Composite Restorative Resins

Part 1 Setting Characteristics of UV and Visible Light-activated Composite Resins

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The setting times of 18 commercial light-activated composite resins were measured using an oscillating rheometer, and the effects of specimen thickness, exposure time and distance from the fiber tip of light curing unit to the specimen on the setting time were examined. In addition, the curing shrinkage in the direction of tooth axis and the effect of inorganic filler content on the curing shrinkage were evaluated.

The setting times for specimens of maximum depths of cure recommended by the manufacturers were in the range 0.9–2.5 min. These values varied with the thickness of the specimen, exposure time and distance from the fiber tip of light curing unit to the specimen. The curing shrinkage in the direction of tooth axis for a 2.5 mm specimen was in the range 1.5–2.8 %.

Key words: Composite restorative resin, UV and visible light-activated resin, Setting characteristics

INTRODUCTION

Recently, several commercial light-activated composite resins have been used as a material for restoration. However, little is known of their physical and mechanical properties¹⁾. To produce a clinically favourable restoration, it is important to understand these properties during and after setting. The present investigation was undertaken to determine the setting time and the curing shrinkage of light-activated composite resins.

MATERIALS

The materials used in this study are listed in Table 1, together with shades, batch numbers, codes, exposure times and maximum depths of cure recommended by manufacturers.

All materials were cured using appropriate light curing units recommended by the manufacturers at $37 \pm 0.5^\circ\text{C}$.

APPARATUS AND METHODS

1. Setting time

The apparatus used was an oscillating rheometer modified for light-activated composite

Table 1 Materials and lights used

Code	Material	Shade	Manufacturer	Batch number	Light and batch number	Exposure time and depth of cure recommended by manufacturer	
						Exposure time (s)	Depth of cure (mm)
For anterior teeth							
Bri	Brilliant lux	U	Coltene ¹⁾	090783	Pluraflex HL150, 01737	40	2.0
Dur	Durafill	U	Kulzer ²⁾	Jan84136	Translux CL, 15/11462	20	3.0
Est	Estilux	GO	Kulzer ²⁾	Aug83142	Duralux UV-300, 505/1919	40	2.5
Fir	First Lite	U	Phase alloy ³⁾	40404	Wite Lite™, 60610	20	2.5
Hel	Heliosit	20	Vivadent ⁴⁾	011283	Heliomat, 18518	20	4.0
LAR	Light Activated Restorative	L	Teledyne Getz ⁵⁾	603081	Focus™ Activator Light, 35408	20	3.5
LiA	Lite-Fil A	U	Shofu ⁶⁾	018401	Daylight Lamp, 0184704	30	3.0
Pal	Palfique Light	U	Tokuso ⁷⁾	LU204	Wite Lite™, 60610	30	3.0
Pho	Photo Clearfil A	US	Kuraray ⁸⁾	1046	Quick Light, 1205	20	2.5
Plu	Plurafil Super	U	Lite™ ⁹⁾	40545	Pluraflex HL150, 01737	30	3.5
Pri	Prismafil	L	Caulk ¹⁰⁾	111683	Prisma Lite, 24520-1	10	3.5
PrC	Prismafil Compules	LG	Caulk ¹⁰⁾	0720834	Prisma Lite, 24520-1	10	2.5
Sil	Silux	U	3M ¹¹⁾	072484	Optilux, 00287	20	2.5
SLD	Super Lux Daylight	U	Shofu ⁶⁾	30344	Daylight Lamp, 0184704	30	3.0
For posterior teeth							
LiP	Lite-Fil P	—	Shofu ⁶⁾	018413	Daylight Lamp, 0184704	30	3.0
Occ	Occlusin	—	ICI ¹²⁾	450181/1/B	Luxor, 06462	40	3.0
P30	P-30	—	3M ¹¹⁾	4U6P	Optilux, 00287	20	2.5
Pyr	Pyrofil Light Bond	—	Sankin ¹³⁾	163-502	Suncure Light, 1013	20	3.0

1) Altstätten, Switzerland 2) Homburg, West-Germany 3) Los Angeles, USA 4) Schaan, Lichtenstein 5) Grove Village, USA 6) Kyoto, Japan 7) Tokuyama, Japan 8) Kurashiki, Japan 9) Barden-Barden, West Germany 10) Milford, USA 11) St. Paul, USA 12) Macclesfield, England 13) Osaka, Japan

resin. This instrument consists of a lower platen assembly as previously described by Bovis *et al.*²⁾ The upper platen assembly is markedly modified so that the specimen can be exposed to the light.

