A STUDY ON THE ADHESION OF A SOFT LINER CONTAINING 4-META TO THE BASE METAL ALLOY AND ITS VISCOELASTIC PROPERTY

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Statement of problem. Soft lining materials, also referred to as tissue conditioning materials, tissue treating materials, relining materials, soft liners or tissue conditioners, were first introduced to dentistry by a plastic manufacturer in 1959.
Since the introduction of the materials to the dental field, their material properties have been continually improved through the effort of many researchers. Soft lining materials have become widely accepted, particularly by prosthodontists, because of their numerous clinical advantages and ease of manipulation. Unfortunately, few reports have been issued upon the topic of increasing the bond strength between the base metal alloy used in cast denture bases and PMMA soft liner modified with 4-META, nor upon the pattern of debonding and material change in wet environment like a intra oral situation.
Purpose. The purposes of this study were comparing the bond strength between base metal alloy used for the cast denture bases and PMMA soft liner modified with 4-META, and describing the pattern of debonding and material property change in wet environment like the intraoral situation.
Material and Methods. This study consisted of four experiments:
1. The in vitro measurement of shear bond strength of the adhesive soft liner.
2. The in vitro measurement of shear bond strength of the adhesive soft liner after 2 weeks of aging.
3. A comparison of debonding patterns.
4. An evaluation the gelation time of modified soft liner.

The soft liner used in this study was commercially available as Coe-soft (GC America.IL.,USA), which is provided in forms of powder and liquid. This is a PMMA soft liner commonly used in dental clinics. The metal primer used in this study was 4-META containing primer packed in Meta fast denture base resin (Sun Medical Co., Osaka, Japan).
The specimens were formed in a single lap joint design, which is useful for evaluating the apparent shear bond strength of adhesively bonded metal plate by tensile loading.
Using the 20 × 20mm transparent grid, percent area of adhesive soft liner remaining on the shear area was calculated to classify the debonding patterns.
To evaluate the change of the initial flow of the modified adhesive soft liner, the gelation time was measured with an oscillating rheometer (Haake RS150W / TC50, Haake Co., Germany). It was a stress control and parallel plate type with the diameter of 35mm.
Conclusion. Within the conditions and limitations of this study, the following conclusions were drawn as follows.
1. There was significant increase of bond strength in the 5% 4-META, 10% 4-META containing groups and in the primer coated groups versus the control group($P<0.05$).
2. After 2 weeks of aging, no significant increase in bond strength was found except for the group containing 10% 4-META ($P<0.05$).
3. The gelation times of the modified soft liner were 9.3 minutes for the 5% 4-META containing liner and 11.5 minutes for the 10% 4-META liner.
4. The debonding patterns of the 4-META containing group after 2 weeks of aging were similar to those of immediaely after preparation, but the debonding pattern of the primer group showed more adhesive failure after 2 weeks of aging.

Key Words
Adhesive soft liner, Gelation time, 4-META, Shear bond strength, Oscillating rheometer, PMMA

Soft lining materials, also referred to as tissue conditioning materials, tissue treating materials, relining materials, soft liners or tissue conditioners, were first introduced to dentistry by a plastic manufacturer in 1959.[1,2] In 1961, Chase reported on their use as a tissue conditioning or functional impression materials.[3] Since the introduction of the materials to the dental field, their material properties have been continually improved through the effort of many researchers. Soft lining materials have become widely accepted, particularly by prosthodontists, because of their numerous clinical advantages and ease of manipulation.

Today, soft lining materials have become an essential treatment tool, because of their viscoelastic properties, they are mainly used as re-conditioners of abused tissue. That is to say, their unique properties enable a more even distribution of functional and nonfunctional stresses and thereby limits the occlusal impact. The materials are effective for treating patients with 1) ridge atrophy and resorption 2) bruxing tendency 3) xerostomia 4) bony undercut 5) denture opposing natural dentition in the opposing arch, and 6) congenital or acquired oral defect.[4]

In addition to the treatment of patients with abused tissue and ill fitting dentures, the materials can be used as aids for stabilizing the recording base or as diagnostic aids for evaluating the problems of old denture wearers.[5]

For successful treatment, soft lining materials should be:[6]

1) biocompatible with the intraoral tissue,
2) compliant and resilient,
3) dimensionally stable,
4) acceptable in terms of their rupture properties,
5) resistant to microorganisms,
6) easy to manipulate in dental clinics.

Although these materials have various clinical applications and advantages, they also have several disadvantages such as short or insufficient intra-oral durability, tendency to evoke Candidiasis, lack of a durable bond with the denture base, a tendency to absorb water.

Of the above, the lack of a durable bond to the denture base is one of the most critical shortcomings, particularly so when a cast metal denture base are used, as failure of this bond has often led to somewhat embarassing situations for many dentists. Metal based dentures have been preferred over resin based complete dentures they have because of several advantages including:[5]

1) reduced bulkiness-providing more patient comfort,
2) better tactile sensitivity,
3) better strength and thermal conductivity,
4) stability in the lower denture,
5) greater biocompatibility,
6) better polished surfaces with a lower tendency to accumulate plaque.

