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Adhesive strength and its improvement referring to the laminated-type mouthguard

Takeda T, Ishigami K, Kawamura S, Nakajima K, Shimada A, Sumii T, Swain M. Adhesive strength and its improvement referring to the laminated-type mouthguard. © Blackwell Munksgaard, 2006.

Abstract – The manufacture of laminated-type mouthguards requires skill in fusing sheets of mouthguard materials together. Adequate adhesive strength is required to use mouthguards in a stable condition for a long time. Therefore, in this study, the exfoliation test was applied and some treating techniques and conditions that improve the adhesive strength on a laminated surface were examined. Samples were laminated with two pieces of mouthguard material (3 mm thickness) having an adhesive area of 5×5 mm^2, and whose other end was the holding part. The experimental factors used were as follows: heating time, use of solvent, elimination and direct heating of the laminate surface, colour of materials and water sorption. The result was measured at the time of breakage of the maximum load (N) and the form of destruction was examined. At 165 s of heating time, material failure was shown at under a load exceeding 5.0 N when compared to an untreated condition. Material failure was measured when a solvent was used and during the elimination of the laminated surface at a heating time of 150 s, which is 15 s lesser than in an untreated condition. Material failure was also measured by direct heating on the bonding surface of a second sheet of material at a heating time of 135 s, which is 30 s lesser than in an untreated condition. The differences in colour of the materials influence adhesion. Clear and light coloured materials showed higher adhesion ability. One-way analysis of variance confirmed a statistically significant difference in heating time differences, usage of solvent, elimination, direct heating on bonding surface and colour ($P < 0.05$). The decrease of adhesive strength by water sorption at 23° and 37°C was not observed significantly. Maximal laminated bond strength can be obtained by minimal heating time and proper treatment with the use of solvent, elimination and direct heating on bonding surface. The differences in the colour of the materials influenced adhesion. Clear and light coloured materials showed higher adhesive ability. Water sorption did not affect the adhesive strength. Therefore, if laminated-type mouthguards were manufactured properly, it can be used for a longer time and in a good condition.

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Key words: adhesive strength; adhesive strength improvement; laminated-type mouthguards; delamination test

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It is desirable to use an appropriate mouthguard that has maximal effect in preventing injuries in the orofacial area, especially in contact sports. Today, the use of mouthguards is increasing. Most players use boil-and-bite-type mouthguards because they are cheap and easily available. However, they are defective with regard to fit and occlusion, so they interfere with pronunciation, cause discomfort, decrease preventive ability, etc. (1, 2). Therefore, it is strongly desirable to use a mouthguard that is custom-made. Even with the custom-made type, a protective effect cannot be expected unless adequate thickness is ensured in the labial surface of the front teeth (3, 4) and on the maxilla, which is frequently injured by direct impact. Hence, the shock absorption ability is proportional to the thickness of the mouthguard (5–12). It is also necessary to maintain adequate thickness on the occlusal surface to establish suitable occlusion (13) and protect from an impact force applied on the mandibule (14–18). Nowadays, vacuumed-type mouthguards are mostly used. In this type, it has been reported that the entire thickness decreases because of heating and vacuuming and that it becomes stronger as the angle of the model surface becomes steeper (19). Therefore, it is difficult for this type of mouthguard to secure the adequate thickness required to demonstrate the ability to absorb impact forces after it has been manufactured.

On the other hand, laminated-type mouthguards have higher shock absorption ability as they are fused with another sheet of material, which restrains the whole thickness but provides adequate thickness to the necessary part where dental injuries often occur. Hence, the application of laminated-type mouthguards (2, 20–23) is considered to be necessary from the standpoint of safety and comfort.

However, there are no clinical reports of problems with defective adhesion during manufacture and delamination of the adhesive surface when the mouthguard is worn. The fabrication method of the laminated-type mouthguard should be improved. Therefore, some reliable methods to reduce failures are necessary. Furthermore, it is necessary to control the thickness of the mouthguard after fabrication by reducing the heating time.

Using a mouthguard material of the EVA type, this study examined not only adhesive strength on the laminated surface but also some methods of promoting adhesive ability by means of a delamination test.

