Effect of Adhesive Primer Developed Exclusively for Heat-curing Resin on Adhesive Strength between Plastic Artificial Tooth and Acrylic Denture Base Resin

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Despite progress in the development of denture base resin and artificial tooth materials, dental clinics are still plagued with artificial teeth falling off the denture base – due to poor bond strength – after denture delivery. Against this background, this study sought to examine the effect and durability of an adhesive primer developed exclusively for heat-curing resin on the adhesive strength of heat-curing denture base acrylic resin to plastic artificial tooth. Test specimens were divided into four groups according to the treatment method of the artificial tooth's test bonding surface: air abrasion, adhesive primer application, adhesive primer application after air abrasion, and pretreatment only (control). After heat curing of acrylic resin onto the bonding surface, shear test was performed for two storage periods: 24-hour versus 100-day water storage. From the results obtained, it was revealed that the evaluated adhesive primer was significantly effective in increasing adhesive strength between artificial tooth and acrylic resin, although specimens were stored in water for 100 days.

Key words: Adhesive primer, Adhesion, Artificial tooth

INTRODUCTION

Denture fracture is one of the usual problems encountered after denture delivery. This problem is often related to the material properties of the acrylic denture base resin used^{1–4}. Due to poor bond strength, artificial teeth falling off the denture base is another usual problem encountered after denture delivery to patients^{5,6}. Therefore, in the fabrication of removable dentures, adhesive strength between denture base resin and artificial teeth is one of the most important considerations in the technical procedure^{5,7}. Although plastic teeth adhere chemically to heat-curing resin during the curing process, defective bond between these materials has been highlighted in some studies^{8,9}. Of late, hard resin teeth have been regarded as the present-day plastic teeth for removable prostheses, where the former's body is made with hard resin. However, hard resin adheres poorly to acrylic denture base resin. Therefore the basal part of these teeth is made with acrylic resin, as acrylic resin can adhere chemically to acrylic denture base without any bonding agent (Fig.1). In this study, the acrylic resin material used for the adhesive surface of the hard resin teeth can be regarded as the general material used for the basal surface of plastic artificial teeth. However, even with the aforementioned design considerations incorporated in the structure of a hard resin tooth, defective bond be-

tween plastic teeth and cured acrylic is still relentlessly pointed out in many studies^{6,8–10}. Therefore, if an effective method were developed to improve the bond strength between these materials, it would be extremely invaluable to the technical procedure of denture fabrication.

To improve bond strength, various surface treatment methods of artificial tooth base have been proposed – such as air abrasion¹¹ and use of bonding agents^{12,13}. Many kinds of adhesive primer for the acrylic resin have been developed in the past. However, with these primers, the benefit has been limited

Fig. 1 Scheme of specimen made with polished basal surface of plastic artificial tooth.

to strengthening the adhesion between the denture lining material (self-curing resin or silicone type) and cured denture base acrylic resin^{12,14-17}. In the review of reports concerning the adhesion of artificial tooth to denture base, Marchack *et al.*¹² have reported on a bonding agent for porcelain bonding that improved the adhesion of porcelain tooth to heat-curing denture resin. However, no information is available regarding the adhesive primer for acrylic resin using heat curing system. Therefore, the development of an adhesive primer that can be used exclusively to paint the basal surface of plastic artificial teeth before packing the heat-curing acrylic resin in the flask¹ is highly desirable.

