Mechanical Properties of Silver-added Glass Ionomers and their Bond Strength to Human Tooth

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The mechanical properties of glass ionomers, with and without silver addition, for manual mixing and for mechanical mixing, were examined using commercial products. The shear bond strength of the same materials to non-treated human tooth was also examined. The mechanical properties and bond strength of silver-added materials were significantly lower than those of conventional ones. The bond strength to dentin was similar regardless of the addition of silver; however, that to enamel was statistically lower with silver-added materials. No significant difference was observed between the materials for manual mixing and mechanical mixing, whether the material was silver-added or not. The bond strength correlated highly with diametral tensile and flexural strength, but the compressive strength did not have such a relationship.

Key words: Mechanical properties, Bond strength, Silver-added glass ionomers

INTRODUCTION

Glass ionomers have been developed as a favorable restorative material in selected clinical situations, based on their inherent physical and chemical properties. They have a number of useful characteristics, for instance: the ability to bond to both enamel and dentin to such a degree as to reduce a microleakage¹⁻⁴⁾, gradual fluoride releasing anticariogenic properties⁴⁻⁶⁾, slight reactivity to pulpal tissue⁷⁾, and hygroscopic expansion that closes any marginal gaps which may form during setting shrinkage⁸⁾.

Silver-added glass ionomers, which are for mechanical mixing have been commercially available since a few years ago. Their fluoride release and influence on the bond strength to enamel and dentin⁴⁾, setting shrinkage in the dentinal cavity and bond strength⁸⁾, compressive strength and abrasion resistance⁹⁾, the possibility for use as a core material¹⁰⁾, microleakage and mechanical properties^{11,12)}, have been reported. Currently, a similar material, which is for manual mixing, is commercially available from the same manufacturer.

In this study, the mechanical properties of these glass ionomers and their bond strength to human tooth were examined. The difference between the materials for manual mixing and for mechanical mixing was also examined using commercially available products.

MATERIALS AND METHODS

Materials

Two glass ionomers, with and without silver addition, were used for this experiment (Table 1). These materials have similar composition except for Ketac-Silver and Chelon-

Table 1 Materials used

Name	Batch no.	P/L ratio
Mechanical mixing		
Ketac-Fil	N 146	270mg/40mg (=6.75g/1.0g, precapsulated)*
Ketac-Silver	N 135	270mg/40mg (=6.75g/1.0g, precapsulated)*
Manual mixing		
Chelon-Fil	N 232	3.2g/1.0g
Chelon-Silver	N 288	3.8g/1.0g

Manufacturer : ESPE, W.-Germany

* : from Manufacture's sales manual

Silver which contain silver particles in 40 wt%. The mixing procedure recommended by the manufacturer was applied throughout the experiment. In the case of mechanical mixing, it was made by a high speed mixer*, for 10 seconds.

Mechanical properties

A cylindrical specimen, 5 mm in diameter and 10 mm in height, was made using a Teflon mold and a glass plate. Mixed materials were placed into the mold and allowed to set in a humidior at 37°C and 100% R. H. for 30 minutes prior to their removal. Removed specimens were stored in distilled water for 24 hours. The compressive, diametral tensile and flexural strengths were determined by a universal testing machine** at a cross-head speed of 0.5 mm/min. In the case of the flexural strength, a specially designed 3-point transverse bending jig, having a supporting distance of 5 mm, was used. The following equation¹³⁾ was used for calculating the flexural strength.

$$\text{Flexural strength} = 8 PL / \pi D^3$$

Where, P=force at fracture, L=supporting distance, D=diameter of specimen

Shear bond strength

A human tooth, extracted and immediately stored in cold water, was embedded in slow setting epoxy resin***. A flat surface of enamel or dentin was obtained by grinding with a wet silicon carbide paper (No. 600). The surface was not treated otherwise.

A split Teflon mold having a cylindrical space, 3.6 mm in diameter and 2.5 mm in height, was attached to the prepared surface. A mixed glass ionomer was placed into the mold using a syringe. It was then placed in a humidior at 37°C and 100% R. H. for 30 minutes. The mold was removed and the specimen was stored in distilled water at 37°C for 24 hours. The assembly was mounted on a testing machine and shear stress was applied by a cross-head speed of 0.5 mm/min. The force required to break the bonding was measured. The preparation procedure was done in an air conditioned room, maintained at $22 \pm 0.5^\circ\text{C}$ and $50 \pm 5\%$ R. H.

* Silamat, Vivadent, Schaan, Liechtenstein

** Auto-Graph DCS-2000, Shimadzu, Kyoto, Japan

*** Epofix Resin, Struers, Copenhagen, Denmark

RESULTS AND DISCUSSION

Previous studies of a silver-added glass ionomer (Ketac-Silver)^{4,9,11,12,14} indicate many characteristic behaviors in mechanical properties and bonding to tooth. This product was supplied by a manufacturer to be mixed mechanically. Unfortunately, in these reports, the merit of silver addition was not indicated properly. Currently, products for manual mixing (either silver-added or not) are commercially by the same manufacturer, which make it possible to examine the effect of silver addition and the difference between the materials for manual mixing and mechanical mixing.