However, when patients suffer from denture sore spots due to fragile supporting mucosa and advanced residual ridge resorption, relining the metal surface with soft lining materials can be one of the most demanding procedures for clinicians. In addition, it is not uncommon to have to make a single maxillary complete denture to oppose mandibular natural teeth. Patients with such single dentures will often experience fracture of the resin denture base. As an alternative to minimizing the risks of fracture, clinicians can offer a metal or gold based complete denture, but even in such a case relining with a soft liner is required every few years. Moreover, the debonding a soft liner from its base creates a localized unhygienic condition and causes the functional failure of complete or partial dentures.

Nowadays more and more implant prostheses are relpacing the conventional dentures, but even in the case of implant prosthodontics relining the old denture with soft liner is necessary during the healing phase to enable pressureless or least pressured healing. However, at the time of inserting a relined denture, the lack of bond durability between the soft liner and the metal base can be a problem. To lessen the difficulty of retaining the relining material beneath a cast metal framework, micro-retentions were used, which were difficult to achieve because of the hardness of the base metal alloy. As an alternative to these retention systems, Garfield[7] reported upon an etched metal framework technique, and suggested that such microscopic retention could increase bond strength.

However, such mechanical retention has proven to be insufficient in the wet intra oral environment, and the absence of a chemical bond could cause fatigue failure due to oral fluids and micro-organisms and may result in or may accelerate discoloration.[8]

Tanaka et al.[9] recently reported using 4-META (4-methacryloxyethyl trimellitate anhydride), at 5% on MMA monomer, could overcoming their problems, and Watanabe et al.[10] reported that stronger bonds were attainable with 4-META containing PMMA resin, which chemically bonds to metal oxides. However all efforts to date have been limited to hard PMMA denture base resins.[11-18]

Unfortunately, few reports have been issued upon the topic of increasing the bond strength between the base metal alloy used in cast denture bases and PMMA soft liner modified with 4-META, nor upon the pattern of debonding and material change in wet environment like a intra oral situation.[19]

The purposes of this study were comparing the bond strength between base metal alloy used for the cast denture bases and PMMA soft liner modified with 4-META, and describing the pattern of debonding and material property change in wet environment like the intraoral situation.

MATERIALS AND METHODS

1. Materials

The soft liner used in this study was commercially available as Coe-soft, which was provided in forms of powder and liquid. This is a PMMA soft liner commonly used in dental clinics. The metal primer used in this study was 4-META containing primer packed in Meta fast denture base resin.

The 4-META used in this study was provided by the courtesy of Sun Medical Corporation. Other materials used in this study are in Table Ⅰ. The chemical structure of 4-META is shown in Fig. 1.

$$\begin{array}{l} \quad\ \ CH_3 \\ \quad\ \ \ | \\ CH_2{=}C{-}COO(CH_2)_2{-}OOC{-}(CH_2)_6{-}CO{-}O{-}CO \end{array}$$

Fig. 1. The chemical structure of 4-META.

Table Ⅰ. Materials used in this study

Products	Manufacturers	Materials
Coe-Soft	GC America.,USA	PMMA liner
4-META	Sun medical Co., Japan	4-META
Ticonium	Ticonium Co., USA	Co-Cr alloy
Metafast bonding liner	Sun medical Co., Japan	Primer

2. Methods

This study consisted of four experiments:

1. In vitro measurement of shear bond strength of the adhesive soft liner,
2. In vitro measurement of shear bond strength of the adhesive soft liner after 2 weeks of aging,
3. A comparison of debonding patterns,
4. An evaluation the gelation time of modified soft liner.

In vitro measurement of shear bond strength of the adhesive soft liner

To evaluate the bond strength of modified adhesive soft liner to metal alloy, 10 specimens were prepared for each of 4 groups. The dimension and the design of the test specimens were determined according to ASTM standard D 1002-99 with some modification(Fig. 2).[20] The specimens were formed in a single lap joint design, which is useful for evaluating the apparent shear bond strength of adhesively bonded metal plate by tensile loading.

To prepare specimens, 80 metal plates ($60 \times 20 \times 2$mm)were prepared by the conventional casting method using a Co-Cr alloy. After casting, the sprue was cut away and the metal plate were cleaned with an ultrasonic cleaner for 3 minutes to remove any residual metal particles or investment particles and then sandblasted with 50μm aluminium oxide(Al_2O_3) by the pressure at 30 bar.

The experiment involved 80 plates divided into 4-different groups. As a control group, the specimens of group 1 were not engaged in the 4-META powder and primer. According to the manufacturer' s instruction, the powder and the liquid mixed for 30 sec, and 10 single lap joint specimens, with a $20 \times 20 \times 2$mm adhesive layer were prepared using 2 metal plates and a Coe-soft liner.