A new resin primer (Surface liner, Nissin Dental Product Inc., Kyoto, Japan) for heat-curing lining material (Physio Liner, Nissin Dental Product Inc.) has been developed to strengthen the adhesion between a lining material and heat-cured denture base acrylic. This primer, which was used in this study, has methylmethacrylate (MMA) as its main ingredient (85 weight%) and some low molecular weight polyethylmethacrylate (PEMA) (15 weight%) as an accessory ingredient. This primer was originally developed for application on the mucosal surface of cured acrylic denture base at the relining procedure of a new type of lining material in the dental laboratory. The new denture lining material was made with heat-curing resin. In this connection, the authors came up with a hypothesis that if the basal surface of plastic teeth — prior to denture packing with heat-curing acrylic resin — were painted with this primer, it could be an effective means to ensure prolonged adhesive strength between heat-cured acrylic resin and plastic artificial teeth after denture delivery. As dentures are stored in water if they are not in the patient's mouth, long-period resistibility to water is also another important consideration clinically.

This study therefore sought to examine the effect and durability of a new adhesive primer for heat-curing resin on the adhesive strength of heat-curing acrylic resin to plastic artificial tooth.

MATERIALS AND METHODS

Specimen preparation

Materials used to make the specimens in this study are listed in Table 1. Eighty plastic artificial teeth (ENDURA POSTERIO, Shofu Co., Kyoto, Japan) were embedded in resin rings (RING FORMS, BUEHLER, Lake Bluff, IL, USA) with self-curing resin (Unifast II Clear, GC Corp., Tokyo, Japan). The basal surfaces of artificial teeth were polished with #1200 silicon carbide abrasive paper (Struers A/S, Rodovre, Denmark) under water irrigation to remove pollutants and to obtain a flat uniform surface in the same manner described in a previous report¹⁸ (Fig. 1). Specimens were divided into four groups equally according to the treatment procedure for the bonding surface: adhesive primer application, air abrasion, adhesive primer application after air abrasion, or only pretreatment (for control).

1) For the control, the bonding surface was merely finished up with an abrasive paper as described above.

2) For air abrasion group and adhesive primer application after air abrasion group, air abrasion was performed using a laboratory sandblaster (HIBLASTER III; particle size: 50- μ m aluminum oxide, emission pressure: 0.4 MPa; Shofu Co.) onto the bonding surface — which was prepared with the same manner as for the control group. Device setup was a distance of 5 mm between the nozzle and bonding surface, and the surface was blasted for 30 seconds.

Besides these steps, column-form casting wax (Blue Inlay Wax, Shofu Co.) of 2-mm thickness and 3.6-mm diameter was put onto the bonding surface of all the specimens in the four groups. After flasking all specimens with plaster (Fig. 2), wax elimination was performed with hot water (100°C).

3) For adhesive primer application group and adhesive primer application after air abrasion group, the bonding surface was applied with a thin layer of adhesive primer (Surface liner, Nissin Dental Product Inc.) using a disposable paintbrush. The wetted surface was then left to dry in the air for two minutes.

After packing heat-curing acrylic resin (Acron, GC Corp.) in a dough consistency into the flask, heat

Table 1 Materials used in this study

Material (category)	Batch No.	Manufacturer
Acron (heat-curing acrylic resin)	Powder 0009051	GC Corp., Tokyo, Japan
	Liquid 0211132	
Unifast II (self-curing acrylic resin)	Powder 0106064	GC Corp., Tokyo, Japan
	Liquid 00407249	
Surface liner (adhesive primer)	BGD-G	Nissin Dental Product Inc., Kyoto, Japan
ENDURA POSTERIO (artificial tooth)	0504	Shofu Co., Kyoto, Japan

Fig. 2 Scheme of specimen in the flask. If flash of cured resin was grown at the test surface, it would lead to an incorrect adhered area of the specimen. Therefore, the flash should appear at the top of the cured acrylic column to avoid the flash at the test surface. This flash can be removed carefully with a grinding bur.

curing was performed in hot water at 74°C for 24 hours using a hot water bath (Curing Unit, J. Morita Corp., Osaka, Japan). After completing the curing course, specimens were de-flasked and stored in distilled water.

