Setting Characteristics of Fissure Sealant

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Working time and setting time of four commercial sealants were investigated by means of a rheometer for composite resins. Temperatures in extracted human tooth were also examined by means of a thermocouple during the setting of sealant brushed into the pit and fissures. In addition, unreacted substances in the cured materials were studied using a high performance liquid chromatography.

The working time was 0.52-1.09 min at 23°C and the setting time was 0.91-1.38 min at 32°C. The temperature of the dentin at a point 1.20 mm from the roof of the pulp chamber ranged from 0.18 to 0.21°C, as specimens of each material 0.5 mm thickness were cured. The percentages of unreacted substances were calculated as the ratio of the area under the chromatogram curve obtained before and after curing, and these ranged from 19.7 to 45.8 for specimens of the four materials with a thickness of 0.5 mm.

Probably, temperatures during the setting of the four materials tested had little effect on the damage to dental pulp.

Key words: Fissure sealant, Setting characteristics, Temperature rise

INTRODUCTION

Recently, composite materials that used MMA and Bis-GMA resins as a matrix have been extensively employed in order to prevent caries, by sealing the pit and fissures of teeth. To provide adequate protection of the teeth agansist caries for a long-time under normal oral conditions, these materials have to possess excellent properties, with regard to bonding to the tooth substance, sealing of the pit and fissures, wear resistance, durability to fatigue, water proof capacity, and mechanical properties (i.e. tensile and compressive strengths and impact strength). Therefore, in this field, many studies on sealants have been reported formerly1-6). However, it is not evident whether or not these materials are compatible with tooth tissue.

The purpose of this paper was to investigate working and setting times, temperature in the tooth and unreacted substance in the cured material.

MATERIALS AND METHODS

There were four materials used in this study: Delton*, Fissureseal**, Teethmate-s*** and White sealant****, and they were cured in accordance with the manufacturers' instruc-
(1) Determination of working time and setting time

Measurements were performed using an oscillating rheometer for resin-based dental filling materials (BS 5119)7,8).

The working time for each material was determined in an air cabinet maintained at 23±0.5°C. This time was measured as the time from when the mixing began until a continuous trace of viscosity change during the setting reached 95% of its initial width. On the other hand, the setting time was determined at 23±0.5°C and 32±0.5°C by means of the same method as that described for the working time, as the time from the start of the mix until the curve flattens out to the final straight portion.

(2) Measurement of temperature

The measurement of temperature was carried out in three parts (A, B, C) between the roof of the pulp chamber and the pit of an extracted human molar, using a thermocouple*****, as shown in Fig. 1. A high vacuum silicone grease was brushed in a thin layer over pit and fissures without any acid-etching treatment on the tooth enamel.

(3) Determination of unreacted substances in the cured materials

Each material was cured in the PTFE mold, 3 mm in diameter and 0.5 mm deep. Thereafter, they were immediately immersed in 10 ml tetrahydrofuran (THF) and were

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* Johnson and Johnson, East Windsor, USA, Batch number 051782
** GC, Tokyo, Japan, Batch number 060331
*** Kuraray, Okayama, Japan, Batch number 12001
**** 3M, St. Paul, USA, Batch number 012683
***** Copper-constantan wire, Ø 0.3 mm, 1 m in length

Fig. 1 Schematic representation of extracted tooth which indicates the position measured.
maintained for 24 hours at $37\pm 0.5^\circ C$ prior to the measurement. Materials before curing were also immersed, using the method described above. All measurements of unreacted substances were performed at $23\pm 0.5^\circ C$, using a high performance liquid chromatography*, incorporating two steel columns ($0.3 \text{ m} \times 8.0 \text{ mm} \phi$). A sample of each solution ($20 \mu l$) was injected into the chromatography and three measurements were made for each solution. The percentage of unreacted substance in the cured materials was calculated as the ratio of the area under the curve of the chromatogram obtained before and after curing the materials.

**RESULTS AND DISCUSSION**

The working and setting times for each material tested are shown in Figs. 2 and 3. The working time ranged from 0.52 min for Fissureseal to 1.09 min for Delton, and the setting time in air at $32^\circ C$ ranged from 0.91 min for Teethmate-s to 1.38 min for White sealant. Considered from a viewpoint of clinical application, it can be seen that these values are too short for working and are too long for setting. For the setting times at 23 and $32^\circ C$, White sealant was strongly affected by the temperature. On the other hand, Delton was almost independent of the temperature.

