Effect of Immersion in Water on Linear Expansion and Strength of Three Base/liner Materials

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The purpose of this study was to characterize three base/liner materials in the initial stage of setting, *i. e.*, dual-cured (light-activated glass ionomer), light-cured (light-activated glass ionomer analogue), and chemically-cured (conventional glass ionomer) materials. Changes in dimension, weight and flexural strength were examined from immediately after setting to the end of one week following immersion in water. The results obtained from these three types of material differed from each other, and showed a statistically significant correlation between changes in dimension and weight. This may be attributed to hygroscopic expansion. They also showed different flexural strength, indicating differences in the polymerization system. From these results, it appeared that marginal gaps following setting would be compensated for by hygroscopic expansion during immersion in water for one day or more. Physical properties would be favourably improved following the setting process which continues to advance during immersion in water.

Key words: Cavity liner, Hygroscopic expansion, Flexure strength

INTRODUCTION

With the growing use of the “sandwich technique”, many base/liners having properties comparable to restorative materials have become available¹⁻⁴). These materials can be roughly divided into three groups. The first is conventional, made from a chemically-cured glass ionomer. This material has characteristics similar to conventional glass ionomers, such as (1) mildness to the pulp, (2) fluoride release, and (3) the ability to bond to dentin. The second is dual-cured, made from a light-activated glass ionomer. This is easier to manipulate while having characteristics similar to a chemically-cured glass ionomer. The third is light-cured, made from a light-activated glass ionomer analogue. It releases fluoride and is easy to manipulate.

Clinicians often complete a restorative procedure at one appointment, with little consideration about the type of base/liner material or potential damage to the pulp⁵⁻⁷). In this respect, some reports state that early large gaps and the consequent exposition of freshly cut dentin could cause bacterial penetration and pulpal damage^{8,9}). Other state that restoration failure may occur in the earliest stage of the restoration¹⁰).

Because the lining or base is one of the restorative procedures, we evaluated this on the same basis as a restorative material, and according to the effect of setting shrinkage on the marginal gap^{2,11}). Hygroscopic expansion of restorative materials following immersion in water may be useful to compensate for marginal gap in cases of either composite resin^{9,12,13}) or chemically-cured glass ionomer¹⁴). Because the matrix of hardened chemically-cured

glass ionomer forms a polymer complex^{6,15,16}, we have considered that hygroscopic expansion might improve marginal seal¹⁷. Moreover, we would expect an increase in strength because the setting process advances during immersion in water. A flexural test was used to assess the strength to the base/liner materials¹⁸⁻²¹.

In this study, we examined changes in the dimension, weight and the flexural strength of three base/liner materials, from immediately after setting to the end of one week following immersion in water, to characterize the initial stage of setting. A more effective way of handling base/liner materials in restorative procedures was also discussed.

MATERIALS AND METHODS

Two dual-cured, one light-cured, and two chemically-cured base/liner materials were used in this study (Table 1). Mixing was done according to the manufacturer's instructions. The light-activated material was exposed to a visible light source*. To determine the changes in dimension and weight, specimen sized 3.5mm in diameter and 2mm in height were prepared. In the case of dual-cured glass ionomer and light-cured base/liner material, they were cured for 30 s. Chemically-cured glass ionomers were stored at 37°C and 100% R. H. for four min after mixing. Dimension and weight were measured immediately after light-activation, 30 min, 24 h and one week following immersion in distilled water at 37°C. The height of each cylindrical specimen was measured with an electric micrometer** and the change in the specimen's dimensions during storage was observed²². Water sorption was examined by a balancer# and the change in the specimen's weight during storage was observed²². A specimen size of 15×4×2.5 (mm) was used for the determination of flexural

Table 1 Materials used

Product	Batch No.	P/L ratio
Dual-cured (Light-activated glass ionomer)		
Vitrebond ¹	P890426	1.4g/1.0g
Fuji Lining LC ²	250803	1.4g/1.0g
Light-cured (Light-activated glass ionomer analogue)		
Ionosit ³	81187	
Chemically-cured (Conventional glass ionomer)		
Dentin Cement (for base) ⁴	270401	2.2g/1.0g
Lining Cement (for lining) ⁵	260111	1.2g/1.0g

¹ 3M, St. Paul, USA

² GC, Tokyo, Japan

³ DMG, Hamburg, Germany

⁴ GC, Tokyo, Japan

⁵ GC, Tokyo, Japan

* Luxor, Model 4000, ICI, Cheshire, England

** Digimicro MU-1001B, Nikon, Tokyo, Japan

Balancer AJ100, Mettler, Greifensee, Switzerland

strength. Dual-cured glass ionomer and light-cured base/liner materials were cured again in three overlapping sections. Each section was cured for 30 seconds. Chemically-cured glass ionomers were stored at 37°C and 100% R. H. for four minutes after mixing. Measurements were done using the 3-point bending method with a 10 mm-span and a loading speed of 0.5 mm/min^{##} at 23±2°C and 50±10% R. H..