In group 2, the 4-META powder was mixed with the Coe-soft liquid to 5% weight until the powder was totally incorporated.

Then another 10 specimens of group 3 were prepared using Coe-soft liner containing 10% 4-META

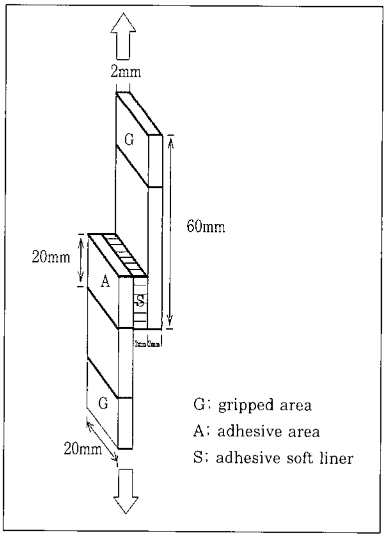

Fig. 2. The diagram of the specimen.

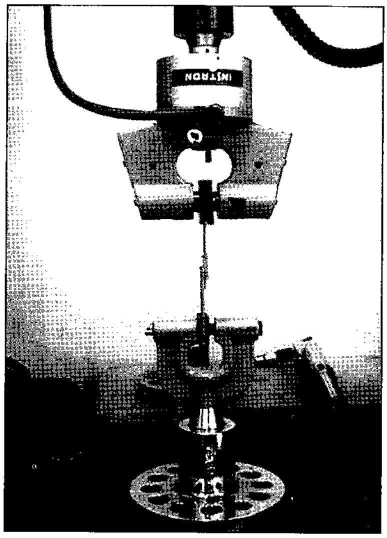

Fig. 3. Instron with test specimen on the pneumatic grip.

powder by weight in Coe-soft liquid.

In the case of group 4, before making the single lap joint specimen with Coe-soft, the adhesive primer was brush-coated once on the shear area of two adhering metal surface. To preserve the prepared specimens without deformation, base plate wax was used to support the cantilevered portion of the metal plate. The diagram of each specimens are shown in Fig. 2.

Apparent shear bond strength by tension loading was tested with Universal testing machine Instron-6000(Instron Co., USA) at a 10mm/sec cross head speed.

The upper arm was the pneumatic grip, and the lower one was the tensile grip(Fig. 3).

The grips and attachment were constructed to move into alignment with the test specimen as soon as the load was applied, so that the long axis of test specimen would coincide with the direction of the applied load pull through the center line of the grip assembly. The peak value of shear bond strength was recorded.

Table II. Composition and ratio of artificial saliva (g/l)

NaCl	0.4
KCL	0.4
$CaCl_2.H_2O$	0.8
$Na_2S.5H_2O$	0.01
$CO(NH_2)_2$urea	1.0

<u>In vitro measurement of shear bond strength of the adhesive soft liner after 2 weeks of aging</u>

To evaluate the shear bond strength after aging, ten specimens were prepared for each of the 4 groups in a method identical to that of the previous experiment. To represent the wet and warm intra-oral environment, 10 L of artificial saliva was prepared using a magnetic stirrer according to the composition suggested by Indiana University as shown in Table Ⅱ.

In a 37℃ water bath filled with artificial saliva, 40 specimens of the 4 groups were stored for 2 weeks. After 2 weeks, the aged specimens were tested using the same method and the apparatus as those of the previous experiment.

The short description of these two test for the shear bond strength is shown in Table Ⅲ.

<u>Comparison of debonding patterns</u>

After measuring the apparent shear bond strength, the debonding pattern of all the separated specimens was observed. Using the 20×20mm transparent grid, percent area of adhesive soft liner remaining on the shear area was calculated.

The debonding pattern was classified into 3 groups by the following criteria:

(1) cohesive failure--over 80% area of remaining soft liner,

Table III. Short description of the experiment for shear bond strength

No. of Exp.	Condition	Group 1	Group 2	Group 3	Group 4
Exp. 1	4-META	0 %	5 %	10 %	0 %
	Aging	NO	NO	NO	NO
	Primer	NO	NO	NO	Once
Exp. 2	4-META	0%	5%	10%	0%
	Aging	2 weeks	2 weeks	2 weeks	2 weeks
	Primer	NO	NO	NO	Once

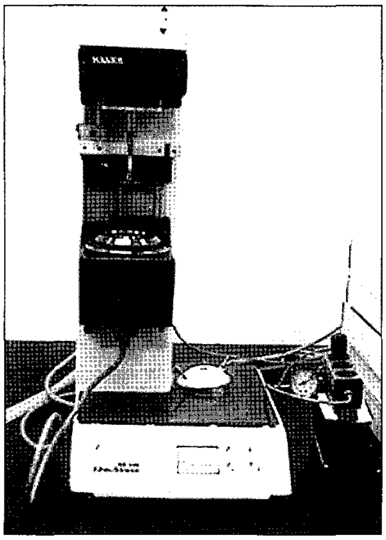

Fig. 4. Oscillating rheometer used in this study.