Measurement of shear bond strength and statistical analysis

Shear bond strength was determined respectively after 24-hour storage and 100-day storage in distilled water at 37°C. For each of the four groups, the twenty specimens were further divided into two groups. Each specimen were then mounted on a testing machine (Autograph DCSC-2000, Shimadzu, Kyoto, Japan), and shear stress was applied at a cross-head speed of 0.5 mm/minute. Data obtained for each of the four groups were statistically compared using two-way analysis of variance with Tukey-Kramer test.

After the shear test, all failed specimens were

analyzed using a light microscope ($\times 4$) (SMZ-10, Nikon, Tokyo, Japan) to determine the mode of failure. Failure type was classified as adhesive, cohesive, or mixed. Fracture mode results between different storage periods were then compared using Fisher's exact test. The effect of bonding surface treatment on the failure type was also analyzed using two-way analysis of variance and Holm-Sidak post-hoc test.

SEM observation of bonding surface

Before the shear test, scanning electron microscopy (SEM) (DS-720, TOPCON Corp., Tokyo, Japan) was employed to detect and observe morphological changes of the bonding surface due to the different treatment methods. Prior to SEM observation, specimens were dried in a desiccator for 24 hours and then gold-coated using ion sputter (JFC-1100E, JOEL, Tokyo, Japan).

RESULTS

Table 2 presents the mean values and standard deviations of shear bond strength for the four groups. Based on statistical analysis using two-way analysis of variance, both treatment method and storage period significantly affected the adhesive strength ($p < 0.001$). The shear bond strength of the 100-day storage groups showed a significantly smaller value as compared to the 24-hour storage groups ($p < 0.05$) (Table 2). In the 24-hour storage groups, all the three treatment groups showed a higher value than that of the control (Table 2). However, in the 100-day storage groups, the air abrasion group did not show any significant difference from the control group, whereas adhesive primer application group and adhesive primer application after air abrasion group were significantly different from the control. In addition, adhesive primer application and adhesive primer application after air abrasion groups showed larger values than the control and air abrasion groups (Table 2).

Whereas all the specimens in the control groups failed adhesively for both storage periods, other groups showed cohesive failure or mixed failure in some specimens. The effects of both storage period

Table 2 Shear bond strength values of specimens in different storage periods

Group	24-hour storage	100-day storage	p value ^a
	Mean (SD)	Mean (SD)	
Air abrasion	15.65 (3.61) ^A	8.44 (1.52) ^D	<0.001
Adhesive primer application	19.60 (4.85) ^{AB}	12.81 (4.66) ^E	<0.001
Adhesive primer application after air abrasion	19.80 (2.77) ^B	15.20 (3.56) ^E	0.004
Pretreatment (control)	11.61 (4.85) ^C	8.28 (3.03) ^D	0.032

Unit: MPa, n=10.

^aTukey-Kramer test.

With storage time, mean values with the same letter are not significantly different ($p > 0.05$, Tukey-Kramer test).

and treatment method on the failure type were not observed statistically (Table 3).

Fig. 3 shows the SEM images of the bonding surfaces subjected to different surface treatments before shear test. The SEM image of the bonding surface for control group showed slight grooves of straight and parallel lines due to pretreatment with abrasive paper. Conversely, with air abrasion group, a roughened irregular surface was obtained due to sandblasting. As for adhesive primer application group and adhesive primer application after air abrasion group, the primer layer that dissolved the plastic tooth surface was observed on the bonding surfaces of these two groups.

DISCUSSION

For the results of 24-hour storage groups, the mean shear bond strength values of the four groups ranged approximately from 12 to 20 MPa (Table 2). These values were in agreement with the results of a previous report that investigated the shear bond strength of acrylic resin for denture repair¹⁹⁾. From the ANOVA results, it was shown that both the storage period and treatment method affected adhesive strength significantly. When the storage period was prolonged, the adhesive strengths of all treatment groups decreased (Table 2). This decrease in adhesive strength could be caused by a complex combination of factors that occurred as the storage period was prolonged: deterioration of the heat-cured