The thicknesses of the specimens tested were 1.5 mm and maximum thickness recommended by the manufacturer. The exposure times were designated 1T, 2T and 3T. The value means the time which the specimen was exposed to the light, thus the value, 2T, means a period twice that recommended by the manufacturer. The distances from the fiber tip of light curing unit to the specimen were 1, 2 and 3 mm. The setting time was determined according to the British Standards recommendations for composite filling material³⁾.

2. Curing shrinkage

A schematic diagram of the apparatus used is shown in Fig. 1. The material was filled in a Teflon ring (8 mm in diameter) supported on a 1 mm-thickness quartz glass plate, using a syringe dispenser. An aluminum target (6 mm in diameter and 0.2 mm in thickness) was placed on the material. The specimen was exposed for the recommended exposure time (1T) from the bottom surface. The dimensional change during setting was measured by using a gap detector. The specimens tested were 1.0, 2.0 and 2.5 mm in thickness. The percentage of curing shrinkage at 10 min from the start of exposure was measured.

In addition, the volumetric curing shrinkages were measured by using a pycnometer. The values were calculated from the change of specific gravity before and after polymerization.

Fig. 1 Schematic diagram of apparatus used for measuring curing shrinkage.

3. Inorganic filler content

To investigate the effect of filler on the curing shrinkage, inorganic filler content of each material was measured.

Three cylindrical specimens (8 mm in diameter and 3 mm in height) were placed in crucible and were weighed. After firing at 700°C for 60 min, their weights were determined again. The percentage of inorganic filler content was expressed as the ratio of weight before and after firing.

RESULT AND DISCUSSION

The setting times, the curing shrinkages and the inorganic filler contents of the materials used are shown in Table 2. The setting times for the 1.5 mm specimens ranged from 0.2 min for P30 to 1.4 min for Est. Est exposed to UV light had a longer setting time than those of the other materials exposed to visible light. On the other hand, for specimens of maximum thickness, the setting times ranged from 0.9 min for Fir and P30 to 2.5 min for Hel. The setting time could not be measured for the Est specimen of maximum thickness because of incomplete polymerization. All materials, except for Est, had longer setting times than those for the 1.5 mm specimens, and these values were 2-10 times as long as the exposure times recommended by the manufacturers.

The effects of exposure time and distance from the fiber tip of light curing unit to the specimen on the setting time are shown in Figs. 2 and 3. With all materials except for Plu, the setting times for maximum thickness decreased with the increase of exposure time. Concerning the distance, the setting times for the 1.5 mm specimens became longer with increasing distances (1-3 mm), in case of all materials except for Fir, Hel, Sil, LiP and Pyr. This tendency was particularly marked for Bri, Est and Pri. These results indicate that the

Table 2 Setting times, curing shrinkages (in the direction of tooth axis), volumetric curing shrinkages and inorganic filler contents of materials used.

Material	Setting time (min)		Curing shrinkage (%)			Volumetric curing shrinkage (%)	Filler (inorganic) content (wt%)
	thickness 1.5mm	of specimen maximum	thickness 1.0mm	2.0mm	2.5mm		
Bri	1.1	1.7	4.2	3.0	2.7	2.3	73.8
Dur	0.9	1.4	3.3	2.8	2.6	2.6	51.8
Est	1.4	—	3.6	2.7	1.8	1.7	77.8
Fir	0.7	0.9	4.8	2.7	2.3	2.5	77.9
Hel	1.1	2.5	2.8	2.5	2.0	2.0	36.5
LAR	0.4	1.6	3.7	3.2	2.6	2.4	79.5
LiA	0.6	1.7	2.3	2.0	1.5	1.7	80.3
Pal	0.6	1.2	2.8	2.5	2.3	1.9	67.0
Pho	0.7	1.1	3.2	2.6	2.1	1.8	83.2
Plu	0.8	1.2	2.6	2.0	1.9	2.3	57.8
Pri	0.7	1.8	4.2	2.9	2.7	2.5	74.8
PrC	1.1	1.4	3.6	2.9	2.6	2.7	75.4
Sil	0.6	1.1	3.1	2.5	1.9	1.9	52.6
SLD	1.0	1.7	4.3	3.1	2.8	—	34.3
LiP	0.7	1.7	3.7	2.1	1.7	1.7	82.4
Occ	0.4	1.0	4.0	2.6	2.6	2.4	85.4
P30	0.2	0.9	4.6	3.3	2.4	2.3	83.9
Pyr	0.4	1.2	3.3	3.0	2.4	2.3	81.0

