

## Physical properties and additional characteristics of current elastomeric impression materials

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**Purpose:** This study evaluated physical properties and additional characteristics of six commercial elastomeric impression materials and effect of a silicone remover on peel bond strengths between impression materials and an acrylic resin tray.

**Materials and Methods:** Four silicones (Examixfine, Exadenture, Coltex, and President), one polyether (Impregum F) and one polysulfide (Surflex F) were used. One Type 3 (New Plastone) and one Type 4 (New Fujirock) dental stone casts were used. The tests of detail reproduction, compatibility with dental stones, working time and liner dimensional change were tested by means of International Organization for Standardization (ISO) 4823 and Japanese Industrial Standard (JIS) T6513. The surface roughness ( $R_a$ ) of dental stone casts was measured using a profilometer. The peel bond strengths were measured with mechanical testing device. Statistical analysis was performed using one-way and two-way analysis of variance, Student-Newman-Keuls multiple comparison tests and t-test ( $\alpha=0.05$ ).

**Results:** Large differences were not found among the compatibility with dental stone in all type impression materials ( $p>0.05$ ). The silicone and polyether impression materials indicated the smallest change between 0 day and 1 day of liner dimensional changes than the polysulfide impression material. The Polysulfide impression material exhibited longer working time and setting time, and higher bond strength between acrylic resin trays than the other type of impression materials. When the silicone remover was applied, the values were approximately 0 N/mm.

**Conclusion:** The physical properties and additional characteristics of current impression materials are different among the material. The silicone remover was useful for removal of polyether and polysulfide impression materials from an acrylic resin tray as well as that of silicone impression materials.

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**Key Words:** bond strength, detail reproduction, impression material, physical property, surface roughness.

### Introduction

Impressions of the teeth and oral soft tissues are frequently obtained during dental treatment.<sup>1</sup> Elastomeric impression materials have been used with good clinical results for all kinds of restorations because of their superiority in accuracy, dimensional stability, and reproduction. To obtain well-fitting cast restoration, a high degree of accuracy should be kept in the impression making. Recently, various elastomeric impression materials were developed, and widely used. The dimensional change of impression material is the important index which the timing to pour dental stone to the impressions is decided. Furthermore, the detail reproduction, compatibility with dental stones, working time, setting time, and elastic recovery after setting of these materials, are important for making the dentures and restorations accurately. The impression material must possess sufficient elasticity to permit removal from the mouth without permanent deformation.<sup>2</sup> Furthermore, when an elastomeric impression is removed from the oral cavity, the impression material and the tray must remain attached to each other.

Previously the research concerning setting characteristics,<sup>3,4</sup> compatibility with dental stone<sup>5-12</sup> and dimensional stability<sup>13-15</sup> of impression materials were reported. Relationship between impression materials and a tray material has been also validated by numerous studies.<sup>1,16,17</sup> Sometimes dentists fail to take an accurate impression because of air bubbles and so on. In that case, it is necessary to remove the failed impression material quickly and completely. Recently the silicone remover for resilient denture liners has been developed.

There is little information concerning this effect.

The purpose of this study was to evaluate detail reproduction, compatibility with dental stone, liner dimensional change, working time, setting time and elastic recovery after setting and peel bond strengths to a tray material of six elastomeric impression materials. In addition, the effect of the silicone remover material on removal of the impression materials from the tray material was examined.

## Materials and Methods

Table 1 gives details of the materials used in this study. Four silicone impression materials, one polyether impression material, and one polysulfide impression material were used. One Type 3 and one Type 4 dental stone products were used. The impression material and dental stone were mixed according to the manufacturers' instructions.

**Table 1.** Elastomeric impression materials and dental stone products used.

Material	Type	Batch number	Manufacturer
Examixfine	Silicone	009094	GC Corp., Tokyo, Japan
Exadenture	Silicone	10781	GC Corp.
Coltex	Silicone	JB425	Coltene/Whaledent Inc., Mahwah, NJ, USA
President	Silicone	JF049	Coltene/Whaledent Inc.
Impregum F	Polyether	FW0060620	Espe, Seefeld, Germany
Surflex F	Polysulfide	170881/280881	GC Corp.
New Plastone	Type 3*	0102161	GC Corp.
New Fujirock	Type 4*	0404211	GC Corp.

\*The International Organization for Standardization (ISO) specification 6873 (1998).