Materials and methods

Materials

In the manufacture of samples, mouthguard materials of Drufosoft (Dreve-Dentamid GMBH, Unna, Germany) and an air pressure machine of Drufomat (Type SO, Dreve-Dentamid, Unna, Germany) were used.

Manufacture of samples

Two pieces of mouthguard materials (3 mm thickness) were laminated together with an adhesive area of 5×5 mm^2 and with one end used for holding it (Figs 1 and 2). Before lamination, 10-mm-width tape was pasted on the surface of the first material to cover the holding part. Later, it was cut with a heated knife, and the process completed with a bar and a disc. Three laminated samples were manufactured under each condition and two specimens were cut from each sample. Five samples were tested and the one remaining was assumed to be a spare. We took up six factors that influence adhesion: heating time, the use of solvent in the laminate surface, elimination of the laminate surface, direct heating of the second material in the laminate surface, the colour of the material and water sorption. In the untreated condition, the heating times used were 120, 135, 150, 165 and 180 s. Before lamination, chloroform, as a solvent, was applied to the laminate surface with a writing brush. Ultrafilm-soft was used to eliminate the adhesive surface. As for direct heating of the

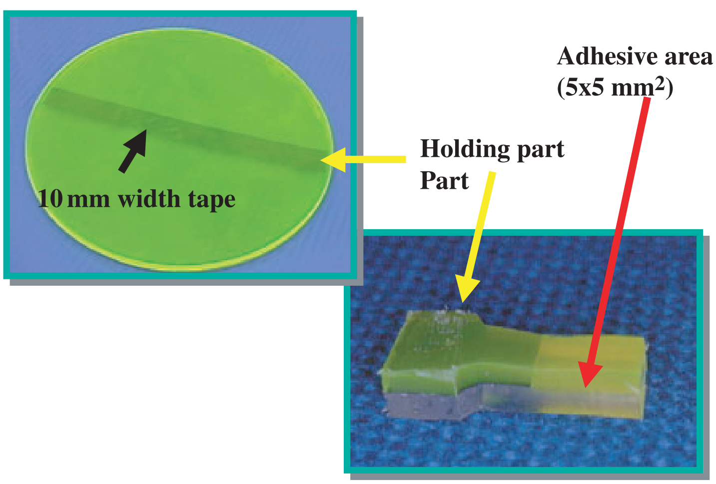

Fig. 1. Specimens were laminated with two pieces of 3-mm thick mouthguard material together with an adhesive area of 5×5 mm^2 and with one end as the holding part.

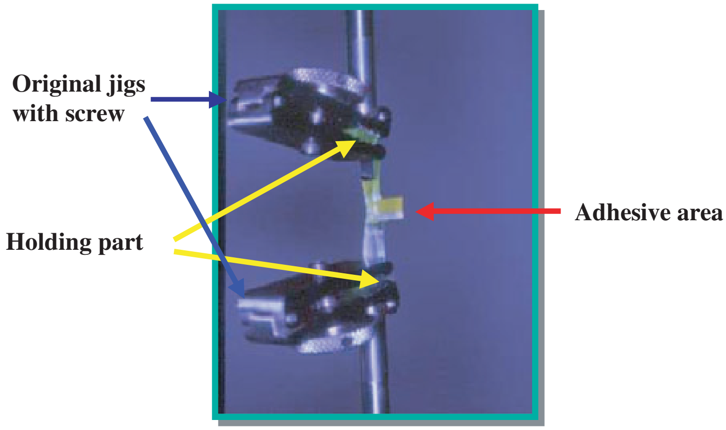

Fig. 2. An autograph (Shimazu Company, AG-50K NE) for delamination test employed at a cross-head speed of 40 mm/min and with a loading cell of 500 kg force.

laminate surface, after heating the laminate surface of the second material directly for 60 s, the material was reversed, maintaining heat and pressure. The influence of colour was examined as to whether it was clear, neon-yellow, neon-green, white and black. The influence of water sorption was examined by facilitating water sorption at 23 and 37°C for a month. The colour and water sorption parameters were examined at a heating time of 165 s, which showed good adhesive ability after considering the influence of heating time.