Table 3 Failure modes of specimens after shear test

Group	24-hour storage	100-day storage	P ^{a1}
Pretreatment (control)	N=10 (AF: 10, MF: 0, CF: 0)	N=10 (AF: 10, MF: 0, CF: 0)	NS
Air abrasion	N=10 (AF: 8, MF: 2, CF: 0)	N=10 (AF: 10, MF: 0, CF: 0)	NS
Adhesive primer application	N=10 (AF: 6, MF: 3, CF: 1)	N=10 (AF: 9, MF: 1, CF: 0)	NS
Adhesive primer application after air abrasion	N=10 (AF: 6, MF: 3, CF: 1)	N=10 (AF: 8, MF: 2, CF: 0)	NS
P ^{a2}	NS	NS	

N: Number of specimens; AF: Adhesive fracture at bonding site; MF: Mixed fracture; CF: Cohesive fracture; NS: Not significant.

^{a1}Fisher's exact test; ^{a2}Two-way analysis of variance with Holm-Sidak post-hoc test.

Fig. 3 SEM images of the bonding surfaces on artificial tooth side before test: (a) pretreatment; (b) air abrasion; (c) adhesive primer application; (d) adhesive primer application after air abrasion.

resin itself and the dissolution of the resin by the adhesive primer which was applied to the bonding surface¹⁶⁾. Indeed, adhesive primer application group and adhesive primer application after air abrasion group showed significant decrease in adhesive strength with prolongation of the storage period. However, the adhesive strengths of these two groups were significantly larger than the control and air abrasion groups (Table 2). Failure type results also seemed to support these results although statistical significance was not observed (Table 3). In particular, adhesive primer application after air abrasion group showed approximately twice the adhesive strength value of the control. Taken together, these results showed the effectiveness and durability of the primer used in this study. As little information is available regarding the adhesive strength between plastic tooth and heat-cured acrylic resin, these results were useful in a technical aspect.

When packing acrylic resin into the flask, free MMA in the dough-state resin causes the plastic tooth resin surface to swell and dissolve. This action makes the plastic tooth adhere to heat-curing acrylic resin. However, as packing is performed at the late dough stage, free MMA in the dough resin decreases. This decrease weakens the adhesive strength between plastic tooth and acrylic denture base resin. The MMA of the primer used in this study acted on dissolving the plastic tooth surface. The PEMA in the primer was compounded originally to increase the adhesive strength of lining materials to denture base resin. On the surface that had adhesive failure after test, a slight layer containing applied primer was observed by examination using a light microscope (SMZ-10, Nikon). With this primer-containing layer adhered to the bonding surface of plastic tooth, it indicated the dissolving action of the primer against the plastic tooth surface.

The SEM image of the air abrasion group showed a distinct roughening effect on the bonding surface (Fig. 3). The air abrasion group showed significantly higher adhesive strength than the control after 24-hour storage (Table 2). However, after 100-day storage, the air abrasion group showed a small value which was not significantly larger than that of the control. This overall small value of 100-day storage groups could be attributed to the deterioration of the heat-cured resin or roughened tooth resin at the bonding area, whereby this deterioration was brought about by water sorption of the material^{16,20)}. These findings therefore suggested that the primer used in this study was superior to the air abrasion treatment, especially when the specimens were stored in water for a long time.

This study examined the effectiveness of an adhesive primer on the adhesion of plastic teeth to heat-curing resin. Further investigation would be beneficial, since limited products were used for testing in

this study. In addition, it would also be beneficial to clarify the process of the dissolving action of MMA in the primer.

Within the limitations of this study, it was concluded that the adhesive primer examined in this study was effective in increasing the shear bond strength of plastic artificial tooth to heat-curing acrylic resin.

CONCLUSION

The adhesive primer examined in this study was significantly effective in increasing the adhesive strength between artificial tooth and acrylic resin, although specimens were stored in water for 100 days.

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