### Detail reproduction test

The detail reproduction of impression materials was examined according to the International Organization for Standardization (ISO) 4823 (2000)<sup>18</sup> test method 9.4. Before mixing the impression materials, the test block was cleaned with 99.5% ethanol, and air-dried. The test block and ring mould were stored in the 35±1°C oven for conditioning for at least 15 minutes.

The mixed impression material was placed on the surface of test block, covered polyethylene film (50×50×0.035 mm) and pressure applied by the glass plate (50×50×3 mm). After 60 s of the mix, three specimens were stored to 35±1°C water bath for the minimum time to remove impression from the mouth recommended by manufacturers' instructions. Specimens were removed from test ring, and surfaces were analyzed using the stereoscopic microscope at ×4 to ×12 magnification under low-angle light. The level of reproduction of the A-line, B-line and D-line in the impressions was used to assess the detail reproduction.

### Compatibility with dental stones

The test for compatibility with dental stone was conducted according to the ISO 4823 (2000)<sup>18</sup> test method 9.6. The impressions of the test block were made in accordance with ISO 4823 (2000)<sup>18</sup> test method 9.4.

The dental stone was mechanically mixed with water according to the manufacturers' instructions' water/powder ratio for 30 s in a vacuum. The dental stone mixture was poured to the slit mould, via mechanical vibration. The dental stone/impression material assembly was stored in the laboratory environment (temperature: 23±2°C, relative humidity: 50±10%) for the minimum time to remove cast from the impression

material recommended by manufacturers' instructions.

All cast surfaces were examined according to the ISO 4823 (2000)<sup>18</sup> to assess the compatibility with dental stone. On the other hand, the surface roughness of dental stone specimens was measured after store for 1 day at laboratory environment. The surface roughness was measured by profilometer (Surfcorder SE-3300, Kosaka Laboratory Ltd., Tokyo, Japan) with a tracing length of 2.5 mm and cut-off value of 0.8 mm. Five measurements were carried out per material at laboratory environment, and totally 15 points averaged roughness ( $R_a$ ) per product were calculated.

#### Liner dimensional change test

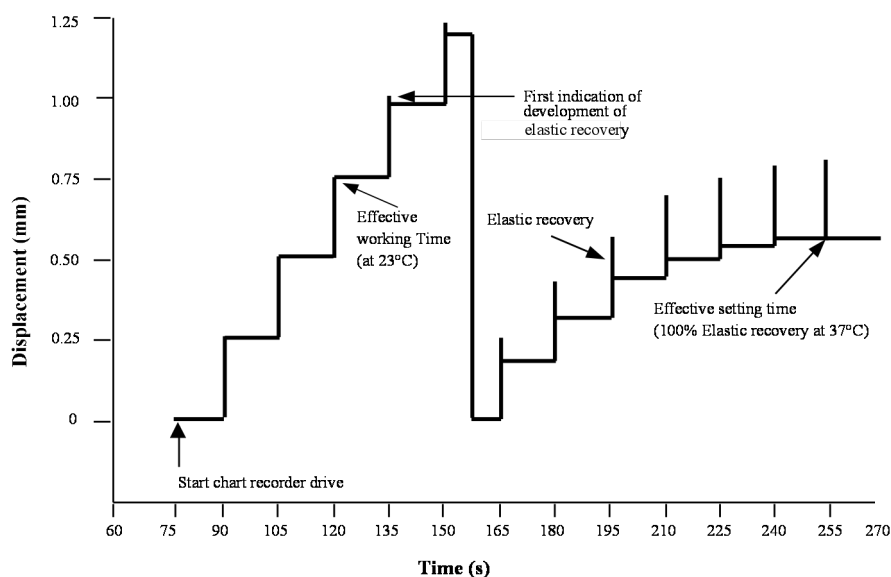
The five specimens were made according to the ISO 4823 (2000)<sup>18</sup> test method 9.4. The dimensional changes 0 day and 1 day after storage were examined according to the ISO 4823 (2000)<sup>18</sup> and the Japanese Industrial Standard (JIS) T6513 (1991)<sup>19</sup> respectively. The test block line-length and specimens dimensions were measured by microscope (MM-40, Nikon, Tokyo, Japan) and data processor (DP-202, Nikon) according to the ISO 4823 (2000)<sup>18</sup> test method 9.5 after store for 0 day and 1 day in distilled water at  $35 \pm 1^\circ\text{C}$ . The liner dimensional changes ( $\Delta L$ ) after 0 day and 1 day were calculated as follows:

$$\Delta L = 100 [(L_1 - L_2) / L_1]$$

where  $L_1$  is the distance between lines  $D_1$  and  $D_2$  on the block,  $L_2$  is the distance between lines  $D_1$  and  $D_2$  on the impression material specimen at 0 day or 1 day.