Examination methods

The delamination test (Fig. 2) was carried out by employing Autograph (AG-50K NE, Shimazu Corporation, Japan) at a cross-head speed of 40 mm/min and with a loading cell of 500 kg force. Original jigs with screw were used to grip the specimen firmly. The holding part was opened and gripped with this jig, and the specimen was pulled with the Autograph up and down until the specimen showed an interface or material failure. All tests were conducted in an air-conditioned room at 25°C.

Analysis methods

The results were measured at the time of breakage of the maximum load (N) and the form of destruction of the broken surface was examined with the naked eye.

By observing the delaminated surface, it was inferred that the interface failure (the left-hand side of Fig. 3) separation on the adhesive surface or the material failure (the right-hand side of Fig. 3) was not on the adhesive surface but on the mouthguard material (because adhesive strength exceeds the strength of the mouthguard material).

Statistical comparisons were made using a one-way analysis of variance (ANOVA) test followed by Tukey multiple-comparison tests for further comparisons between sensors and impact objects ($P < 0.05$). A Student's *t*-test for only direct heating was carried out using SPSS® (SPSS Japan Inc., Tokyo).

Results

The influence of heating time demonstrated interface failures (white column in the graph) under a load of 1.46 N at 120 s; interface failures were also seen at 130 and 150 s. As the heating time prolonged, adhesive strength increased linearly, and material failures (grey column) were seen under a load of 5.40 N in 165 s (Fig. 4). One-way ANOVA confirmed a statistically significant difference among the four heating times ($P < 0.05$) (Table 1). A

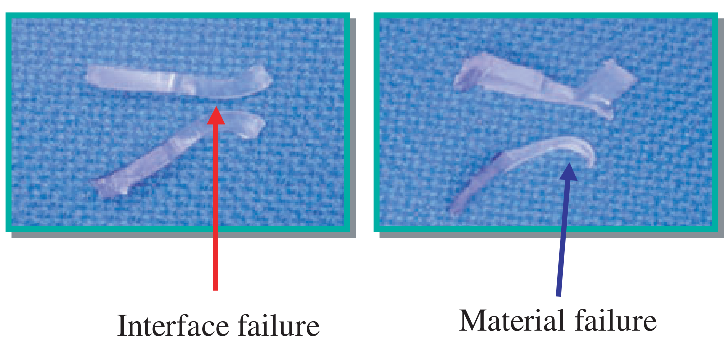

Fig. 3. Examination of the broken surface to judged whether it was due to interface failure (left) or due to material failure (right).

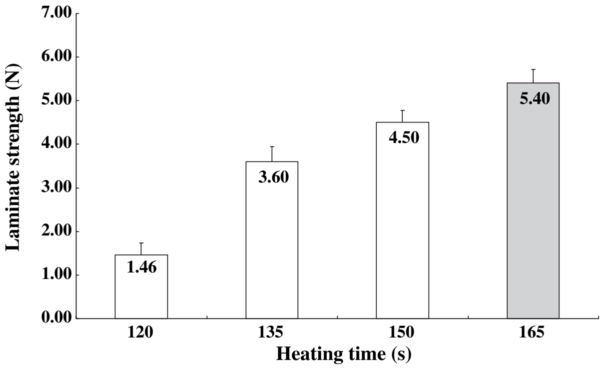

Fig. 4. The influence of heating time on interface failures (white columns) under a load of 1.46 N at 120 s, after which interface failures are shown until 150 s. As the heating time prolongs, adhesive strength increases appropriately, and material failures (grey column) are shown under a load of 5.40 N at 165 s.