Fig. 2 Variation of setting time with exposure time. (1), Bri; (2), Dur; (3), Fir; (4), Hel; (5), LAR; (6), LiA; (7), Pal; (8), Pho; (9), Plu; (10), Pri; (11), PrC; (12), Sil; (13), SLD; (14), LiP; (15), Occ; (16), P30; (17), Pyr

setting times for most materials are affected by exposure time and distance from the fiber tip of light curing unit to the specimen.

The curing shrinkages for the 2.5 mm specimens ranged from 1.5 % for LiA to 2.8 % for SLD. On the other hand, those for the 1.0 mm specimens ranged from 2.3 % for LiA to 4.8 % for Fir. With all materials, the curing shrinkages increased with the reduction of specimen

Fig. 3 Variation of setting time with distance from the fiber tip of light curing unit to the specimen. (1), Bri; (2), Dur; (3), Est; (4), Fir; (5), Hel; (6), LAR; (7), LiA; (8), Pal; (9), Pho; (10), Plu; (11), Pri; (12), PrC; (13), Sil; (14), SLD; (15), LiP; (16), Occ; (17), P30; (18), Pyr

thickness. These changes may be caused by differences in the degree of polymerization of the specimens. However, for Bri, Fir, Pri, SLD, LiP, Occ and P30, the values for the 1.0 mm specimens were markedly larger than those for the specimens of other thicknesses. It is speculated that the marked shrinkage occurred due to adhesion to the quartz glass plate and Teflon ring and to increased polymerization.

It was reported that the inorganic filler contents differ widely from one product to another, and these differences are associated with the curing shrinkage⁴⁾. However, as shown in Fig. 4, there was no correlation between the inorganic filler content and the curing shrinkage ($r=0.110$). Therefore, the curing shrinkage appears to be affected by the other factors, such as the degree of polymerization and the type of monomer used⁵⁾.

For most materials, good agreement was observed between the values of curing shrinkage obtained by using a gap detector and those obtained by using a pycnometer. When the linear shrinkage of the material occurs evenly in all directions, it should be one-third of the volumetric shrinkage. In this investigation, however, shrinkage mainly occurred in the direction of tooth axis. This means that in clinical use, the curing shrinkage of light-activated composite resin results in a gap between the filled resin and the bottom surface of cavity.

CONCLUSION

The setting times for specimens of maximum thickness recommended by the manufacturer were in the range 0.9–2.5 min. The values were greater than those for the 1.5 mm specimens. For most materials, the setting times varied with exposure time and distance from the fiber tip of light curing unit to the specimen.

The curing shrinkages at 10 min from the start of exposure for the 2.5 mm specimens

Fig. 4 Curing shrinkage for the 2.5 mm thickness of specimen vs. inorganic filler content.

were in the range 1.5-2.8 %. The values increased with the reduction of specimen thickness. No correlation was seen between the curing shrinkage and the inorganic filler content. The values of curing shrinkage in the direction of tooth axis approximately agreed with the values of volumetric curing shrinkage. Most of the curing shrinkage of light-activated composite resin was considered to occur only in the direction of tooth axis.

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は 13~17 kg/cm² であったが、グラスアイオノマーセメントで裏層を施すことにより 41~46 kg/cm² の接着強さを示した。接着性レジンセメントのアマルガム修復へ

の応用は、アマルガムを歯質に接着させるだけでなく、二次う蝕の予防にも有効であると思われる。

コンポジットレジン修復の象牙質窩縁からの微小漏洩に及ぼす Cleaning Agents の効果

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われわれはこれまでにキレート剤, 低濃度の無機・有機酸, あるいは象牙質有機質に対する効果を期待して, 収れん作用を持つ塩類などのなかから, 生体に対してマイルドで象牙質清掃効果の高い処理剤 (Cleaning Agent) として使用できるものを模索してきた。今回, それらのなかから, 比較的低濃度で, 被処理象牙質面の SEM 観察によっても良好な smear 層の除去効果が認められた 10% アスコルビン酸, 10% クエン酸, 10% リン酸および 3% 乳酸アルミニウムを処理剤として用いて,