#### Working time, setting time and elastic recovery test

The working time test was examined according to the ISO 4823 (2000)<sup>18</sup> test method 9.3. Working time and setting time of the test materials were determined using a displacement rheometer. Five tests were carried out for each material both at  $23^\circ\text{C}$  (working time) and at  $37^\circ\text{C}$  (setting time). The mixed impression material was placed between the specimen pedestal and sliding block/perforated test plate assembly of the rheometer. The sliding block/perforated test plate assembly of displacement rheometer was momentarily displaced 0.25 mm using finger pressure (1 s at maximum displacement) at intervals of 15 s. The working time was defined as the time 15 s before corresponding to when initial elastic recovery of the material was observed at  $23^\circ\text{C}$ . The setting time was the time when the elastic recovery reached a final constant value at  $37^\circ\text{C}$  (Fig. 1). The elastic recovery was measured for 10 minutes.



**Fig. 1.** Displacement/time graph for an impression material tested using the displacement rheometer.

### Peel bond strengths test

Ostron II (Powder 260882, Liquid 270192, GC Corp.) was used as a tray material which is a self-polymerizing acrylic resin. Acrylic tray resin was prepared to 75×25×3 mm according to the manufacturer's instructions. The resin plates were smoothed with 240 grit emery papers to remove any adherent adhesive, and were washed with water for 15 s, allowed to air dry for at least 5 minutes. The test surface of each specimen was coated with the adhesive supplied by the manufacturer and allowed to air dry at room temperature (23±2°C) for 15 minutes. Every effort was made to confine the adhesive strictly to the prepared area. The mixed impression material was placed on the surface of the test blocks, which had been coated with adhesive. Five specimens were prepared for each material and experiment.

Peel specimens consisted of an acrylic tray resin plate 75×25×3 mm and impression materials 75×25×3 mm which were bonded over 25 mm of the acrylic specimen and separated over the remaining 50 mm. The impression materials were set at room temperature (23±2°C) for 20 minutes before testing. The specimens were set to the universal testing machine (Model 5565; Instron Corp., Canton, MA, USA) and tested in tensile mode with a crosshead speed of 20 mm/minute at room temperature (23±2°C) until complete separation occurred.

The load (N) at which failure occurred was recorded. The peel bond strengths were calculated as follows:

$$P_s = F W^{-1} [1 + (1 + E)/2]$$

where  $P_s$  is Peel strength,  $F$  applied force,  $W$  width of specimen in the peeling area, and  $E$  extension ratio of material (the ratio of stretched to unstretched length).

The modes of debonding were characterized as tear and peel, or snap,<sup>20</sup> dependent on whether the debonding surface was in the impression material only, at the tray resin-impression material interface only, or the impression material was fracture on the way.

In order to determine the effect of the silicone remover (Siliconeremover, 00642, Tokuyama Dental Corp., Tokyo, Japan) on bond strength to a resin tray material, approximately 20 mL of the silicone remover was applied between the impression material and resin tray material.

### Statistical analysis

All data were analyzed independently by one-way analysis of variance (ANOVA) and two-way ANOVA to determine whether statistically significant differences existed among the materials. These differences were tested with the Student-Newman-Keuls multiple comparison tests. In the surface roughness test, dimensional change test and bond strength test, the t-test was also conducted to determine the effect of the dental stone type, measuring time and silicone remover, respectively. All data were analyzed at a 0.05 level of significance. All analyses were computed with the SPSS for Windows operating system (SPSS 10, SPSS Inc., Chicago, IL, USA).