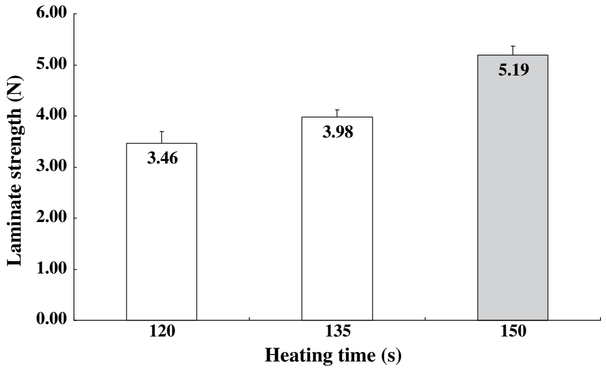

Fig. 5. The influence of solvent on interface failures are shown at 120 and 135 s with increasing values, and material failures are shown under a load of more than 5.20 N at 150 s.

significant difference was found between each heating time (Tukey test) (Table 1).

The influence of solvent usage and elimination of the laminating surface showed almost the same results. Interface failures were seen at 120 and 135 s with the increasing values; material failures were seen under a load of 5.20 N at 150 s (Figs 5 and 6). One-way ANOVA confirmed a statistically significant difference in both the usage of solvent and elimination of laminating surface (Tables 2 and 3). A significant difference was found between each heating time (Tukey test) (Tables 2 and 3). The influence of direct heating showed that interface failures were seen at 120 s. Material failure was seen under a load of 5.22 N at 135 s (Fig 7). Student's *t*-test confirmed a statistically significant difference in the effect of direct heating (Table 4). The difference of colour influenced results. Clear, neon-yellow and neon-green colours indicated

Table 1. One-way analysis of variance confirmed a statistically significant difference between the four heating times ($P < 0.05$), and a significant difference was admitted between each heating time (Tukey test)

Heating time: ANOVA

Strength

	Sum of squares	df	Mean square	F	Sig.
Between groups	42.85	3	14.28	156.868	0.000
Within groups	1.46	16	0.09		
Total	44.30	19			

Heating time: Multiple comparisons
Dependent variable: Strength
Tukey HSD

Heating time (I)	Heating time (J)	Mean difference (I − J)	Std. error	Sig.	95% Confidence interval Lower bound	95% Confidence interval Upper bound
120	135	−2.14*	0.19	0.000	−2.690	−1.598
	150	−3.04*	0.19	0.000	−3.590	−2.498
	165	−3.94*	0.19	0.000	−4.490	−3.398
135	120	2.14*	0.19	0.000	1.598	2.690
	150	−0.90*	0.19	0.001	−1.446	−0.354
	165	−1.80*	0.19	0.000	−2.346	−1.254
150	120	3.04*	0.19	0.000	2.498	3.590
	135	0.90*	0.19	0.001	0.354	1.446
	165	−0.90*	0.19	0.001	−1.446	−0.354
165	120	3.94*	0.19	0.000	3.398	4.490
	135	1.80*	0.19	0.000	1.254	2.346
	150	0.90*	0.19	0.001	0.354	1.446

*The mean difference is significant at the 0.050 level.

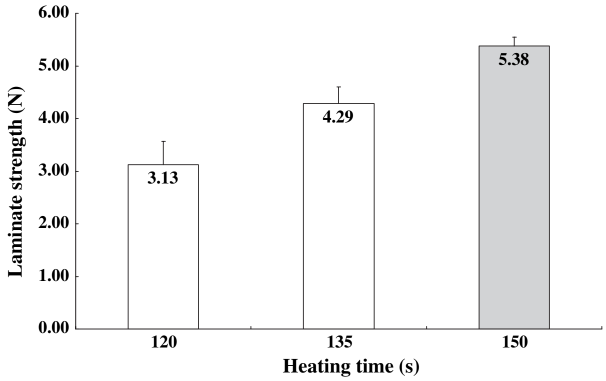

Fig. 6. The influence of elimination on interface failures are shown at 120 and 135 s with the increasing values, and material failures are shown under a load more than 5.20 N at 150 s.

material failure under a load of more than 5.30 N at 165 s when heated in the untreated condition, in comparison with the colour of black and white, which indicated interface failure under a load of less than 4.30 N (Fig. 8). One-way ANOVA confirmed a statistically significant difference between colour differences (Table 5). A significant difference was found between clear, neon-yellow, neon-green, black and white (Tukey test) (Table 5). As for water sorption, the lowering of adhesive ability on water sorption at both 23° and 37°C was not observed (Fig. 9). A statistically significant difference was not observed with regard to water sorption (Table 6).