全象牙質窩洞への処理効果を辺縁封鎖性の評価によって検討した。

各種の清掃剤の中では 10% クエン酸が最も高い処理効果を示したが, 歯頸側窩縁からの微小漏洩を完全には阻止することができないことがわかった。さらに, 被験歯の条件をよく揃えた今回の実験でも, 歯によって辺縁封鎖性に大きい差を生じることなどから, 象牙質窩縁の封鎖の困難性が示唆された。

純チタンの既製ポストへの応用 —— 金属鑄造コア用ポストとしての適応性 ——

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チタンを既製ポストとして応用することを試み, 本報ではまず金属鑄造コア用ポストとしての適応性について検討するために, 機械的性質ならびにコア鑄造による影響について現在市販されている Ni-Cr 合金および 18-8 ステンレス鋼の既製ポストと比較した。

純 Ti の素材線材は冷間引抜後焼なまし処理を施されており, 機械的性質はほぼ金合金 Type III に相当する。Ni-Cr 合金と 18-8 ステンレス鋼ポストのかたさおよび引張強さは Ti ポストよりかなり大きい, コア鑄造過

程での加熱によって焼なましを受けて, かたさは Ti ポストを下回るようになり, 強さにも大きな差は認められなくなった。しかし, 純 Ti は 1,300°C まで加熱すると, 酸化などによって著しくもろくなってしまう。Ti ポスト上への他合金コアの鑄造性は非常に良好であり, 1,000°C まではもろさも生じなかったことから, 鑄込みが 1,000°C 前後までで完了する合金コアに対しては, Ti ポストは有用であると考えられる。

充てん用コンポジットレジンに関する研究 第 1 報 光重合型コンポジットレジンの硬化特性

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市販されている光重合型コンポジットレジン 18 種類について改良型オシレーティングレオメータを使用して硬化時間の測定をおこない、試料の厚さ、光照射時間、試料と光照射器先端部との距離が硬化時間におよぼす影響を調べた。また硬化時の歯軸方向における重合収縮率を測定し、各材料のフィラー含有率と重合収縮率との関係について検討した。

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メーカーが指示する最大厚さ（最大重合深度）の試料の場合、硬化時間は短いもので 0.9 分、長いもので 2.5 分であった。また硬化時間は試料の厚さ、光照射時間、試料と光照射器先端部との距離によって変化した。各材料の歯軸方向における重合収縮率は 1.5～2.8 % (試料厚さ 2.5 mm) の範囲であったが、フィラー含有率と重合収縮率との間には有意な相関はみられなかった。

充填用コンポジットレジンに関する研究

第 2 報 光重合コンポジットレジンの物理的並びに機械的性質

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18 種類の市販光重合コンポジットレジンにメーカー指示にしたがって重合し、その物理的並びに機械的性質を測定するとともに、それらの物性間の相関性を検討した。

メーカー指示の照射時間をまもって重合した試料の硬さは、照射後も増加し、24 時間後にはほぼ一定値に達する傾向を示した。

硬さと弾性率はフィラーの充填率に大きく依存した。

一方、圧縮強さとフィラー充填率の間には相関性は認められなかった。また光重合レジンに光を照射した表面から硬化するために、重合時のレジンの収縮量の殆どが窩底部に生じていた。歯ブラシ摩耗においては、無機物フィラーの含有量が増加すると耐摩耗性が増加する傾向が認められた。

コンポジットレジンの組成および重合方式が象牙質窩壁適合性に与える影響について

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コンポジットレジンと象牙質窩壁との間の良好な接着性を得るためには適切な歯面処理を行ない強力な bonding system を用いることの他に、窩壁に密着して重合収縮するレジン量を窩洞表面から補うためのコンポジットレジンのフローが重要な条件となる。本実験においては、このような条件を満足するレジンを試作する目的でベースレジン内に UDMA と 3G を含み、66～74 % のフィラーを含有する可視光線重合型コンポジットを試作しこ

れらの象牙質に対する接着性を市販の化学重合型、可視光線重合型コンポジットレジンと比較した。その結果、最良の接着性は 35 % HEMA と Clearfil New Bond および 4META を含有する試作ボンディングエイジェントを用いて填塞した化学重合型マクロフィラーコンポジットレジンの組合せにおいて得られ、試作可視光線重合型コンポジットの中では、フィラー量 70 %, ベースレジンに 3G を含有しない場合に良好な結果が得られた。