### Results

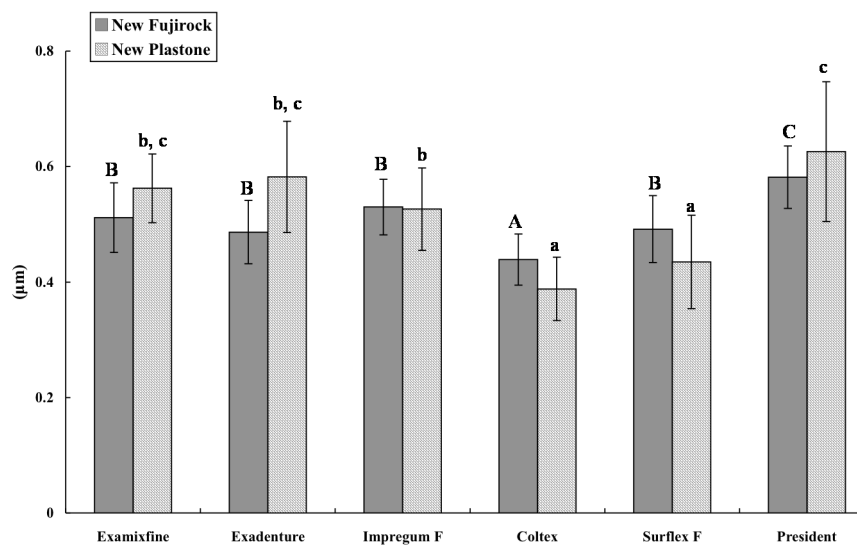
In the detail reproduction test, all impression materials reproduced 20 µm-line. Table 2 shows the outcome of the compatibility with dental stone test. In the New Plastone casts, Coltex reproduced 50 µm-line, and the remainder specimens reproduced 20 µm-line. In the New Fujirock casts, Exadenture and President reproduced 50 µm-line, and the remainder specimens reproduced 20 µm-line.

The outcome of the surface roughness test and dimensional change test 0 and 1 day after storage is illustrated in Figs. 2 and 3. The significant differences were found among the surface roughness of dental stone with made from the impression materials ( $p < 0.05$ ). In the New Fujirock casts, President exhibited highest values of surface

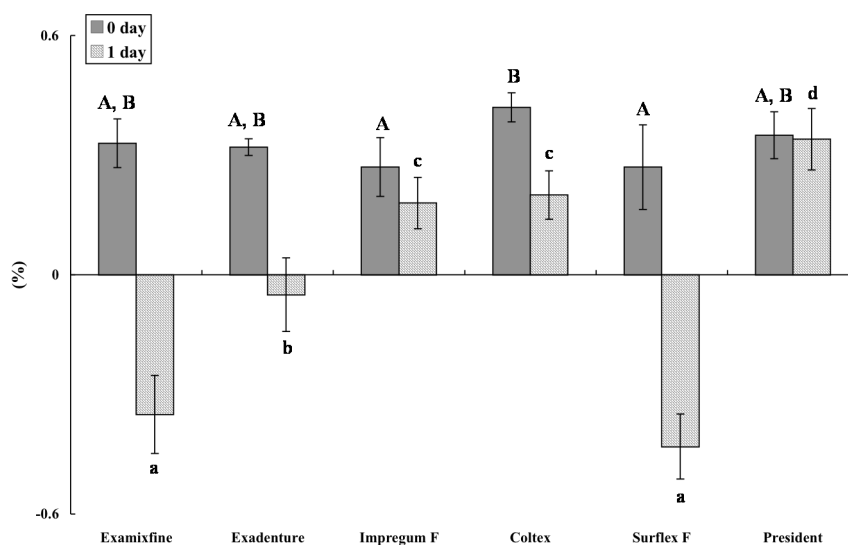
roughness (0.58  $\mu\text{m}$ ) among the materials tested. Coltex exhibited lowest values (0.44  $\mu\text{m}$ ). No significant differences were found among the other impression materials (Fig. 2). In the New Plastone casts, Coltex and Surfex F exhibited lower values (0.39  $\mu\text{m}$  and 0.43  $\mu\text{m}$ ), and President, Exadenture, and Examixfine exhibited higher values than the other materials (Fig. 2). The significant differences were found between two dental stone products except for Impregum F and President (t-test).

**Table 2.** Compatibility with dental stone casts ( $\mu\text{m}$ ).

Material	Examixfine	Exadenture	Coltux	President	Impregum F	Surflex
New Plastone	20	20	50	20	20	20
New Fujirock	20	50	20	20	50	20



**Fig. 2.** Surface roughness of dental stone cast from six impression materials. Identical letters indicate no statistical differences.

