

Influence of composition on the adhesive strength and initial viscosity of denture adhesives

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To investigate the effect of composition on the initial viscosity and adhesive strength between denture adhesives and the denture base. Two types of water-soluble polymers (methoxy ethylene maleic anhydride copolymer [PVM-MA] and sodium carboxymethyl cellulose [CMC]) were used. Samples were divided into three groups. Group 1 contained only PVM-MA; Group 2 contained only CMC; and Group 3 contained PVM-MA and CMC. The initial viscosity and adhesive strength were measured. For Group 1, the initial viscosity increased significantly as PVM-MA content increased. The adhesive strength of Group 1 lasted longer than Group 2. The adhesive strength of Group 3 varied greatly. The ratio of CMC and PVM-MA has a significant effect on the initial viscosity and adhesive strength of denture adhesives. Our results suggest that it is possible to improve the durability of a denture adhesive by combining different water-soluble polymers.

Keywords: Denture adhesive, Viscosity, Adhesive strength, Water-soluble polymer

INTRODUCTION

Denture adhesives emerged almost at the same time as modern denture prostheses¹. Although the use of denture adhesives has been a controversial topic in the dental profession¹, millions of denture wearers continue to buy and use denture adhesives. In 1980, 15% of denture wearers used denture adhesives in the US^{2,3}. In 1990, Wilson *et al.*⁴ reported that 30% of denture wearers used or had used denture adhesives, although Coates⁵ reported that a significant number of subjects in his study did not know that denture adhesives existed. More than 5 million Americans use denture adhesives, and approximately 75% of all dentists recommended the use of denture adhesives to their denture patients¹. Along with the aging of the world population and the increase in edentulous individuals, the total consumption of denture adhesives is increasing. Many studies on the use of denture adhesives *in vitro* and *in vivo* confirm the efficacy of denture adhesives in improving the denture retention and stability of both well-fitting and ill-fitting dentures^{6,7}, increasing the bite force^{8,9}, improving taste discrimination and taste perception¹⁰, minimizing problems with food particles under the denture, and increasing the wearer's confidence during social activities and chewing¹¹. There is no doubt that denture adhesives make life easier for denture wearers and are an effective adjunct to denture treatment and denture aftercare¹².

Modern denture adhesives are available in different forms such as creams, strips, powders and cushion to

fulfill a range of consumer preferences. Dental professionals are most likely to recommend a cream-type denture adhesive. Denture adhesives contain active and non-active ingredients. The active ingredients which confer the adhesive properties include karaya gum, tragacanth, acacia, pectin, gelatin, methyl cellulose, hydroxymethyl cellulose, sodium carboxymethyl cellulose and synthetic polymers (polyethylene oxide, acrylamides, acetic polyvinyl)^{12,13}. The non-active ingredients, such as petrolatum, mineral oil, and polyethylene oxide, act as binding materials to facilitate placement^{12,13}. Variations in the composition of denture adhesives affect denture retention¹⁴. A carboxymethyl cellulose (CMC) and methoxy ethylene maleic anhydride copolymer (PVM-MA) is also used as an active ingredient. However, most cream-type commercial denture adhesives use CMC as active ingredients¹. We guess the ability of denture adhesive may be improved by using together the CMC and PVM-MA as active ingredients.

The adhesive strength and viscosity are the most important properties of a denture adhesive. An ideal denture adhesive should retain its adhesive properties for 12 to 16 h before requiring re-application¹², and the initial viscosity should allow easy manipulation without compromising the adhesive strength. Koronis *et al.*¹¹ and Uysal *et al.*¹⁵ found that only 68–75.7% of people surveyed reported that the retentive capacity of denture adhesives lasted for more than 2 h. However, several other studies reported that denture adhesives can remain effective for more than 8 h^{16,17}, with the retentive capacity peaking at 2 to 4 h after application^{16,17},

and declining thereafter. There is a problem with commercial denture adhesives, that is maximum adhesive strength can be lasted only 2–4 h^{16,17}. In our preliminary study, a lot of commercial denture adhesives were the tendencies that the adhesive strength decreased after immersed into water for 1–2 h. However, in the clinical situation, it is necessary that the maximum adhesive strength can be lasted for more than 6 h. The small period of effective retention offered by denture adhesives may be the reason why some denture wearers apply them several times a day in order to keep their dentures in place. Such overuse may increase the side effects of the denture adhesive. Increasing the period of adhesive efficacy is an important challenge in the improvement of denture adhesives. However, few studies have examined how composition affects their adhesive properties.