(2) adhesive failure--under 20% area of remaining soft liner,

(3) adhesive cohesive failure--from 20% to 80% area of remaining soft liner.

An evaluation of the gelation time of modified soft liner

To evaluate the change of the initial flow of the modified adhesive soft liner, the gelation time was measured with an oscillating rheometer (Haake RS150W/TC50, Haake Co., Germany). It was a type of stress control and parallel plate type with the diameter of 35mm(Fig. 4).

The tested specimens were prepared with same method described in the previous two experiments with the 5% and 10% of 4-META in liquid. Each group was tested ten times with same conditions and method.

The thickness of the specimen used was 2mm, and the temperature was controlled at 37℃ throughout the experiment. The frequency of oscillation was 1 rad/sec.

For the sake of reference, each group was given a preliminary testing over twelve hours for two or three times. According to the preliminary test, all specimens were keeping the state of plateau after about 3 hours. This study documented the G' value of all the specimens for 3 hours.

Statistical analysis

The resulting data were analyzed with PC-SAS(ver. 6.12) statistical soft ware. Data for apparent shear bond strengths were analyzed with the Kruskal-Wallis test(nonparametric ANOVA test) and Wilcoxon rank sum test. Overall, follow up comparisons were then carried out with the Bonferroni method and 95% confidence intervals.

RESULT

In vitro measurement of shear bond strength of the adhesive soft liner

The test results on the adhesive soft liner to determine the apparent shear bond strength and the mean values are shown in Table Ⅳ and Fig. 5. The mean shear bond strength of soft liner to the metal alloy was 5.44 N/㎟, that of adhesive soft liner containing 5% 4-META and 10% 4-META were 9.26 N/㎟, 9.62 N/㎟ each. Therefore, the mean value of the group coated with adhesive primer was 11.75 N/㎟. So, the apparent shear bond strength of group 4 was found to be the highest value among those four groups and that of group1 was the lowest value. Between the mean value of group 2 and 3, there were little difference was observed.

Group 1 and 2 showed a significant difference by a 95% confidence interval and applying the Bonferroni correction. And the results of comparison with group 1, group 3 and 4 were recorded the higher shear bond strength with the significant difference by same confidence interval. But, no significant statistical difference was found between group 2 and 3. Between group 2 and 4, group 3 and 4, there were significant difference by 95% confidence interval,

Table IV. Shear bond strength of the adhesive soft liner(N/㎟)

	Group1	Group2	Group3	Group4
1	6.18	8.80	9.73	10.16
2	6.99	8.30	6.93	11.72
3	7.85	8.83	8.48	16.09
4	4.82	8.94	8.05	9.69
5	3.87	10.20	15.82	8.20
6	6.51	10.47	9.12	9.29
7	7.52	9.12	9.71	9.76
8	5.04	8.48	9.75	18.75
9	2.58	10.45	9.48	10.55
10	3.07	9.07	9.18	13.28
Mean	5.44	9.26	9.62	11.75
S.D.	±1.85	±0.80	±2.35	±3.34

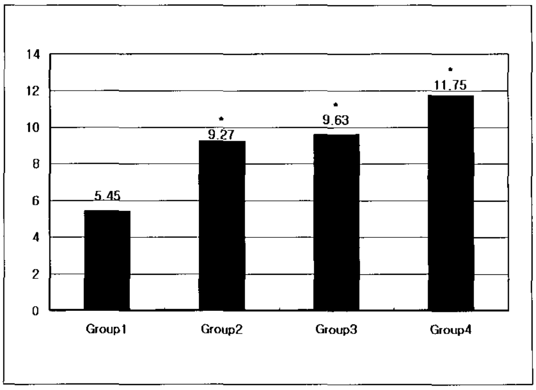

Fig. 5. Shear bond strength of the adhesive soft liner. * denotes the statistically significant increase of shear bond strength compared to that of group 1($P<0.05$).

but after Bonferroni correction, no difference was evident between groups.

In vitro measurement of the shear bond strength of the adhesive soft liner after 2 weeks of aging

The data and the mean values of apparent shear bond strength after 2 weeks aging are shown in Table V. and Fig. 6.

As shown in Table V., the mean shear bond strength of group 1 was 18.09 N/㎟, and those of group 2, 3, and 4 were 19.95 N/㎟, 24.09 N/㎟, 19.61 N/㎟ respectively.

Group 3, which contained 10% 4-META in soft liner liquid, exhibited the highest apparent shear bond strength. But, group 4 ,which was treated with Meta-Fast primer, exhibited a higher bond strength than group1 and a slight lower bond strength than group 2.

As a result of the statistical analysis, a significant difference was apparent between group 1 and 3 only.