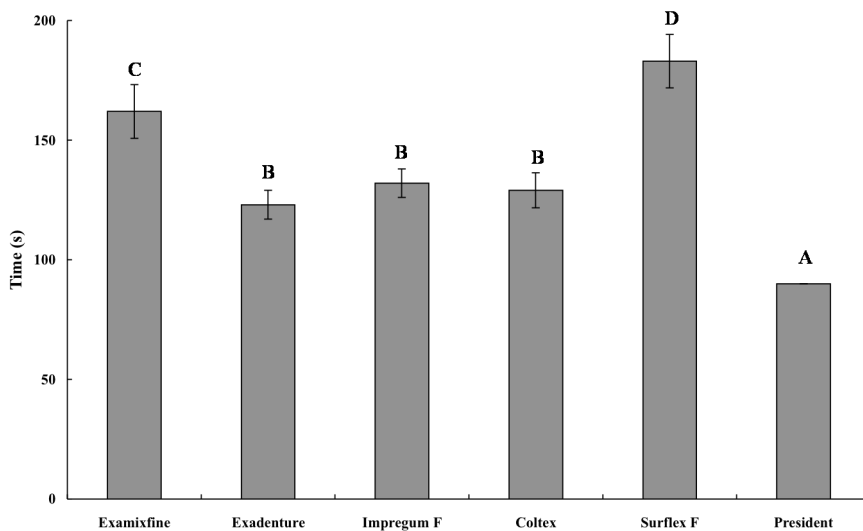


**Fig. 3.** Liner dimensional change of impression materials after 0 day and 1 day. Identical letters indicate no statistical differences.

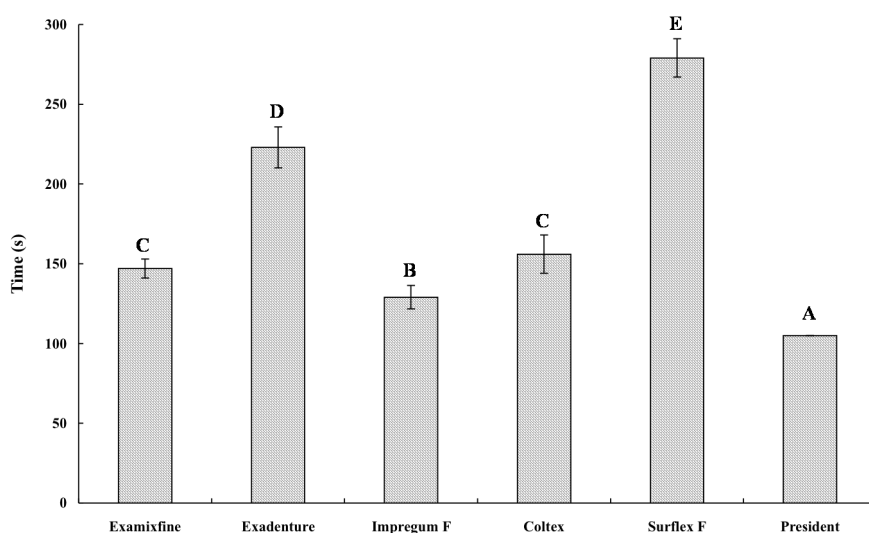
The significant differences were found among the liner dimensional change of impression materials at 0 day and 1 day ( $p < 0.05$ ). The values of liner dimensional change after 0 day ranged from 0.27 % to 0.42 %. No significant differences were found among Impregum F, Surfex F, Exadenture, Examixfine, and President, and among Exadenture, Examixfine, President, and Coltux (Fig. 3). President exhibited highest values of liner

dimensional change after 1 day (0.34 %) among the impression materials. No significant differences were found between Coltex and Impregum F, and between Examixfine and Surfex F. Examixfine and Surfex F exhibited lower values of liner dimensional changes (-0.35 % and -0.43 %) than the other materials (Fig. 3). From results of t-test, the significant differences were found between 0 day and 1 day except Impregum F and President ( $p < 0.05$ ).

The significant differences ( $p < 0.05$ ) were found among the working time and setting time of impression materials. Surfex F exhibited longest values of working time ( $183 \pm 11$  s), and President exhibited shortest values of working time ( $90 \pm 0$  s). No significant differences were found among the Exadenture, Impregum F, and Coltex (Fig. 4). Fig. 5 shows the outcome of the setting time test. Surfex F exhibited longest values of setting time ( $279 \pm 12$  s), and President exhibited shortest setting time ( $105 \pm 0$  s). No significant differences were found between the Examixfine and Coltex. All impression materials complied with the ISO standard of detail reproduction, compatibility with dental stone, liner dimensional change, and working time.