The aim of this study was to investigate the use of water-soluble polymers to try and produce a more durable denture adhesive. We used CMC and PVM-MA as active ingredients, and white petrolatum (WPL) and liquid paraffin (LP) as non-active ingredients, to examine the influence of composition on the initial viscosity and adhesive strength between the denture adhesive and the denture base.

MATERIALS AND METHODS

Two types of water-soluble polymers (PVM-MA and CMC) and two base materials (WPL and LP) were used in this study. Details of the components and the composition of the samples are shown in Tables 1 and 2, respectively. The samples were mixed using a vacuum mixer (VM-II, GC Co., Tokyo, Japan), and were divided into three groups. Group 1 contained only PVM-MA and WPL (TDA-I, TDA-II, TDA-III); Group 2 contained only CMC and WPL (TDA-IV and TDA-V); and Group 3 contained PVM-MA and WPL and/or LP (TDA-VI, TDA-VII, TDA-VIII and TDA-IX). A control of 100% WPL was used.

The initial viscosity of the materials was measured using a controlled-stress rheometer (CarriMed CSL500, TA Instruments Ltd, New Castle, DE, USA) in dynamic mode with a cone-and-plate configuration. The radius of the upper cone was 10 mm with a 2° cone angle, and the gap between the plates was 54 μm (Fig. 1)¹⁸. The instrument was used in a constant strain mode with an angular velocity of 10 rad/s at 37°C.

Adhesive strength was measured according to ISO-10873 recommended procedures¹⁹. The hole of the sample holder (Fig. 2)¹⁸ was filled with denture

Table 1 Components used

Polymer	Manufacturer	Lot no.
Methoxy ethylene maleic anhydride copolymer (PVM-MA)	ISP Japan LTD., Tokyo, Japan	CC600150446
Sodium carboxymethyl cellulose (CMC)	Daiichi Kogyo Seiyaku Co., LTD., Kyoto, Japan	353847
White petrolatum (WPL)	Nikko Pharmaceutical Co., LTD., Hashima, Japan	669319
Liquid paraffin (LP)	Wako Pure Chemical Industries Ltd., Osaka, Japan	609061

Table 2 Composition of samples

Groups	Code	PVM-MA	CMC	WPL	LP
	Control	0 wt%	0 wt%	100 wt%	0 wt%
Group 1	TDA-I	40 wt%	0 wt%	60 wt%	0 wt%
	TDA-II	50 wt%	0 wt%	50 wt%	0 wt%
	TDA-III	60 wt%	0 wt%	40 wt%	0 wt%
Group 2	TDA-IV	0 wt%	30 wt%	70 wt%	0 wt%
	TDA-V	0 wt%	35 wt%	65 wt%	0 wt%
Group 3	TDA-VI	35 wt%	15 wt%	50 wt%	0 wt%
	TDA-VII	25 wt%	25 wt%	50 wt%	0 wt%
	TDA-VIII	35 wt%	15 wt%	40 wt%	10 wt%
	TDA-IX	35 wt%	20 wt%	40 wt%	5 wt%

wt%: weight percentage

adhesive, and the surface was flattened. The immersion time was referred to former study¹⁸⁾. The sample holder was immersed in water at 37°C for 0, 1, 10, 30, 60, 180 or 360 min, taken out and shaken once to remove water from the sample surface. The sample holder was fixed on the sample stand, and a load of 9.8±0.2 N was applied to the sample using a constant load compression