Discussion

Laminated-type mouthguard

Dorney, Padilla and co-workers (2, 20, 21) used Drufomat in manufacturing laminated-type mouthguards, which facilitated lamination by using high pressure (6 atm). They stated that its operation and conformity were excellent, and also mentioned that its loss could be prevented by including information such as player's name and telephone number, team logo, favourite sticker, etc.. Padilla and co-workers (21), when comparing the pressure-laminated type with the vacuumed type, indicated that the pressure-laminated type was more uniform and could maintain better the thickness of the mouthguard to preventing injury. Jagger and co-workers (22) reported the manufacture of a mouthguard of the bi-maxillary type. They mentioned that if the pressure-laminated method was applied, it was possible for the bi-maxillary-type mouthguard to adhere more strongly and easily than the vacuum-formed one. In Japan, Takeda and co-workers (23) reported a fundamental way of manufacturing an improved laminated-type mouthguard. Generally, mouthguard materials should have adequate strength, and it is desirable that a mouthguard withstand the hard force of occlusion, even when used over long periods of time. Chaconas and co-workers (24) reported the following: as a result of measuring the change in the thickness of a mouthguard before and after use, all mouthguards were

Table 2. One-way analysis of variance confirmed a statistically significant difference in both usage of solvent, and a significant difference was admitted between each heating time

Solvent: ANOVA

Strength

	Sum of squares	df	Mean square	*F*	Sig.
Between groups	7.94	2	3.97	111.867	0.000
Within groups	0.43	12	0.04		
Total	8.37	14			

Solvent: Multiple comparisons
Dependent variable: Strength
Tukey HSD

Heating time (I)	Heating time (J)	Mean difference (I – J)	Std. error	Sig.	95% Confidence interval Lower bound	95% Confidence interval Upper bound
120	135	−0.52*	0.12	0.003	−0.836	−0.200
	150	−1.74*	0.12	0.000	−2.054	−1.418
135	120	0.52*	0.12	0.003	0.200	0.836
	150	−1.22*	0.12	0.000	−1.536	−0.900
150	120	1.74*	0.12	0.000	1.418	2.054
	135	1.22*	0.12	0.000	0.900	1.536

*The mean difference is significant at the 0.050 level.

Table 3. One-way analysis of variance confirmed a statistically significant difference in elimination, and a significant difference was admitted between each heating time

Elimination duration: ANOVA

Strength

	Sum of squares	df	Mean square	F	Sig.
Between groups	12.70	2	6.35	59.247	0.000
Within groups	1.29	12	0.11		
Total	13.99	14			

Elimination duration: Multiple comparisons
Dependent variable: Strength
Tukey HSD

					95% Confidence interval	
Heating time (I)	Heating time (J)	Mean difference (I − J)	Std. error	Sig.	Lower bound	Upper bound
120	135	−1.16*	0.21	0.000	−1.712	−0.608
	150	−2.25*	0.21	0.000	−2.806	−1.702
135	120	1.16*	0.21	0.000	0.608	1.712
	150	−1.09*	0.21	0.001	−1.646	−0.542
150	120	2.25*	0.21	0.000	1.702	2.806
	135	1.09*	0.21	0.001	0.542	1.646

*The mean difference is significant at the 0.050 level.

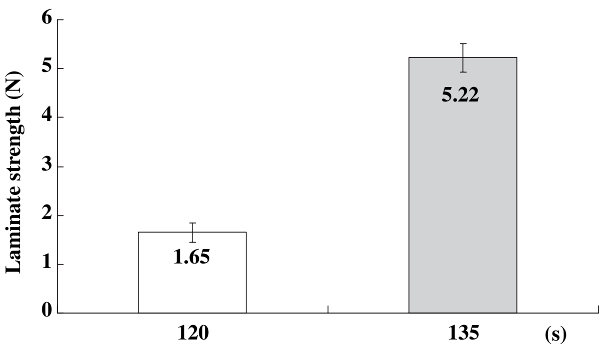

Fig. 7. The influence of direct heating on interface failures are shown at 120 s, and material failures are shown under a load of more that 5.22 N at 135 s.