Specimens of 0.5 mm thickness for each material were cured into the pit and fissures of extracted human molar in an air cabinet maintained at $32^\circ C$, and the temperature of the inner parts (A, B, C) of tooth are shown in Fig. 4. The temperature at A part for each material had the highest values and C part had the lowest. Figure 5 shows the variations of temperature at parts A and C during the setting of materials, when the atmospheric temperature was at 23, 32 and $37^\circ C$. With the increase of atmospheric temperature, the tooth temperature had a tendency to increase, especially, at A part. Figure 6 shows the variations in the time required until the temperature reached the maximum peak after the mixing began. The maximum peak of the temperature at C part occurred later than that of A part and in

![Graph showing working times of sealants](image-url)

**Fig. 2** Working times of sealants measured in an air cabinet maintained at $23\pm 0.5^\circ C$ after mixing for 7 sec at $23^\circ C$.

* LC-3A, Shimadzu, Kyoto, Japan
Fig. 3 Setting times of sealants

* in the case of the setting in an air cabinet maintained at 23±0.5°C after mixing for 7 sec at 23°C.

** in the case of the setting in an air cabinet maintained at 32±0.5°C after mixing for 7 sec at 23°C.

Fig. 4 Variation of the temperature of the inner part (A, B, C) of tooth with the distance from the pit of extracted tooth to the roof of the pulp chamber, during the setting of sealants in 0.5 mm thickness brushed into the pit and fissures in an air cabinet maintained at 32±0.5°C.

- Delton
- Fissureseal
- Teethmate-s
- White sealant

Fig. 5 Variation of the temperature of the inner part of tooth with the environment temperature, during the setting of sealants in 0.5 mm thickness.

- Delton
- Fissureseal
- Teethmate-s
- White sealant

According with the increase of atmospheric temperature, it also required less time to reach the maximum peak. From these results, it is guessed that according to the increase of atmospheric temperature, the heat generated during the setting is diffused rapidly toward the inner part of tooth. The temperature at C part, in the vicinity of the roof of the pulp chamber, becomes a serious problem, because it might damage the dental pulp in clinical application. The temperature at C part for the four materials tested was 0.18-0.21°C in air at 32°C, with
Fig. 6 Variation of time until the temperature reaches maximum peak after the mixing began with the environment temperature, during the setting of sealants in 0.5 mm thickness.

- ○: Delton; ○: Fissureseal; □: Teethmate-s; △: White sealant.

Fig. 7 Variation of the temperature in extracted tooth with the thickness of sealants tested, during the setting in an air cabinet maintained at 32°C.

- ●: Delton; ○: Fissureseal; □: Teethmate-s; △: White sealant.

the mouth opened. From these results at C part and from the report published already⁹, it is wholly presumed that there is little damage of the dental pulp.

Figure 7 showed the variations of the temperature at part A and C in relation to
Table 1 Unreacted substances in the cured materials

<table>
<thead>
<tr>
<th>Material used</th>
<th>Unreacted substance (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Delton</td>
<td>19.7</td>
</tr>
<tr>
<td>Fissureseal</td>
<td>45.8</td>
</tr>
<tr>
<td>Teethmate-s</td>
<td>35.3</td>
</tr>
<tr>
<td>White sealant</td>
<td>35.3</td>
</tr>
</tbody>
</table>

thickness, when each material was cured in an air cabinet maintained at 32°C. As the thickness of the materials increased, so did the amount of heat generated. Consequently, the temperature of the inner part of tooth becomes higher. When the thickness of materials was more than 0.5 mm, the temperature of the inner part of tooth rose markedly and particularly, for Delton and Teethmate-s, it rose excessively. Therefore, in clinical application, these materials should be used in as a thin layer as possible.

Table 1 shows the ratio of the area under the curve of chromatogram obtained before and after curing. The percentages of unreacted substances in the cured materials ranged from 19.7 to 45.8. This made little difference in filling composite resins\(^{10-12}\). Therefore, the effect of the unreacted substances on tooth tissue is a serious problem which should be investigated in the near future. In addition, there was almost no correlation between the unreacted substances in the cured materials and the temperature in extracted tooth during the setting of sealants in 0.5 mm thickness.