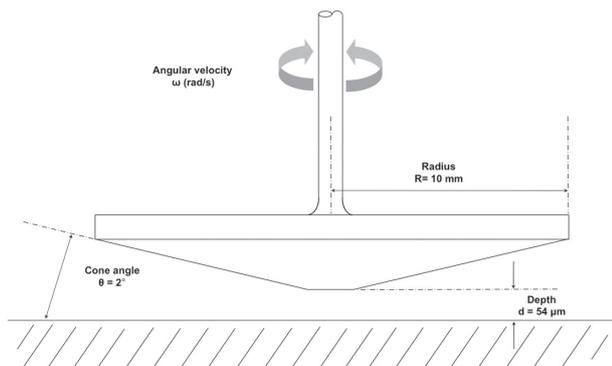


Fig. 1 Block diagram of jig for viscosity test¹⁸⁾.

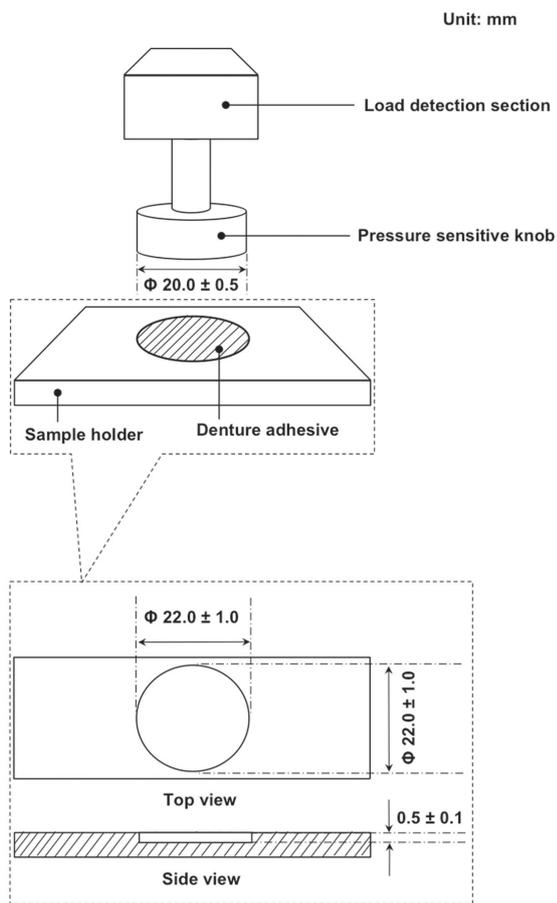


Fig. 2 Block diagram of sample holder for adhesive strength test¹⁸⁾.

testing machine (A-001, Japan Mecc Co. Ltd, Tokyo, Japan) at a pressurizing velocity of 5 mm/min using a 20±0.5 mm pressure sensitive knob, and maintained for 30 s. The sample was then pulled in the reverse direction with tensile velocity using a materials testing machine (Model 5565, Instron Co., Canton, MA, USA) at a crosshead speed of 5 mm/min. The maximum force on the pressure sensitive knob was measured at that time and the force per unit area was set as the adhesive strength. The sample holder and pressure sensitive knob were prepared using denture base acrylic resin (Acron, Lot No. Powder-030471, Liquid-0112203; P/L:10/4.3 g; GC Co., Tokyo, Japan), and were polymerized according to the manufacturer's instructions. The surfaces were abraded with 400 grit waterproof abrasive papers, scrubbed with tap water for 15 s, and allowed to air dry for at least 5 min.

All the data were analyzed independently by one-way analysis of variance (ANOVA) combined with a Student-Newman-Keuls (SNK) multiple comparison test at a 5% level of significance.

RESULTS

Figure 3 shows the initial viscosity of the samples. Identical letters indicate no significant differences. The control group (100% WPL) had the lowest initial viscosity. For Group 1 samples, the initial viscosity significantly increased as the content of PVM-MA increased. For Group 2, the initial viscosity significantly decreased as the content of CMC slightly increased from 30 wt% to 35 wt%. Amongst the Group 3 samples, the initial viscosity of TDA-VIII was lower than TDA-VI, TDA-VII and TDA-IX. The TDA-IX samples had the highest initial viscosity of all the samples, but were not significantly different from TDA-VI and TDA-VII samples.