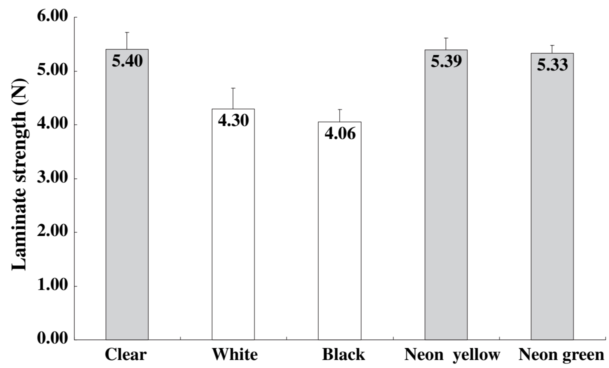

Fig. 8. The influence of differences in colour. The colours of clear, neon-yellow and neon-green showed material failure under a load of more than 5.30 N at 165 s heating in the untreated condition, in comparison with the colour of black and white, which showed interface failure at a small value under a load of 4.30 N.

Table 4. The Student's t-test confirmed a statistically significant difference in the effect of direct heating

Direct heating of laminating surface; Dependent variables: Strength

					95% Confidence interval	
Heating time (I)	Heating time (J)	Mean difference (I − J)	Std. error	Sig	Lower bound	Upper bound
120	135	34.3615*	5.44335	0.000	22.3868	46.3482

recognized to decrease in thickness, but the decrease in thickness varied depending on the materials used. The decrease was marginal in hard and soft pre-laminated materials other than urethane, and a single layer of EVA. It could be suggested that the laminated-type mouthguard of sufficient thickness was the most suitable of all mouthguards. The laminate-type mouthguards are manufactured by fusing some sheets of mouthguard materials together. Sometimes they are damaged on the adhesive surface by saliva, occlusal force, change of temperature, etc. Therefore, it is desirable that laminate-type mouthguards are fused with sufficient strength during manufacture. But the laminated strength of the mouthguard systems of the laminated types has not been measured. If a necessary step is neglected even though these systems are used, a valid result with not be achieved. The purpose of this study was to measure the adhesive strength in

Table 5. One-way analysis of variance confirmed a statistically significant difference between colour differences, and a significant difference was admitted between clear, neon-yellow, neon-green and black and white (Tukey test)

Color: ANOVA

Strength

	Sum of squares	df	Mean square	F	Sig.
Between groups	8.80	4	2.20	29.855	0.000
Within groups	1.47	20	0.07		
Total	10.28	24			

Color: Multiple comparisons
Dependent variable: Strength
Tukey HSD

					95% Confidence interval	
Color (I)	Color (J)	Mean difference (I − J)	Std. error	Sig.	Lower bound	Upper bound
Clear	White	1.11*	0.17	0.000	0.592	1.620
	Black	1.35*	0.17	0.000	0.834	1.862
	Neon yellow	0.01	0.17	1.000	−0.502	0.526
	Neon green	0.07	0.17	0.994	−0.446	0.582
White	Clear	−1.11*	0.17	0.000	−1.620	−0.592
	Black	0.24	0.17	0.629	−0.272	0.756
	Neon yellow	−1.09*	0.17	0.000	−1.608	−0.580
	Neon green	−1.04*	0.17	0.000	−1.552	−0.524
Black	Clear	−1.35*	0.17	0.000	−1.862	−0.834
	White	−0.24	0.17	0.629	−0.756	0.272
	Neon yellow	−1.34*	0.17	0.000	−1.850	−0.822
	Neon green	−1.28*	0.17	0.000	−1.794	−0.766
Neon yellow	Clear	−0.012	0.17	1.000	−0.526	0.502
	White	1.094*	0.17	0.000	0.580	1.608
	Black	1.336*	0.17	0.000	0.822	1.850
	Neon green	0.056	0.17	0.997	−0.458	0.570
Neon green	Clear	−0.068	0.17	0.994	−0.582	0.446
	White	1.038*	0.17	0.000	0.524	1.552
	Black	1.28*	0.17	0.000	0.766	1.794
	Neon yellow	−0.056	0.17	0.997	−0.570	0.458

*The mean difference is significant at the 0.050 level.