- Observations of the debonding pattern

After the debonding test, all the specimens were divided into 3 types of debonding patterns according to the remaining liner area, as explained in the materials and methods section of this paper. The resulting debonding patterns of the 4 groups in each experiment are shown in Table VI.

In the debonding test, the specimens of group 1 exhibited 4 adhesive-cohesive failures and 6 adhesive failures, of which three were totally debonded from the adherend metal plate.

In the group 2, there were 5 adhesive-cohesive failures, 3 adhesive failures and 2 cohesive failures. In the group 3, there was only 1 adhesive failure, 4 adhesive-cohesive failures, and 5 cohesive failures. In the group 4, which was coated with primer, there were 2 adhesive failures (including one totally debonded specimen), 3 adhesive-cohesive failures and 5 cohesive failures(Fig. 7). The photographs of the debonded specimens are shown in Fig. 9~11. On observing the debonding patterns after 2 weeks

Table V. The test results of shear bond strength after 2-week aging (N/㎟)

	Group1	Group2	Group3	Group4
1	16.03	24.27	23.09	17.34
2	26.28	23.84	20.52	16.80
3	17.47	23.03	30.50	14.06
4	16.62	16.66	20.82	20.70
5	22.25	12.99	26.34	32.97
6	12.34	18.97	18.28	23.36
7	18.53	16.16	24.69	24.45
8	12.66	17.72	20.69	17.03
9	17.82	20.75	33.31	13.95
10	20.92	25.16	22.72	15.45
Mean	18.09	19.95	24.09	19.61
S.D.	±4.24	±4.09	±4.75	±5.93

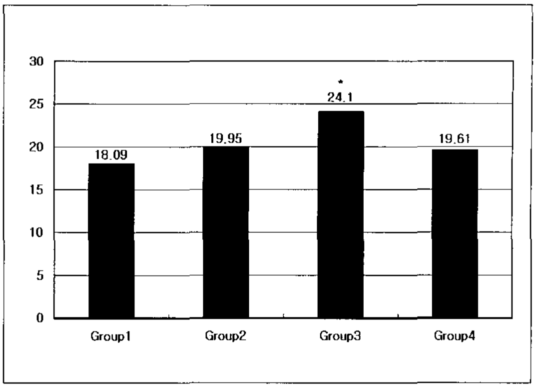

Fig. 6. Shear bond strength after 2-week aging.
* denotes the statistically significant increase of shear bond strength compared to that of group 1(P<0.05).

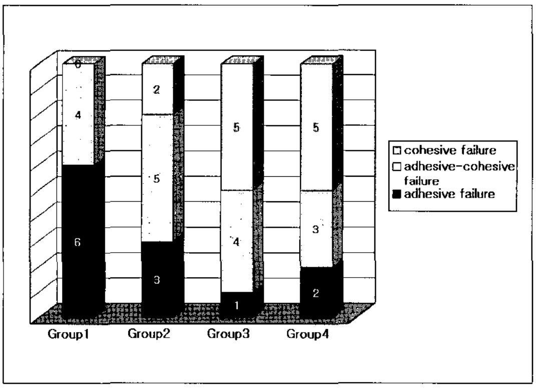

Fig. 7. Debonding patterns of the specimens.

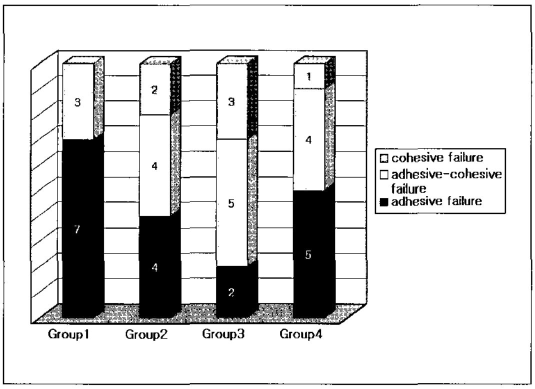

Fig. 8. Debonding patterns of 2-week aging.

Table VI. Debonding pattern in each group (the number in parenthesis indicate the number of totally debonded specimen)

	Group1	Group2	Group3	Group4	Group1 -aging	Group2 -aging	Group3 -aging	Group4 -aging
Adhesive failue	6(3)	3(1)	1	2(1)	7(5)	4(2)	2	5(2)
Adhesive-cohesive failure	4	5	4	3	3	4	5	4
Cohesive failure	0	2	5	5	0	2	3	1

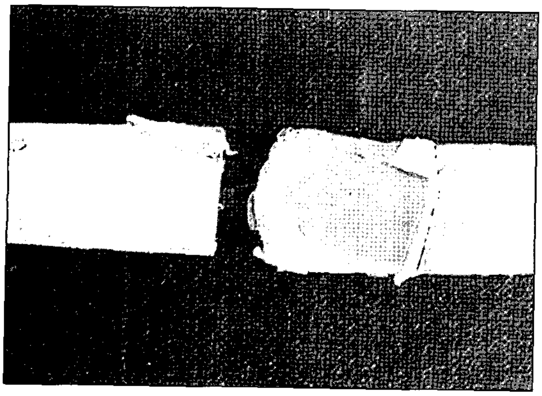

Fig. 9. Adhesive failure.