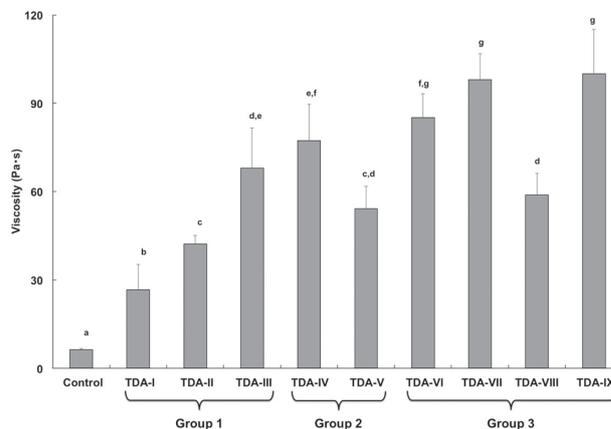


Fig. 3 Initial viscosity of materials with standard deviation. Identical letters indicate no significant difference ($p > 0.05$, SNK-test).

Table 3 Adhesive strength of each material with standard deviation

Material	Time		Mean of adhesive strength (SD) (KPa)				
	0 min	1 min	10 min	30 min	60 min	180 min	360 min
Control	83.46 (9.25)	58.12 (6.31)	58.94 (7.51)	61.52 (4.45)	60.79 (5.87)	59.04 (5.86)	57.20 (5.86)
TDA-I	53.68 (5.34)	58.49 (9.72)	65.54 (1.57)	65.13 (8.39)	76.68 (17.80)	74.40 (12.85)	83.35 (6.69)
TDA-II	47.46 (8.21)	78.25 (11.68)	70.75 (7.77)	64.89 (5.87)	71.04 (7.41)	75.28 (8.18)	80.02 (7.00)
TDA-III	40.77 (8.78)	69.16 (10.43)	76.90 (6.18)	78.97 (8.54)	74.46 (10.06)	84.15 (15.20)	74.26 (4.13)
TDA-IV	62.51 (6.76)	106.43 (16.85)	105.32 (6.88)	96.94 (12.96)	103.02 (8.09)	82.15 (8.70)	47.75 (9.12)
TDA-V	50.14 (3.99)	100.89 (14.61)	119.86 (6.30)	114.02 (8.54)	110.24 (7.64)	93.24 (5.30)	49.40 (8.56)
TDA-VI	46.91 (10.22)	99.05 (15.15)	76.69 (11.86)	80.36 (20.72)	89.28 (16.91)	106.01 (18.31)	97.71 (15.86)
TDA-VII	29.79 (3.53)	100.13 (12.83)	128.03 (4.79)	100.85 (17.49)	117.97 (6.55)	100.57 (16.31)	78.42 (9.15)
TDA-VIII	54.73 (13.50)	83.78 (6.99)	57.04 (14.50)	52.73 (5.72)	47.85 (3.19)	62.63 (7.00)	71.19 (7.49)
TDA-IX	44.94 (7.40)	97.10 (20.71)	101.15 (3.42)	112.67 (11.58)	109.80 (11.56)	109.14 (16.12)	119.97 (18.67)