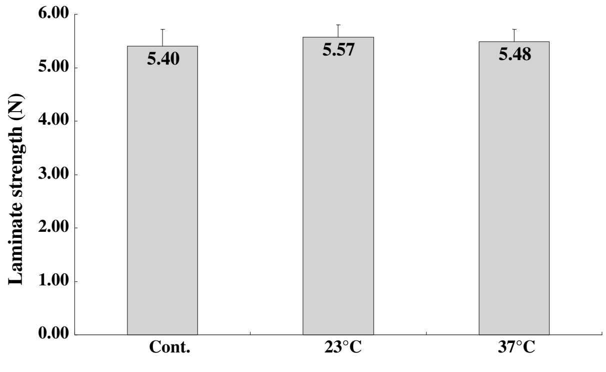

Fig. 9. The influence of water sorption. The lowering of adhesive ability on water sorption at 23° and 37°C was not observed.

the delamination test to determine the factors that influence adhesive strength and find some methods for promoting adhesive ability. The findings of this study will have important implications for establishing reliable methods that can secure long-term lamination.

Method of experiment

Examination method

Although it may seem difficult to measure adhesive strength because mouthguard materials are soft and stretchable, the delamination test was used to measure the adhesive strength between soft plastics and rubbers (25). In the present study this method was applied. Materials were manufactured to laminate two pieces of mouthguard materials with an adhesive area of 5×5 mm^2, with one end used as the holding part to measure the constant area. An original jig with screw was made to grip the specimen firmly.

The factors that influences adhesion

There are three methods for adhering plastics together: adhesion by heating, adhesion by solvent (adhesion by melting the surface) and adhesion by bonding agent (26). Adhesion by heating and adhesion by solvent are applied to thermoplastics, which are softened by heating. Adhesion by a

Table 6. A statistically significant difference was not observed in water sorption

Water sorption: ANOVA

Strength

	Sum of squares	df	Mean square	F	Sig.
Between groups	0.07	2	0.04	0.533	0.600
Within groups	0.81	12	0.07		
Total	0.89	14			

Water sorption: Multiple comparisons
Dependent variable: Strength
Tukey HSD

					95% Confidence interval	
Water sorption (I)	Water sorption (J)	Mean difference (I − J)	Std. error	Sig.	Lower bound	Upper bound
Cont.	23°C	−0.17	0.16	0.572	−0.610	0.270
	37°C	−0.08	0.16	0.879	−0.520	0.360
23°C	Cont.	0.17	0.16	0.572	−0.270	0.610
	37°C	0.09	0.16	0.850	−0.350	0.530
37°C	Cont.	0.08	0.16	0.879	−0.360	0.520
	23°C	−0.09	0.16	0.850	−0.530	0.350

*The mean difference is significant at the 0.050 level.

bonding agent is applied to thermosetting plastics that are mainly hardened by continuous heating. Adhesion by heating is the method by which the material is softened and pressed. Therefore, many contributing factors or techniques affect the adhesion. This mechanism is performed by the system of Drufomat, primarily, and by other methods in the manufacture of laminated-type mouthguards. Thus, many factors also seem to influence adhesion of mouthguard materials (27). In this study, we took up heating time as the chief effect to adhesive ability; as for surface treatment we chose solvent application, elimination and direct heating of adhesive surface. Colour of materials and the influences of water sorption were considered as clinical matters. Considering the manufacturer's heating time of 135 s, we used heating times of 120, 135, 150 and 165 s for the adhesive side in the untreated condition. The influence of chloroform as solvent and the elimination were measured by dissolving and removing the effect of impurities from the surface. Direct heating was targeted to improve a wetting of the bonding surface. The influence of colour was discussed as clear, semi-transparent as neon-yellow and neon-green and opaque as white and black. The influence of water sorption was examined at about 23° and 37°C for a month to maintain room temperature and the temperature inside the mouth when in use.