CONCLUSIONS

The working and the setting times, unreacted substances in the cured materials, and the temperature of the inner part of tooth during the setting of the four materials were investigated, and their results are as follows.

(1) The working time ranged from 0.52 min for Fissureseal to 1.09 min for Delton at 23±0.5°C.

(2) The setting time ranged from 0.91 min for Teethmate-s to 1.38 min for White sealant at 32±0.5°C.

(3) In the air cabinet maintained at 32°C, the heat generated during the setting for the four materials occurred with a temperature rise between 0.18 and 0.21°C at C part for a point 1.2 mm from the roof of the pulp chamber. The time until these temperature reached the maximum peak ranged from 1.8 to 2.12 min from when the mixing began.

(4) With the increase in atmospheric temperature, the degree of temperature rise in the extracted tooth increased, when the materials were cured into the pit and fissures.

(5) With the increase in thickness of the materials brushed on, the heat generated during the setting increased and the temperature in extracted teeth also increased.

(6) The percentages of the unreacted substances in the cured materials were between 19.7 and 45.8, and there were almost no correlation between these unreacted substances and the temperatures in extracted tooth during the setting of materials of 0.5 mm thickness.
REFERENCES


不自然な文章です。
コンポジットレジン用レオメータにより4種類の市販小窩製溝塗装材の操作時間、硬化時間を測定した。また、人の抜去歯の小窩製溝塗装された材料の硬化中の温度上昇を熱電対を用いて調べ、さらに、高速液体クロマトグラフを使って硬化後の未反応物質の分析を行った。
23℃でのそれぞれの材料の操作時間は0.52～1.09minの間であり、そしてまた32℃での硬化時間は0.91～1.38minの間であった。0.5mmの厚さで硬化させた時、槽室蓋から1.2mm離れたデンチック部分での温度上昇は0.18～0.21℃の間であった。未反応物質は、硬化前のそれぞれの溶液で測定されたクロマトグラフの面積比で計算され、0.5mmの厚さの試料では、それぞれの材料で19.7～45.8％の範囲であった。
本実験中の小窩製溝塗装材では、硬化時の温度上昇の歯髄に対する影響はほとんどないと思われる。

充てん用コンポジットレジンに関する研究
—第3報—光重合型コンポジットレジンのL細胞に対する毒性試験

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現在市販されている18種類の光重合型コンポジットレジンについて、マウス由来の培養細胞に対する各材料の細胞毒性試験を行った。細胞障害の程度は、細胞死を含む核増殖率と細胞形質に萎縮がみられる領域の2領域にわけ、それぞれの領域の面積で表示した。また、各材料の硬化物中に含まれる未反応物の量を高速液体クロマトグラフを用いて定量し、未反応物量と細胞障害がみられる領域との関係を調べた。その結果、4種類の材料（パルフィークライト、プリズマフィル、プリズマフィルコンピュール、バイオフィルライトポンド）を除く他の材料間では、硬化体中の未反応物量と細胞障害を引き起こす領域との間の相關がみられ、未反応物の増加に伴って細胞障害を起こす領域が大きくなっていることがわかった。除外した4種類の材料については、硬化体中の未反応物量は少ないにもかかわらず、細胞障害を起こす領域が著しく大きくなっている。この理由については現在検討中である。

接着性ブリッジの耐久性に関する破壊力力学的解析
——接着性ブリッジの設計要因が接着層でのき裂成長に与える影響——

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有限要素法にもとづく破壊力学的手法によって、接着性補綴物の設計条件と接着材層に発生するき裂成長との関係をエネルギー解放率gの概念を用いることにより解析した。解析は接着材層でのき裂成長に伴うエネルギー解放率とクリティカルロードの値が、水平のなき長さα、安全率n、メタルフレームの構造および金属の厚さのファクターによりどの様に変化するかにより行なった。解析の結果、gは金属の厚みが増すと減少し、1.0mm以上では非常に小さくなった。また肢接面にウィングを設定すると設定しない場合に比べgは小さくなり、その差△gはウィング長の半分程度で最大となった。そして△gは、金属厚みの増加に伴い減少した。結果とクリティカルロードを用いた考慮により、接着性補綴物の耐久性を向上させる為のブリッジの設計要因について検討を行なった。