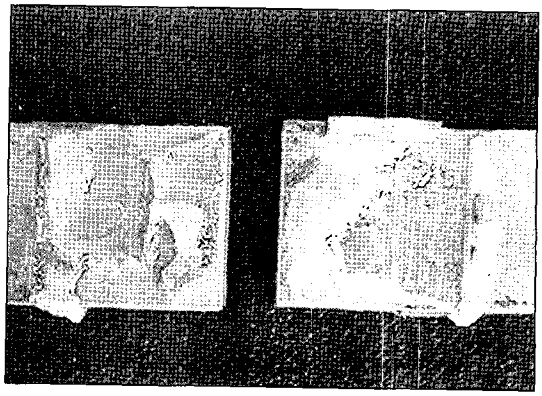

Fig. 10. Adhesive-cohesive failure.

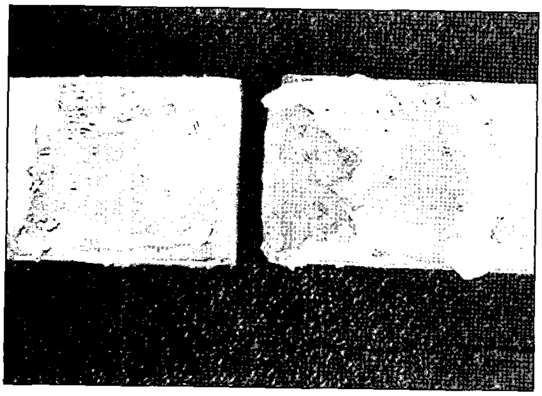

Fig. 11. Cohesive failure.

Table VII. The gelation time of modified soft liner

	Soft liner with 5% 4-META in liquid	Soft liner with 10% 4-META in liquid
1	650.5 sec.	798.9 sec.
2	568.5	718.7
3	571.1	635.6
4	682.1	600.9
5	630.4	750.8
6	651.0	711.3
7	650.0	667.3
8	602.1	681.3
9	602.5	676.5
10	601.7	680.7
Mean	560.7 sec.	692.2 sec.

of aging, group 1 was found to be comprised of the 7 adhesive failures, including 5 totally debonded specimens and 3 adhesive-cohesive failures, and there was no cohesive failure. In the group 2, there were 2 cohesive failures, 4 adhesive-cohesive failures, and 4 adhesive failures including 2 totally debonded specimens. In the group 3, there were 2 adhesive failures, 5 adhesive-cohesive failures, and 3 cohesive failures and no totally debonded specimens.

And there were 5 adhesive failures including 2 totally debonded specimens, 4 adhesive-cohesive failures and only one cohesive failure in group 4(Fig. 8).

- Evaluation of gelation time of modified soft liner

To evaluate the change of gelation time, we recorded the G' values for 3 hours. In this study, the gelation time of each test was defined as the time when the G' value reached the 75% of that of plateau. The results of these evaluation are shown in Table Ⅶ. The mean gelation time of the group with 5% 4-META and the group with 10% 4-META were 560.7 and 692.2 seconds, respectively.

DISCUSSION

The typical viscoelastic properties of the soft liner can be a very useful tool for the dentist in solving troublesome problems. With further developments in dentistry, the application of soft liner is widening. Not only is the soft liner used for relining ill- fitting dentures, but it can also be used as a temporary relining for temporary dentures during the healing phase after the first stage surgery of implant treatments, and the reinforcement of maxillofacial prostheses.

When a soft liner used for these purposes, affinity of the soft liner with metal frameworks becomes highly important. Studies have already examined the PMMA denture base resin containing 4-META and determined how the bond strength with metal frameworks using air abraded with Al_2O_3 increase with microretention.[13-15,18,21,22]

Only a few researches have been conducted on the adhesive soft liner and most of them have been studies of microretentions on the metal framework or that of application with a primer.[23] However, the microretentions may show limitations in terms of bond strength and may cause weakening of the metal framework. An primer application can improve the bond strength without weakening of the metal framework, but the primer may be easily washed away in the oral cavity by saliva.

The soft liner nowadays can be divided into 2 groups by material, i.e., silicone rubber material and acrylic resin materials.[24]

Coe-soft, the soft liner used in this study, is the PMMA soft liner.

In this study, instead of applying 4-META primer on the metal surface, the 4-META powder was incorporated to the soft liner similar to the 4-META containing hard relining resin.