**Fig. 4.** Working time of six impression materials. Identical letters indicate no statistical differences.



**Fig. 5.** Setting time of six impression materials. Identical letters indicate no statistical differences.

Fig. 6 shows the elastic recovery of each impression materials. The elastic recovery of all impression materials increased with the time, and the values of elastic recovery were 100% when setting. The differences

were found among the age changes of elastic recovery of each impression materials. Surfex F exhibited slow setting behavior.

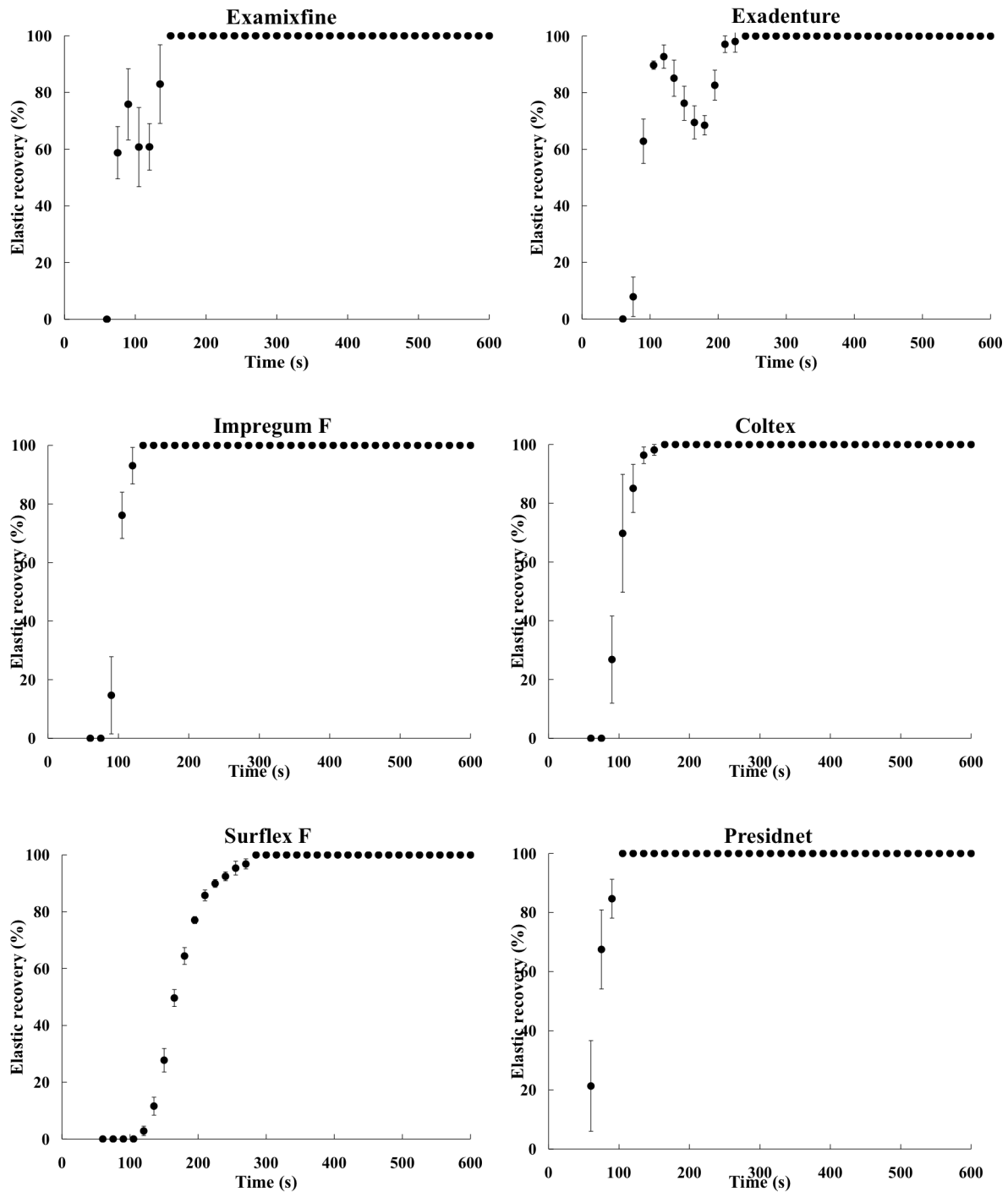


Fig. 6. Elastic recovery of six impression materials.