SD: standard deviation

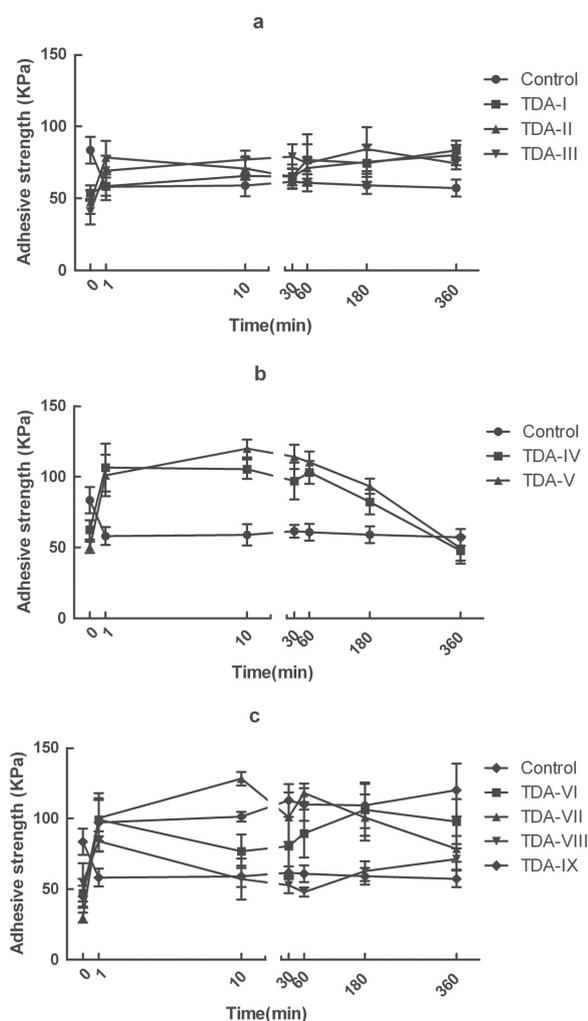


Fig. 4 Variations in adhesive strength of different groups with immersion time.
(a) Group 1, (b) Group 2, (c) Group 3

Table 3 and Fig. 4 show the variations in adhesive strength of different groups according to immersion time. The adhesive strength sharply increased after immersion in water for 1 min for all test groups, while sharply decreasing for the control group. For Group 1 (Fig. 4-a), the adhesive strength slightly increased over time after the samples were immersed into water for 1 min, and achieved the highest values after 180 min or 360 min. There was no significant difference among any of the Group 1 samples (TDA-I, TDA-II, TDA-III) at any of the test points.

For Group 2 samples (TDA-IV and TDA-V) (Fig. 4-b), the adhesive strength peaked at 1–10 min, and then significantly decreased over time. At the end of the test period, the adhesive strength was even lower than the control group. For Group 3 samples (TDA-VI, TDA-VII, TDA-VIII and TDA-IX) (Fig. 4-c), the adhesive strength varied greatly. TDA-IX exhibited the best adhesive properties, consisting of high initial adhesive strength which was maintained for more than 360 min.

DISCUSSION

The active ingredients of denture adhesives swell and become viscous and sticky in the presence of water or saliva. The increased volume fills the voids between the denture base material and the oral mucosa with an intermediary thin film of saliva, creating retentive forces. CMC and PVM-MA are often used as active ingredients in today's denture adhesives, especially in cream type adhesives, which are designed to achieve both short-term and long-term effectiveness¹⁾. CMC has higher solubility and provides a strong initial hold, but it dissolves quickly and loses its effectiveness within a relatively short period. This effect can be observed in Fig. 4-b and Table 3, which demonstrates that the Group 2 samples containing only CMC had a higher initial strength, and achieved their highest adhesive strength

after immersion in water for 1–10 min, followed by a decline in adhesive strength. PVM-MA is less soluble, allowing it to play a positive role later, and last for longer. As seen in Fig. 4-a and Table 3, the initial adhesive strength of the Group 1 samples containing only PVM-MA was lower, but it increased steadily over time, with very little decrease in adhesive strength over the entire test time. These results are consistent with those of Kulak, who reported that denture adhesive paste based on PVM-MA compounds rated higher than CMC adhesive paste on chewing ability and duration of effectiveness in the mouth²⁰. The Group 3 samples containing both CMC and PVM-MA not only had higher initial adhesive strength, but also had a longer duration of effectiveness. However, not all the samples with both CMC and PVM-MA performed better than the samples containing a single component. Figure 4-c and Table 3 shows that TDA-VIII did not perform as well as the other Group 3 samples, demonstrating that the ratio of the different components is also a very important factor.