Except for the strength measurements, all of the procedures were conducted in an air-conditioned room, 22±0.5°C and 50±2% R. H.. Results were analyzed statistically by ANOVA and Duncan's Multiple-Range Test ($p < 0.01$).

RESULTS

Tables 2 to 5 show the changes in dimension, weight and flexural strength.

The dual-cured type of material showed significant increases in dimension, weight and flexural strength up to 24 h, and it was notably affected by water. Changes in the dimension and weight of the light-cured material were similar to that the dual-cured type, but the flexural strength was different. After one day of immersion, the light-cured type had greater flexural strength than the others. Flexural strength significantly declined, -22%, after one week. The chemically-cured type of material did not show any significant changes in dimension or weight, but there was a significant change in flexural strength. Flexural strength increased significantly after one day, although it was lower than that for the other materials.

Fig. 1 shows the relationship between the changes in dimension and weight. All of the types showed significant correlation.

Table 2 Dimensional change following immersion in water

Product	Mean±S. D. (%)*			
	immediately after setting	30 minutes	24 hours	1 week
Vitrebond	0	2.4±0.2	3.4±0.2	3.4±0.4
Fuji Lining LC	0	2.9±0.5	6.2±0.6	6.3±0.6
Ionosit	0	0.2±0.1	1.1±0.4	2.4±0.4
Dentin Cement	0	2.1±0.8	2.3±0.9	2.3±1.0
Lining Cement	0	1.1±0.3	1.1±0.4	0.9±0.5

* The values represent the percent relative to that immediately after setting.

Values joined by vertical or horizontal lines were not significantly different ($p < 0.01$, $n=10$).

^{##} Autograph DCS-2000, Shimadzu, Kyoto, Japan

Table 3 Weight change following immersion in water

Product	Mean±S. D. (%)*			
	immediately after setting	30 minutes	24 hours	1 week
Vitrebond	0	3.1±0.2	7.3±0.2	6.8±0.4
Fuji Lining LC	0	4.4±0.6	9.9±0.6	10.5±0.7
Ionosit	0	0.2±0.1	1.4±0.4	3.3±0.4
Dentin Cement	0	3.0±1.0	4.2±0.7	4.8±0.7
Lining Cement	0	3.1±0.3	2.6±0.4	2.3±0.5

* The values represent the percent relative to immediately after setting.

Values joined by vertical or horizontal lines were not significantly different ($p < 0.01$, $n = 10$).

Table 4 Change in flexural strength following immersion in water

Product	Mean±S. D. (MPa)			
	immediately after setting	30 minutes	24 hours	1 week
Vitrebond	14.6±1.1	22.4±2.3	38.7±2.3	36.4±2.1
Fuji Lining LC	25.2±2.1	33.3±4.4	47.6±7.2	58.6±3.9
Ionosit	60.6±6.0	81.1±8.7	85.4±7.1	47.0±4.1
Dentin Cement	3.2±0.7	6.1±1.0	33.4±2.8	26.7±3.1
Lining Cement	2.0±0.5	3.8±0.7	16.6±5.0	18.2±4.2

Values joined by vertical or horizontal lines were not significantly different ($p < 0.01$, $n = 10$).

Table 5 Comparison of change in flexural strengths following immersion in water

Product	Mean (%)*			
	immediately after setting	30 minutes	24 hours	1 week
Vitrebond	0	53	165	149
Fuji Lining LC	0	32	89	133
Ionosit	0	34	41	-22
Dentin Cement	0	91	944	734
Lining Cement	0	90	730	810

* The values represent the percent relative to that immediately after setting.

Fig. 1 Relationship between dimension and weight change of three base/liner materials.

- Dual-cured type ($r=0.94$, $p<0.05$)
- Light-cured type ($r=0.99$, $p<0.05$)
- ▲---▲ Chemically-cured type ($r=0.83$, $p<0.05$)

DISCUSSION

The three different type base/liner materials showed dissimilar changes in dimension, weight and flexural strength following immersion in water. This may be attributed to differences in composition, the polymerization system and the rate of hygroscopic expansion.