Table 3 shows the outcome of the peel bond strength test. There were significant differences ( $p < 0.05$ ) among the peel bond strengths of the elastomeric impression materials to an acrylic resin tray material. The values of peel bond strength ranged from 0.012 N/mm to 0.876 N/mm. No significant differences were found between Impregum F and Exadenture, and among Exadenture, Examixfine, President, and Coltex (Table 3). Surfex F exhibited the highest values of peel bond strengths (0.876 N/mm) among the materials tested. Impregum F and

Exadenture exhibited lower values (0.012 N/mm and 0.160 N/mm) than the other materials. In the case of no application of the silicone remover, the impression materials exhibited all three kinds of debonding mode.

**Table 3.** Mean peel bond strength (N/mm) of impression materials.

Material	Without silicone remover			With silicone remover		
	Mean	S.D.	Category	Mean	S.D.	Category
Impregum F	0.012	0.005	a	-0.009	0.012	d
Exadenture	0.160	0.018	a b	-0.042	0.042	d
Examixfine	0.277	0.119	b	-0.010	0.007	d
President	0.299	0.045	b	-0.024	0.034	d
Coltex	0.324	0.123	b	-0.008	0.003	d
Surflex F	0.876	0.154	c	-0.020	0.042	d

S.D., Standard deviation. Category, Identical letters indicate no significant difference between or among materials ( $p>0.05$ ).

The debonding mode of all impression materials was peel when the silicone remover was applied. Using the silicone remover produced significantly lower peel bond strength than no application of the remover in all impression materials (t-test,  $p<0.01$ ). Significant differences were not found among the peel bond strengths of the materials ( $p=0.526$ ). The values of each impression material were approximately 0 N/mm.

## Discussion

The physical properties and additional characteristics of impression materials are important for make a high accuracy complete denture, crown and fixed partial denture. Adhesive property of impression materials to tray resin materials is important because this property has an influence the dimensional stability. Therefore in this study, we evaluated physical properties and additional characteristics of six commercial elastomeric impression materials and effect of a silicone remover on peel bond strengths between impression materials and an acrylic resin tray.

In the detail reproduction test, all impression materials reproduced 20  $\mu\text{m}$ -line. All impression material/stone cast combinations reproduced 20  $\mu\text{m}$ -line except Exadenture/New Fujirock, Coltex/New Plastone, and President/New Fujirock cast combinations. All impression materials complied with the ISO standard of detail reproduction. The impression materials used in this study are appropriate for the precision impression clinically. However, when dental stone is chosen, we should choose an appropriate impression material/stone cast combinations carefully, because the compatibility is different among their combinations.

The significant differences were found among the surface roughness of impression material/dental stone combinations. Coltex produced smoother surface than the other impression materials. The Type 3 dental stone casts (New Plastone) from Coltex and Surflex F were significantly smoother than Type 4 dental stone cast (New Fujirock). The Type 4 dental stone casts from Examixfine and Exadenture were significantly smoother than Type 3 dental stone casts. From these results, in the clinic, the Coltex and Surflex F/Type 3 dental stone combination and the Examixfine and Exadenture/Type 4 dental stone combinations may be appropriate combinations. For Impregum F and President, both types of dental stone cast may be appropriate.

The differences that the surface of the impressions is hydrophobicity or hydrophilicity would influence the setting reaction and change the powder/water ratio of dental stone. And it influences the free growth of the needle crystal of dental stone surface. This may be the reason why difference was admitted in the compatibility



with dental stones.

The values of dimensional change after 0 day ranged from 0.27 to 0.42%. The tendency of shrinkage was observed in all specimens of impressions after 0 day. Most impression materials tended to expand 1 day after immersion in water probably due to the water absorption. Significant differences were found among the dimensional change between the 0 day and 1 day except Impregum F and President. Large rate of change was observed in Examixfine (from 0.33% to -0.35%) and Surfex F (from 0.27% to -0.45%). When these materials are used in clinic, dental stones must be poured soon after making the impression. Otherwise, the reproducibility of the impression may be decreased due to the dimensional change of the impression material and the precision impression may not be making. Small rate of change was observed in Impregum F (from 0.27% to 0.18%) and President (from 0.35% to 0.34%). The dimensional stability of these materials is stable with time than the other materials. Therefore, there may be a few dimensional changes when these materials are dipped in the disinfectant solution for a long time.

