylic resin tooth must be adequately dissolved. In the present study, the period used for chemical agent exposure was 15 seconds. This application time was selected because it is practical for laboratory processing. The results of the chemical treated groups (except the 70:30 % v/v group) revealed that the 15 sec dissolving period was effective when compared with the control group.

According to the softening theory, a liquid will act as a plasticizer of a polymeric solid when the solubility parameters and polarities between the liquid and the polymeric solid are close to each other.[29] MMA, MA, and MF have solubility parameters of 18, 19.6, and 20.9 $MPa^{1/2}$ respectively.[30] These values approximate the solubility parameter of poly (methyl methacrylate) (18.3

MPa$^{1/2}$), which is the major component in acrylic denture teeth.

The bond strength of the 70:30 % v/v group was not significantly different from that of the untreated group. This can be attributed to the solubility parameters. The 70:30 % v/v group primarily contained MF that has a solubility parameter farther from that of poly (methyl methacrylate) compared with MA. Therefore, less dissolution and swelling of denture teeth surface likely occurred for the 70:30 % v/v group.

Table 4 show the toxicity and hazards of chemical solutions. NFPA (National Fire Protection Association) uses a standard called "NFPA 704" or "fire diamond" as a chemical hazard label.[31] There are four colored sections on the diamond. Number 0-4 are labelled in each section to indicate the level of hazard. On this scale, 0 indicates "no hazard" while 4 means "severe hazard". The blue section indicates health risk. The red section indicates flammability. Yellow indicates reactivity or explosivity. The white is section is used to describe any special hazards. The fire diamond indicated that the health risk of MMA[32] and MF[33] are level 2 which is greater than MA (level 1).[34] Level 2 means "Intense or continued non-chronic exposure may result in incapacitation or residual injury". Level 1 means "exposure may cause irritation and minor residual injury". Data from New Jersey Department of Health and Senior Services[35-37] indicated that the odor threshold of MF (2000 ppm) and MA (180 ppm) is more than MMA (0.049 ppm). In addition, data from OSHA, NIOSH, ACGIH indicated that the workplace airborne exposure limits of MMA is less or equal to MF and MA. These suggest that MF and MA are safer than MMA and can serve as a substitute to MMA as a surface treatment for denture teeth before packing acrylic resin in the dental laboratory.

Further research using a larger sample size, conditions similar to those found in the oral cavity, and a greater variation in application times is required to confirm the effect of MMA and MF-MA solution on the bond strength between acrylic denture teeth and a heated-polymerized acrylic denture base.

Table 4 *Toxicity and hazard of chemical solutions.*[32-37]

Materials	NFPA 704 or the Fire Diamond	Workplace exposure limit			
		Odor threshold	OSHA (8 hours)	NIOSH (10 hours)	ACGIH (8 hours)
Methyl methacrylate		0.049 ppm	100 ppm	100 ppm	50 ppm
Methyl formate		2000 ppm	100 ppm	100 ppm	100 ppm
Methyl acetate		180 ppm	200 ppm	200 ppm	200 ppm

ppm = parts per million *NIOSH = National Institute for Occupational Safety and Health*
OSHA = Occupational Safety and Health Administration *ACGIH = American Conference of Governmental Industrial Hygienists*

Conclusions

Within the limitations of the present study, the following conclusions can be drawn:

(1) The application of acrylic denture base liquid or a MF-MA solution (25:75, 40:60, 55:45 v/v) for 15 sec. to the base of denture teeth can increase the bond strength of acrylic denture teeth to the denture base.

(2) When comparing all the groups, the conventional denture teeth exhibited higher tensile bond strength than the highly cross-linked denture teeth. When comparing each surface treatment, the type of denture teeth had no effect on bond strength.

Acknowledgements

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