The adhesive strength of a denture adhesive is the most important factor for clinical use of these materials. Several papers have investigated the adhesive strength of denture adhesives; however, it is difficult to compare the adhesive strength values among studies because of the different test methods used^{21–24}. In this study, we used the test method required by the Internal Standard Organization, which is well accepted worldwide. According to the ISO-10873 standard¹⁹, the adhesive strength must be 5 kPa or more in the clinical situation. Hong *et al.*¹⁸ reported that the adhesive strength of commercial cream-type denture adhesives showed from 50 kPa to 90 kPa after 10 min immersion in the distilled water. The adhesive strength of all formulations used in our study was much higher than 5 kPa, and met the ISO standard requirement throughout the test period. The adhesive strength of TDA-IX was found to be much higher than commercial denture adhesives. According to Fig. 4-c and Table 3, the range of adhesive strength of TDA-IX was 97.1 kPa to 119.9 kPa in the test period of 1–360 min, while for commercial denture adhesives the adhesive strength ranged between 50.8 kPa to 87.8 kPa using the same test method¹⁸.

The viscosity of denture adhesives is associated with their manipulation, retention, and oral hygiene^{25–27}. Because main component of the cream-type denture adhesive is water-soluble polymers, the material dissolves progressively during clinical use^{12,13}. Therefore, these type materials are easy to wash. Finally the materials left to the denture base can easily remove by washing it under running water. When denture adhesives are first put into a water bath, the viscosity is relatively low, which allows them to be easily manipulated and adjusted. The polymer particles slowly absorb water and swell, and the viscosity increases until finally the particles come into contact with one another and a maximum viscosity is reached as a continuous polymer matrix is formed, facilitating retention²⁹. However, it is difficult to establish the most acceptable viscosity level for denture adhesives. Ellis²⁵

reported that denture adhesives with viscosities in the order of 10^6 poise provide the most effective retention. At lower viscosities retention may not be adequate, and at higher viscosities hygiene can be a problem because it is difficult to clean the residual adherent mass from the denture after use. The present study measured only the initial viscosity, and did not measure variations in viscosity over time. Figure 3 shows that the control group (100% WPL) had the lowest viscosity, while TDA-IX had the highest initial viscosity. The initial viscosity increased as PVM-MA content increased.

The inactive ingredients (WPL and LP) also have a significant effect on the adhesive strength and initial viscosity of denture adhesives. LP decreases the initial viscosity and may improve the manipulation properties. As shown in Table 2, the TDA-VIII samples varied from the TDA-VI samples by the addition of 10 wt% LP and a decrease of WPL from 50 wt% to 40 wt%. The initial viscosity and adhesive strength of TDA-VIII was significantly lower than TDA-VI. TDA-IX not only had higher initial strength, but also a longer duration of effectiveness, which may be attributed to the protective function of WPL and LP. WPL and LP both counteract swelling and dissolution of PVM-MA and CMC, resulting in good adhesive properties for a longer period of time. This effect has also been observed in clinical comparisons of denture adhesives²⁸.

Recent concerns have arisen about the adverse systemic effects of denture adhesives, due to reports that excessive zinc ingestion from denture adhesive overuse may cause depression of serum copper, resulting in bone marrow depression and widespread sensory and motor neuropathies^{27,29}. Minimizing the use of denture adhesives while maximizing their effectiveness may be the best way to avoid adverse systemic effects. This could be achieved by adjusting the composition of denture adhesives without using zinc-containing ingredients. Proper education of dentists and patients is also very important, and neither dentists nor patients should use denture adhesives as a substitute for good clinical practice or a proper denture maintenance routine³⁰.

The composition of denture adhesives has a significant effect on the initial viscosity and adhesive strength, both of which are related to the clinical manipulation properties and effective acting time. The initial viscosity and adhesive strength can be improved by changing the composition and ratio of different components. The formulation of DA-IX was found to have the highest initial viscosity and a higher adhesive strength without compromise throughout the test period.

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