The dual-cured base/liner material is generally called a light-activated glass ionomer. Unlike chemically-cured glass ionomer, it has a dual setting reaction consisting of an acid-base reaction and a photochemical polymerization process^{15,23,24}. In Vitrebond, the polymeric structure includes both poly (HEMA) and graft copolymers of HEMA as well as carboxylic acid and carboxylates arising from the modified poly (acrylic acid) component²³. The hardened Fuji Lining LC is composed of a matrix of uniformed polyacrylic metal salt and Poly (HEMA) around powder particles²⁴. Because of its hydrophilic nature, HEMA expands when it is immersed in water. This explains why its water absorption, changes in dimension, and hygroscopic expansion is greater than that of chemically-cured glass ionomer²³. While the dimensions of the chemically-cured type did not change after 30 min of immersion, dual-cured type showed changes up to 24 h. This can also be explained by the matrix containing Poly-HEMA. The water absorption rate obtained in our study was different from the results of Nicholson and his colleagues²³. This may be due to differences in the baseline. Nicholson and his colleagues set the baseline at 1 h of storage of the materials in an atmosphere at 37°C, while our baseline was immediately after exposure to the light. Although Vitrebond and Fuji Lining LC showed similar changes following water immersion, Fuji Lining LC provided significantly higher water absorption. Forss²⁵ obtained

similar results. This is probably due to differences in the structure of the included hardened materials and the percentage of incorporated HEMA^{23,25)}.

The same explanation can be applied to the flexural strength of the dual-cured type. The matrix formed is more complex because it contains Poly-HEMA, chemically linked to the polyacrylate matrix²⁶⁾ and powder particles are pretreated with silane²⁴⁾. Because of this structure, the dual-cured type exhibited significantly higher flexural strength than the chemically-cured type, as in compressive strength⁴⁾. Vitrebond and Fuji Lining LC showed similar changes in flexural strength following immersion in water, although the latter was significantly higher. Changes in flexural strength were consistent with Lewis's report⁴⁾.

The composition described by the manufacturer implies that the major organic components of the light cured type (Ionosit) are hydrophobic Bis-GMA and TEGDMA. This probably accounts for the difference in properties after immersion in water between light-cured and dual-cured types of base/liner. The light-cured type contains silica filler up to 66%, comparable to that of restorative composite. This may explain the fact that the light-cured type exhibited significantly higher flexural strength than the dual-cured type following 24 h immersion in water (*e. g.*, flexural strength of Silux Plus (3M) after 24 h immersion: 75.4 ± 8.5 MPa, $n=10$)¹⁷⁾. When this material was immersed in water for 1 week, the value for flexural strength was significantly lower than the value obtained following 24 h immersion in water. This is probably due to the low durability in water. Although the light-cured type has been used as a base/liner for composite restoration, the sealability was not as good as that of the dual-cured type⁶⁾. The lower hygroscopic expansion rate of the light-cured type may have caused such poor results.

A chemically-cured type (Dentin Cement) showed significantly higher hygroscopic expansion and flexural strength than others (Lining Cement). Because of their different powder-liquid ratio, the difference in the microstructure was reflected in the two cements^{27,28)}. When this material (Dentin Cement) was immersed in water for 1 week, the flexural strength was significantly lower than it was following 24 h immersion in water. This is probably due to the low durability in water.

When comparing the setting shrinkage rate of the three base/liner materials¹¹⁾, the specimens used in this study were made with teflon molds of the same size. Table 6 showed that the three base/liner materials were shrunk by light-activation or during the initial

Table 6 Change in dimension following filling

Product	Mean (%)*				
	immediately after filling	immediately after setting ¹¹⁾	30 minutes	24 hours	1 week
Vitrebond	0	-1.4	0.9	1.9	1.9
Fuji Lining LC	0	-0.7	2.2	5.5	5.6
Ionosit	0	-0.3	-0.1	0.8	2.1
Dentin Cement	0	-0.6	1.5	1.7	1.7
Lining Cement	0	-1.1	± 0	± 0	-0.2

* The values represent the percent relative to that immediately after filling.

setting in the first stage¹¹⁾, and then they were expanded by water sorption as suggested by the results of this study. That is, the setting shrinkage rate was theoretically compensated for by the hygroscopic expansion rate following immersion in water for 24 h. In addition, hygroscopic expansion would release stress generated during the setting process²⁹⁾.

Therefore, even though a dual-cured base/liner material can be used clinically, it is not recommended to complete the restoration at one appointment. As Fujii advocated³⁰⁾, we also recommend filling the cavity temporarily with a base/liner material at the first appointment, and then completing the remaining restorative procedures at the following appointment. This should improve the marginal gap in the tooth cavity and the strength of material during immersion in water for one or more days, as suggested by the results of this study. It is not good practice to restore in the early stage as marginal gaps may occur in the tooth cavity¹⁷⁾ and distortion of the material may occur in the early setting stage²⁹⁾. Some studies^{8,9)} have stated that large gaps and the exposure of freshly cut dentin would cause bacterial penetration and pulpal damage.

CONCLUSION

Three base/liner materials having different polymerization systems were tested, and showed different changes in dimension, weight and flexural strength following immersion in water. Some of these changes may be used effectively to eliminate marginal gaps during the early stage of setting. It is recommended to fill the prepared cavity temporarily with a base/liner material at the first appointment and, to perform the remaining restorative procedures at the following appointment.