(1) Mechanical properties of glass ionomers

The results of the mechanical testing are presented in Tables 2 to 4. Compressive, diametral tensile and flexural strengths of silver-added glass ionomers were significantly lower than those of the conventional ones ($p < 0.05$). No significant differences were observed

Table 2 Compressive strength

Mean* $\pm$ SD (kg/cm ²)	Name	Statistical analysis** (Duncan's multiple-range test)
1,892 $\pm$ 191	Ketac-Fil
1,885 $\pm$ 95	Chelon-Fil	
1,860 $\pm$ 52	Chelon-Silver	
1,532 $\pm$ 128	Ketac-Silver	

Number of specimen = 15-16

*: Means are listed in decreasing order.

**: Means linked by vertical lines were not significantly different ($p > 0.05$).

Table 3 Diametral tensile strength

Mean* $\pm$ SD (kg/cm ²)	Name	Statistical analysis** (Duncan's multiple-range test)
130 $\pm$ 18	Chelon-Fil
129 $\pm$ 21	Ketac-Fil	
101 $\pm$ 14	Ketac-Silver
95 $\pm$ 20	Chelon-Silver	

Number of specimen = 15-16

*: Means are listed in decreasing order.

**: Means linked by vertical lines were not significantly different ($p > 0.05$).

Table 4 Flexural strength

Mean* $\pm$ SD (kg/cm ²)	Name	Statistical analysis** (Duncan's multiple-range test)
214 $\pm$ 31	Ketac-Fil
200 $\pm$ 26	Chelon-Fil	
167 $\pm$ 28	Chelon-Silver
154 $\pm$ 29	Ketac-Silver	

Number of specimen = 15-16

*: Means are listed in decreasing order.

**: Means linked by vertical lines were not significantly different ($p > 0.05$).

between the strengths of Ketac-Fil and Chelon-Fil or those of Ketac-Silver and Chelon-Silver, except for the compressive strength of Chelon-Silver and Ketac-Silver ($p < 0.05$).

No significant differences were also observed between the materials for manual mixing and mechanical mixing. Both Ketac-Silver and Chelon-Silver, or Ketac-Fil and Chelon-Fil have the same composition. However, the powder/liquid ratio is apparently different. Although almost two times of powder is used for mechanical mixing, the resultant mechanical properties are similar to those of the materials for manual mixing.

In this study, the flexural strength was measured by cylindrical specimens same as those for compressive test (5 mm in diameter and 10 mm in height) with a specially designed jig. The resultant flexural strength was quite similar to those obtained from the conventional method using beam-shaped specimens: 25 mm long $\times$ 2 mm square¹⁴⁾ and 25 mm long $\times$ 3 mm square¹⁵⁾. This flexural test method has some advantages. One is easier sample preparation and the other is easier comparison of the results with those of compressive or diametral tensile test.

(2) Shear bond strengths to dentin and enamel

The result of the shear bond strength is presented in Table 5. Analysis (Duncan's multiple-range test) indicated that the strength fell into three distinct groups ($p < 0.05$). The bond strengths of silver-added materials to enamel were significantly lower than those of the conventional ones. Although the average bond strength of silver-added materials to dentin was slightly lower than that of the conventional one. On average, whether the silver is added or not, the bond strength to dentin was greater than that to enamel. The shear bond strengths, either to enamel or to dentine, were very similar between the materials for manual mixing and mechanical mixing.

(3) Correlations between the strength of glass ionomer and bond strength

Figures 1 to 3 show the relationships between the compressive, diametral tensile and flexural strengths and the shear bond strength, respectively. A correlation ($p < 0.01$) was observed between either diametral tensile or flexural strength of materials and the shear bond strength. However, such correlation was not seen in the case of the compressive strength.

Table 5 Shear bond strength to human tooth

Mean*±SD (kg/cm²)	Substrate	Name	Statistical analysis** (Duncan's multiple-range test)	
57.0±11.7	Dentin	Chelon-Fil	]]	]
53.9±15.8	Dentin	Ketac-Fil		
49.5±17.4	Dentin	Ketac-Silver		
48.2±10.3	Dentin	Chelon-Silver		
44.9± 9.2	Enamel	Chelon-Fil	]]	]
44.4±12.0	Enamel	Ketac-Fil		
31.7±13.0	Enamel	Ketac-Silver	]]	]
29.3± 7.5	Enamel	Chelon-Silver		

Number of specimen = 9-14

*: Means are listed in decreasing order.

**: Means linked by vertical lines were not significantly different ($p > 0.05$).

Fig. 1 Relationship between compressive and shear bond strength.

Fig. 2 Relationship between diametral tensile and shear bond strength.

During the course of bond strength tests, the majority of failures occurred in the glass ionomer itself rather than at the glass ionomer/tooth interface. This means the bonding failure would occur cohesively and not adhesively, as other recent reports have stated^{1,2,4,8}. Therefore, the mode of bonding failure would apparently correlate to the strength of glass ionomer itself.