Analysis method

The results were recorded at the time of breakage of the maximum load *N*. The material failure was judged as having the proper laminating strength, assuming the laminate strength to exceed the strength of the material. Generally, the process of material failure occurs if the strength of the adherence material is smaller than the bonding strength. Conversely, if the bonding strength is greater than the strength of the adherence material, a breakdown occurs in the bonding material internally or at the interface (26, 27). However, no bonding agent was used in this study, and only interface failures occurred in cases where the laminate strength was larger than the strength of the mouthguard material. It was good lamination when the material failure occurred first.

Results

The influence of heating time showed that interface failure occurred below a load of 5 N until 150 s, but material failure was shown at 165 s, which proved to be a significantly high laminate strength. The mouthguard material of EVA employed in this study was found to have enough adhesive ability when the heating time was considered sufficient. But good adhesion needed about 50 s longer heating time according the manufacturer's instruction. This increase in heating time seems to be attributed to the fact that the heating coil needs more time to reach the necessary melting temperature. Therefore, in order to heat at a fixed time, it seems necessary to warm the machine before softening the material.

Anyway, the thickness of materials is inversely related to the increase of heating time (19). In this study, longer heating time was required in order to increase adhesive strength. Hence, it is necessary to examine the factors that will promote adhesive strength in a short heating time to allow enough thickness for shock absorption in the mouthguard material after manufacturing. Thus, five factors that were previously mentioned were examined. The use of solvent could yield adequate adhesive strength by using a short heating time. It was suggested that applying the solvent promoted adhesive ability because the solvent is applied and immediately volatilized, and at the same time plastic surfaces are softened and mixed with each other and then fused strongly by heating and pressurizing. Moreover, it can be suggested that elimination of the adhesive surface promotes adhesive strength significantly by removing the oil and other foreign substance on the surface and by making the surface rough. As dirt or contaminants on the surface interfere in bonding, it is necessary to remove them because rough surfaces can increase fusing strengths.

Direct heating of the laminating surface literally softened the bonding surface of the materials which can improve surface wetting and laminate strength significantly. Though this method is not used in a conventional production method, it seems to be extremely effective clinically. The differences in the colour of materials also influenced adhesion significantly. Though both clear and semi-transparent (neon-yellow and neon-green) showed high adhesive ability, opaque (white and black) showed low adhesive ability. It is obvious that clear and semi-transparent EVA have high adhesive abilities because the amounts of pigments used were different; another reason is the difference of a pigment itself that is used as colouring material. At any rate, the colour of the mouthguard material influences not only the operation but also adhesive ability; therefore, during treatment before lamination, the heating time should be controlled carefully for each colour. If the material consists of several colours, more attention is required, because it could affect softening temperature and the adhesive ability of different colours. At 37°C and at room temperature continuous water sorption for a month does not influence adhesive strength significantly and it could be suggested that the use of tightly laminated mouthguard maintains good adhesive ability over the long term.

When using a system that employs tools and materials aimed at laminating by high temperature and high pressure, attention should be given to heating time, laminating surface conditions, etc., so that the laminated-type mouthguard obtains good adhesive ability.

Conclusion

The delamination test was used to examine adhesive strength on the laminated surface as a factor in measuring mouthguards. In addition, a method of promoting adhesive ability was also studied.

As a result, the influence of heating time was that under the condition of untreated surface the interface failure was seen until 150 s and good adhesive ability (material failure) was seen at 165 s, which is 50 s longer in heating time according to manufacturer's instruction. But enough adhesive strength was obtained by the use of a solvent, elimination and direct heating of the laminate surface at the short heating time. The differences in the colour of materials influenced adhesion, more transparent materials showed higher adhesion. Furthermore, the influences of room temperature and 37°C in a continuous month-long water sorption test were not seen. In other words, when adhesive strength on the laminated surface, which influences the durability of laminated mouthguard, was considered adequate, the laminated mouthguard had good adhesive ability, while maintaining a decrease in the thickness; that is, when using a mouthguard fabricating system that aimed at lamination by using high temperature and high pressure. If enough attention is given to the heating time, the surface conditions and differences of colour, the laminated-type mouthguard yields good adhesive ability and durability.

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