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歯槽骨欠損部の CPC 填入後の組織学的治癒反応

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自然発生に類似した歯周疾患を惹起させた 3 壁性歯槽骨欠損部に対してフラップ手術を行い, CPC あるいは AP を周囲歯槽骨頂縁部と同じ高さになるまで填入した。術後一定期間経過した後の組織学的変化を調べた結果, 術後 1 カ月では, CPC, AP とともに隣接組織に炎症反応は認められなかった。術後 3 カ月目に一部新生骨の添加はみられたが, AP の全周は密な線維性結合組織で覆われていた。一方, CPC 填入部位は歯槽骨に接している付近あるいは歯冠側結合組織付近から部分的に新生骨に

置換していた。術後 6 カ月目には, CPC 填入部位はほとんど新生骨によって置換されていたが, AP は 3 カ月目と同様の所見を示した。CPC を歯槽骨補填に応用した場合, 術後 3 カ月目には CPC の表層および歯槽骨欠損部の周囲骨面からの骨の形成が認められた。現在このような特性を示す材料が他にみられないことより, 本材料が臨床における未解決の分野に応用し得る可能性があるものと判断された。

酸を用いない象牙質面処理

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従来から, 象牙質面の処理には, その酸性度の高低, 有機・無機に差はあるものの主として酸が用いられてきた。しかし, 歯髄保護の観点から, 処理液は酸でないほうが望ましい。そこで従来全く着目されていなかった無機物の水溶液による酸を用いない象牙質面処理を試みた。前処理剤として用いたのは, チオシアン酸カリウム (KSCN) (ロダンカリ) の水溶液で, ロダン化物水溶液のもつコラーゲンおよびその変成物 (ゼラチン) の膨潤

を促進する salting in 効果に着目し, smear layer が良好に除去されることをあきらかにした。また上記の溶液に架橋剤として三価の金属塩 (塩化第 2 鉄, 鉄ミョウバン, カリウムミョウバン) を添加した処理液を用いて処理した結果, 処理面は smear layer が良好に除去され, dentinal plugs は閉塞されている所見が観察された。さらに, 低濃度のロダンカリ水溶液でも scrubbing することによって良好に処理されることが分かった。

浸水による裏層材の線膨張と強さの変化

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硬化方式の異なる 3 種の裏層材 (デュアルキュア型すなわち光硬化型ガラスアイオノマー, 光重合型すなわち光硬化型ガラスアイオノマー類似物, 化学重合型すなわち従来型ガラスアイオノマー) の硬化初期の特性を把

握するため, 硬化直後から水中浸漬 1 週間後までの寸法, 重量および曲げ強さの変化を測定した。3 種の裏層材はそれぞれ異なった変化を示した。水中浸漬 1 週間までの寸法と重量変化とは 3 種共に有意な関係にあった。これ

は吸水膨張によると想像された。曲げ強さはそれぞれ異なり、硬化方式の違いを示す強さの経時的変化がみられた。この結果から、1日以上水中浸漬されることによる

吸水膨張によって、裏層材の硬化時に生じる窩洞辺縁部との間隙を防止したり、硬化が進行することによる物性の安定化が期待された。

コンポジットレジン機械的性質の低下

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γ -MPTMS によって表面処理した6種類のフィラーを用い、異なったフィラー形状、フィラー粒径、フィラー含有率をもつ16種類の光重合型コンポジットレジンを試作した。試料は0～60日間蒸留水中に浸漬し、曲げ強さおよび曲げによる応力緩和率の測定を行った。曲げ強さは、すべての材料で水中浸漬期間が長くなるとともに減少し、60日後では浸漬前に比べて約10～30%低下した。またフィラー含有率の高い材料(65 vol %)は含有

率の低い材料(40 vol %)よりも大きな低下率を示した。応力緩和率についても曲げ強さの場合と同様の傾向を示し、浸漬期間が長くなるにつれて緩和率は増加した。さらにフィラー含有率の高い材料は、浸漬前の値に比べてより大きな緩和率の増加を示す傾向がみられた。こうした機械的性質の低下は、フィラー表面処理剤の加水分解による劣化が大きな影響を及ぼしているものと思われる。

硬質セッコウの脱水過程に生ずる比熱測定における異常ピーク

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断熱型比熱測定装置により、硬質セッコウ硬化体の脱水過程を検討した。一般には大気中において、二水塩から半水塩を経由して無水塩への二段階の脱水過程を示すことが知られている。しかし、混水比の小さいバルク試

料の脱水は見かけ上、三段階で進行する。これは本質的なことでなく、ち密な硬化体が円滑な脱水をさまたげることにより生じたピークであると考えられる。