Since a high correlation was observed between either diametral tensile or flexural strength of glass ionomers and their bond strength to tooth, it is beneficial to estimate the

Fig. 3 Relationship between flexural and shear bond strength.

bonding characteristics from these mechanical properties without making an actual bond test.

It has become apparent that the mechanical properties of silver-added materials were lower than those of the conventional ones. Also, a significant reduction in bond strength to enamel was observed in the case of silver-added materials, although the less reduction in the bond strength to dentin was seen. At present, the reason why the silver-added materials are less favorable in either mechanical properties or bond strength is not understandable from the present study, nor from the previously reported investigations.

In summary, the silver-added glass ionomers do not have favorable properties investigated in this study. Materials for either manual mixing or mechanical mixing were compared and no merit was obtained from the material for mechanical mixing. So, if this material is apt to be used for occlusal restoration or core build-up as recommended by the manufacturer, further research, other than mechanical properties and bond strength, should be made.

CONCLUSIONS

The mechanical properties and shear bond strength to human tooth of glass ionomers, with and without silver addition, for manual mixing and for mechanical mixing, were examined using commercial products.

The following results were obtained :

- 1) The compressive, diametral tensile and flexural strength of silver-added glass ionomers were lower than those of the conventional (non-silver) materials. These differences were significant with only one exception for the compressive strength of non-silver material.
- 2) The bond strength of silver-added materials to enamel were significantly lower than those of the conventional ones. With respect to dentin, the difference of the bond strength

between the silver-added and conventional materials was not significant.

3) No significant difference in mechanical properties or bond strength was observed between the materials for mechanical mixing and manual mixing, although the powder/liquid ratio was obviously different.

4) A high correlation was observed between either diametral tensile or flexural strength of glass ionomers and their bond strength to tooth. Therefore, it is beneficial to estimate the bonding characteristics from these mechanical properties without making an actual bond test.

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共沈法により調整した $\text{ZnO-Al}_2\text{O}_3$ 系粉末よりなるリン酸亜鉛セメント

—— Ca と F 添加の影響 ——

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Ca と F を添加した $\text{ZnO-Al}_2\text{O}_3$ 系粉末を均一共沈法により調製し, 沈殿物および焼成物の組成と形態に与える影響を検討した。また, リン酸亜鉛セメントとして応用した場合, その歯科理工学的性質に与える両元素の添加の影響についても検討した。

沈殿物は Zn と Al を含む塩基性炭酸水合物であり, 焼成することにより ZnO と ZnAl_2O_4 が生成した。また Ca と F を多量に配合したものでは CaF_2 が生成した。焼成した粉末中の ZnO 粒子の大きさは Ca と F の添加に

より増大したが, ZnO と ZnAl_2O_4 量はほとんど変化しなかった。これらの粉末を市販のリン酸亜鉛セメント液と練和した場合, そのセメント硬化体は両元素の添加により, 圧縮強さが減少し硬化時間が短くなり, セメント表面の中和が促進された。とくに, Zn/Al 比が 5 で Ca, F を少量添加し, 焼成-粉碎により調整した粉末の硬化体は 1% 乳酸および 1% クエン酸中での溶解度が市販品よりも小さい値を示した。

銀配合型充填用グラスアイオノマーセメントの機械的性質と歯質への接着強さ

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銀配合型と従来型（それぞれ器械練和用と手動練和用の 2 種）計 4 種の市販グラスアイオノマーセメントの圧縮強さ, ダイアメトラル引張り強さ, 曲げ強さ, 無処理のエナメル質, 象牙質に対するせん断接着強さを測定した。銀配合型は, 従来型に比較して上述のすべてにおいて統計的に劣っていた。接着強さについて従来型と比較すると, 銀配合型は象牙質に対する接着性には大差がな

いが, エナメル質に対しては有意に低い値を示した。銀配合型と従来型について, 器械練和用と手動練和用とを比較すると, 機械的性質と接着強さのすべてに統計的に有意な差は認められなかった。ダイアメトラル引張り強さと曲げ強さについては, エナメル質, 象牙質に対するせん断接着強さと統計的に有意な関係が認められたが, 圧縮強さについてはみられなかった。

4-META/MMA-TBB レジンセメントとの接着を改善するための

歯科用金銀パラジウム合金の表面処理について

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4-META/MMA-TBB レジンセメントと歯科用金銀パラジウム合金の接着を改善するための金属表面の簡便な処理法の比較を, 金属同志および金属とエナメル質を接着させて行った。サンドブラストを行って鑄造後の酸化膜を除去し, リン酸で陽極処理を行った場合, 37°C の水中浸漬初期から 3 カ月後まで十分な接着強さを保持していたが, 金パラ清掃剤を用いて鑄造後の酸化膜を除去

し, リン酸で陽極処理を行った場合は, 水中浸漬 3 カ月目で接着強さの著しい低下を生じた。サンドブラストのみ行ったものは, 水中浸漬初期の接着強さは小さかったが, 水中浸漬 3 カ月目になると接着強さは増加した。

4-META/MMA-TBB レジンセメントは, 水中浸漬中においても常に新しい接着を形成している事が示唆された。