4-META is synthesized adhesion-promoting chemicals developed by Tanaka et al.[9] This material can easily adhere to the oxide layer of base metal alloys such as chromium metal at room temperature and have been proved not to cause redness or inflammation to skin.[25]

In this study, the 4-META powder was incorporated into the soft liner liquid instead of powder to prevent reaction between the 4-META and the initiator in the powder before gelation. Coe soft is an acrylic resin tissue conditioner type liner and undergoes a typical gelling reaction.[26] The basic polymer used is poly(ethylmethacrylate) and the liquid consist of a mixture of plasticizers and ethyl alcohol, without methacrylate monomer.[27-29]

The test method of bond strength used in this study was designed by a modification of ASTM D1002-99. This method was designed to evaluate the only the apparent shear bond strength, The estimated values obtained from this experiment were not the absolute values but were primarily comparative.[30] In the test of shear bond strength, the group 4 showed the highest mean bond strength and the group 2, 3 which containing 4-META showed higher values than the control group and the group 4 showed primarily cohesive debonding pattern unlike the group with no treatment, which showed the adhesive failure in large.

But the test results of shear bond strength after 2 weeks of storage in a 37℃ water bath were somewhat different from those of the previous experiment. While in the previous experiment the bond strength of the primer coated group was the highest, the group containing 10% 4-META after a 2-week aging period showed the highest values.

No significant difference was found among the control group, the group with 5% 4-META and the group with primer. It is considered that the primer coating on the adherend metal plate more easily washed away or dissolved in the wet oral cavity than the 4-META-metal oxide layer on the adherend metal specimens.[31]

Moreover after 2 weeks of aging, the mean shear bond strength of control group and that of the group with primer showed no significant difference. So, it can be suggested that the effect of primer coating on the metal framework is nearly of no use in the oral cavity at least 2 weeks afterward. When the data between the two experiments are compared the groups with 2 weeks aging exhibited higher values than the non-aged groups. This can be explained by the fact that the shear bond strength is dependant upon specimen geometry, loading configurations or material stiffness.[32] In this study, the design of specimens and the testing method were controlled in the same condition in the two experiments. Therefore, the higher bond strength in the experiment after 2 weeks aging should be caused by the increased stiffness of soft liner.

However, in the debonding pattern observation, the aged specimens produced more adhesive debonding and totally debonding patterns than the non-aged specimens.

That is to say, the increased value after aging does not rely on the increased bond strength, but the increased stiffness of soft liner itself between the metal plates.

The softness of soft liner was achieved by lowering the magic temperature called the glass transition temperature(Tg) using the plasticizer in liquid.[24] The polymer used in the soft liner has the properties of remaining rigid below this temperature, just like a denture base resin, but becoming rubbery above it. That is to say, the Tg of soft liner is below the mouth or room temperature.

The acrylic resin soft liners that contain plasticizer are not stable in an aqueous solution such as the oral cavity and the plasticizer are slowly leaked out into the saliva.[33,34]

According to the amount of the leaked plasticizer, the water is absorbed in the soft liner and the more plasticizer leakage, the stiffer the soft liner becomes. In the patients mouth, the relining material is clinically exposed to condition more closely related to the shear and tear test.[34]

So the shear test is considered an appropriate method for testing the bond strength of resilient lining materials.[35] But, it is worth considering the effect of the test method has on the bond strength on measured strength.

There can be 3 types of test method for evaluating the bond strength of soft lining materials; the peel test, shear test, tensile test. In the peel test, the stress is limited to a line at the edge of the joint contrary to the fact that in the tensile and shear tests the whole cross sectional area of bond surface is under stress.[36] In shear testing, the stress are unevenly distributed with much greater stress at the edge. Anderson et al.[37] reported this effect and suggested that these stress concentration increased as the thickness of liner increased.

Theoretically useful information could be obtained by subjecting the specimen to a deformation rate equal to the speed of chewing. However, the chewing cycle is so fast that test specimens could be subjected to an impact. Laboratory testing doesn' t need to represent the load that the soft liner can withstand clinically because in the lab test only one type of force is applied at a time.[38,39]

However, clinically, dentures are subjected to various masticatory forces. For these reason, it is difficult to interpret the laboratory bond strength. But still the laboratory data could be very meaningful in comparing and ranking the bond strength of lining material itself. This study, for example, is very useful for comparing the effect of 4-META and its primer.

In the observation of debonding pattern, we used the calculated % area by grid. In the several studies of the soft liner, the debonding pattern was classified using a few criteria. For example, they are classified as 100% adhesive, 100% cohesive and mixed, or as in the soft material, on the surface of the soft material, in the adhesive junction.[40]

However, such classifications are very observer dependant due to the absence of objective criteria. In the present study, this ambiguity was minimized by using the grid based the calculated values.

According to test results, the group with 10% 4-META exhibited mainly the cohesive failure in large. But the group with primer exhibited contrary result between the test at the preparation of specimens and after 2 weeks of aging.