The advantages of displacement rheometer are as follows: its operation is easy, it requires only a small amount of material, it can detect the onset of elasticity, and it enables the determination of the working time and setting time of elastomeric impression materials which is based upon readily identifiable and clinically relevant changes in the elastic properties of the setting material.

Surflex F (polysulfide) exhibited longest values and President (silicone) exhibited the shortest values of working time and the setting time. The impression materials having shorter working time, such as President, must be load into the tray in a relatively short time and inserted it into the oral cavity of a patient. The other materials may be margin of the time comparatively. The impression materials such as Surfex F have a longer setting time. If removed impression from the mouth before impression material does not set completely, it may influence a dimensional stability of impression. All impression materials exhibited 100% of the elastic recovery after setting. From this result, these impression materials used in the clinically may be sufficient for the impression taking of the undercut.

In the bonding tests between the impression material and the tray material, silicone, polyether, polyvinylsiloxane and polysulfide materials were often used as the impression material.<sup>21,22</sup> A self-polymerizing acrylic resin, light-polymerizing resin and alloy were often used in the tray material.<sup>1,2</sup> In this study, we evaluated three types of impression materials, and the self-polymerizing acrylic resin as a tray material. The thickness of the impression material was adjusted to 3 mm as reported by Grant et al.<sup>15</sup> and Payne and Pereira.<sup>21</sup> Some studies concerning adhesive properties of impression materials to resin tray materials have been conducted by means of tensile test<sup>2,21-23</sup> and shear test.<sup>24</sup> However, tensile stress and shear stress are not observed at the impression materials/tray interface when the elastomeric impression material is removed from the mouth. The peel testing modality would be thought to simulate clinical behavior more closely when the impression material is removed from the oral cavity and tray. Thus, peel test was used in this study.

The peel bond strengths between the impression materials and resin tray material ranged from 0.012 to 0.876 N/mm. A significant difference was found among the materials. The chemical compositions of impression materials would have an influence on peel bond strength between the impression materials and resin tray material. When silicone remover was applied, the peel bond strengths of all impression materials were near 0 N/mm, and high standard deviations were found on the peel bond strength values. Because peel bond strength were near 0 N/mm, the influence of the weight of the load cell itself is larger. This may be the cause that the

dispersion of peel bond strength values occurred. All three kinds of debonding mode of peel, tear and snap were found in all specimens when the silicone remover was not applied. However, peel had been found when the silicone remover was used.

In the clinical situation, the error of the impression is forecasted. The removal of the impression material from the tray is important. Moreover, it is necessary to remove the failed impression material from the tray easily in order to take the impression again efficiently. To remove the silicone impression material from the tray resin and silicone denture liners from the denture base, the silicon removal material has been developed.

One of the main components of the adhesive material for polysulfide impression material is butyl rubber cement. That for silicone impression material will be poly(dimethyl siloxane) and ethyl silicate. Peel bond strength of the elastomeric impression materials to an acrylic resin tray material decreased significantly by using silicone remover because the silicone remover would dissolve the component of these adhesive materials.

In the silicone impression materials, the differences were found among the compatibility with dental stone except Examixfine, and no differences were found among polyether and polysulfide impression materials. Polysulfide impression material and Examixfine (silicone) indicated largest expansion tendency of dimensional change than other type impression materials. Polysulfide impression material exhibited longer working time and setting time, higher bond strength and later tendency in setting behavior than other type impression materials. The results indicated that the dentists must choose the impression materials and combinations of impression materials/dental stone by the clinical purpose.

When these impression materials are used in the clinic, we should understand physical properties and additional characteristics of elastomeric impression materials.

## Conclusion

The results of this study are summarized as follows.

1. All elastomeric impression materials tested have reproduced the 20  $\mu\text{m}$ -line. No differences were found among the compatibility with dental stone except Exadenture, Coltex, and President. Significant differences in the surface roughness were found among the impression material/dental stone casts combinations.
2. Large differences in the liner dimensional changes after 1 day than 0 day were found among the various impression materials.
3. Surfex F (polysulfide) exhibited longest working time and setting time. The President (silicone) exhibited shortest working time and setting time.
4. Significant differences were found among the peel bond strengths between various impression materials and resin tray material.
5. Silicone remover was effective in removal of not only the silicone impression materials but also of the polyether impression material and polysulfide impression material from the resin tray material.

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