When tested shortly after preparation, cohesive failures appeared predominantly. But after 2 weeks only one specimens failed cohesively. From these experiment, it could be suggested that 4-META binds the metal oxide layer on the adherent and increase the bond strength of the soft liner to the Co-Cr alloy. After aging, the effect of the added 4-META can be longer sustained than that of coated primer.

To evaluate the viscoelastic properties of soft liners modified by 4-META, the author chose the test method employing the rheometer to estimate the gelation time. There can be several other important properties of soft liner such as creep behavior, consistency, and initial flow and so on. Such a property can determine the specific viscoelasticity of each commercial soft liner.

Among them, the initial flow of the material determines the working time and adaptation between the oral mucosa and the denture surface. However, there is no international standard for soft liner except the ISO 10139-1 which determine the initial flow as the consistency test of these materials.[41]

Yoeli et al.[42] tested the initial flow of several soft liners according to the ISO specification and reported that Coe-soft was very close to the ISO specification.

But it was the results obtained from only consistency test under static conditions. There are a few test method to evaluate the viscoelastic properties of polymeric materials such as creep test, stress relaxation test and the dynamic test.

Creep test is the test of measuring the strain at the specific load and the most commonly used and most simple method of viscoelastic properties.[41,43]

Another is the stress relaxation test, which measures the stress necessary to maintain a specific level of the strain as a function of time after rapid deformation of a given volume of specimen.[44,45]

And the dynamic rheologic test measures the behavior of material when subjected to the sinusoidal or cyclic stress. It has the advantage of being able to measure material characteristics in short time accurately. And that material behavior in longer or shorter period can be predicted than the given period at test.

So in this study, the initial flow of the modified soft liner was evaluated by measuring the gelation time with an oscillating rheometer.

The initial flow was estimated by McCarthy and Moser[46] by parallel-plate plastometer in 1978. Before that, Braden[47,48] had measured the initial flow by cone and plate viscometer in 1970. And using reciprocating rheometer, Jones[49] reported the initial flow of soft liner in 1986. Murata et al.[50,51] reported the gelation time of several tissue conditioners by oscillating rheometer in 1998 and 1993.

In these study, the gelation times of the soft liner were between 3 and 15 minutes.

An oscillating rheometer was used to measure the gelation time of the two modified soft liners. Using rheometer, the test specimens were kept at 37°C during the whole experiment at both the internal and external layer throughout the 2mm thickness.

By doing this, the disadvantages of creep test which cannot maintain a constant temperature during whole experiment cannot mimic the thickness of soft liner in denture, were compensate for.[52]

In the present study, the G' was measured as a parameter for gelation time.

G' is the one part of the viscosity(η). Viscosity can be divided into 2 properties of G' and G''. G' is the part of storage modulus and account for elasticity and G'' account for the loss modulus with viscosity. That is to say, G' means the storage energy by elasticity without loss.

$$|\eta^*| = (\eta'^2 + \eta''^2)^{1/2} = [(G''/w')^2 + (G'/w)^2]^{1/2} = |G^*|/w$$

According to the Table Ⅶ, the mean gelation times of modified soft liners were 9.3 minutes and 11.5 minutes respectively for the 5% 4-META and 10% 4-META in liquid. Accordingly, while the modified soft liners appear to have a somewhat longer gelation time, but they were still within the range of 3 to 15 minutes shown by other researches.[53]

Because of these long gelation times, more time is need when removing them from the mouth to reline the denture.[38]

The longer gelation time of the modified soft liner could be considered as a result of adding 4-META in liquid.

There are several factor affecting the gelation time of soft liners; ethanol content, particle size, molecular weight and plasticizers and powder/liquid ratio.[54,55]

The 4-META particle in liquid should behave as a retarder of polymer entanglement in the liquid. So additional chemical analysis would be necessary to explain the detailed cause of longer gelation times.

By the way, the results of this study suggest that it is useful for clinicians who intend to reline the metal based denture with PMMA soft liner to incorporate the 4-META in soft liner. And these modified adhesive liner should fulfill its shock absorption task for the longer duration in oral cavity with an acceptable initial flow.

CONCLUSION

The present study measure the apparent shear bond strength of modified soft liner to the Co-Cr alloy by tensile loading and gelation time through the oscillating rheometer.

Within the conditions and limitations of this study, the following conclusions were drawn.

1. There was significant bond strength increase in the 5% 4-META, 10% 4-META containing groups and in the primer coated groups versus the control group(P<0.05).
2. After 2 weeks of aging, no significant increase in bond strength was found except for the group containing 10% 4-META (P<0.05).
3. The gelation times of the modified soft liner were 9.3 minutes for a 5% 4-META containing liner and 11.5 minutes for a 10% 4-META liner.
4. The debonding patterns of the 4-META containing group after 2 weeks of aging were similar to those of immediately after preparation, but the debonding pattern of the primer group showed more adhesive failure after 2 weeks of aging.

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