Clinical Evaluation of The Handling Properties of Photo-activated Composite Resin Pastes

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Abstract

The purpose of this investigation was to examine the relationship between measured values (rheological properties) of composite pastes, such as viscosity coefficient, peeling rate and external force (objective measurements), and a favorable property of such pastes discriminated by touch by dentists (subjective measurements). The rheological properties were examined using a balance beam plastometer. The subjective measurements were performed by eighteen young dentists and repeated four times. The subjective measurements related to four characteristics; hardness of paste (A), retention of shape (B), self-bonding properties (C) and release properties from an instrument for filling (D). There was a significant correlation between the log of the external force ($\log F$) and the log of the peeling rate ($\log \nu$) in all materials ($r=0.950\sim0.997$). A moderate correlation in all materials, except Degufill ultra, Heliomolar, Litefil II A and Silux plus was also found between the log of the coefficient of viscosity ($\log \eta$) and $\log \nu$ ($r=0.870\sim0.995$). The coefficient of correlation obtained from regression lines between the ratios of favorable properties ($\log PA$, $\log PB$ and $\log PD$) in the subjective test and $\log F$ or $\log \eta$ showed a good approximation with a quadratic equation.

Key words: Handling properties/Composite resin paste/Peeling rate/Rheological properties/
Subjective test

Introduction

Recently, photo-activated composite resins have been frequently used as polymer-based filling and restorative materials. These materials provide sufficient working time,

polymerize easily by light-irradiation, and possess superior mechanical properties. Although many physical and mechanical properties of photo-activated composite resins subsequent to setting are strictly standardized in the International Organization for Standard¹⁾, there is still no description concerning the handling properties of the composite resin pastes except a prescription of a film thickness measurement. The handling properties of photo-activated composite resin pastes are important properties of filling and restorative materials. In the case of commercially available photo-activated composite resins, the kinds of filler, filler particle size, filler contents and base monomers vary considerably from one product to another. Therefore, it is speculated that these differences have a substantial effect upon the clinical handling properties of a resin paste²⁻⁷⁾.

In the case of the low viscosity material, such as composite resin paste, the flow property exceedingly affected the testing speed. Although many reports are available regarding testing speed, proper speed in clinical use is still unknown. Therefore, sensory test was incorporated in the determination of suitable speed.

The purpose of this investigation was to examine the relationship between the rheological properties determined by a balance beam plastometer (objective measurements) and four properties (hardness, retention of shape, self-bonding property and release property) discriminated by finger touch of dentists (subjective measurements) and to discuss the suitable test speed.

Materials and methods

Materials

Materials used in this study were thirteen commercially available photo-activated composite resins as listed in Table 1, together with manufacturer, batch number and code.

Table 1 Materials used in this study

Materials	Manufacturer	Batch No.	Code
Clearfil AP-X	Kuraray, Japan	0423AE	CL
Degufill ultra	Degussa AG, Germany	1011	DE
Durafill	Kulzer, Germany	630167	DU
Estilux	Kulzer, Germany	84143	EL
Estio LC	GC, Japan	970808	ES
Heliomolar	Vivadent, Germany	70729	HE
Heliosit	Vivadent, Germany	20184	HS
Light Activated	Teledyne Getz, USA	72583	LA
Restorative			
Litefil II A	Shofu, Japan	79799	LI
Palfique Estelite	Tokuyama, Japan	14557	PA
Plurafil-super	Litema, Germany	40545	PL
Prisma.fil compules	Caulk, USA	40185	PE
Silux plus	3M, USA	19970605	SI

Measurement of peeling rate and apparent viscosity

The measurement of peeling rate and apparent viscosity was performed by using an experimental balance beam plastometer as shown in Fig. 1. This apparatus consists of 5 parts: (1) micrometer (a) (minimum reading 0.01 mm) for determining specimen thickness, upper and lower platten for fixing specimen paste, weight for peeling specimen from platten; (2) micrometer (b) for holding the beam in a horizontal position until the start of the measurement; (3) a balanced weight for fine adjustment; (4) an electric transducer for detecting the elongation or peeling of the specimen and (5) an amplifier connected to a pen-recorder. The experiment was performed by turning a thumbscrew of micrometer (b) and by way of releasing a specimen from the upper or lower platten.

The coefficient of viscosity (η) of each specimen was calculated on the basis of results obtained from an elongation-time curve, using the following Stefan's equation^{8,9}.

$$\eta = \frac{4Fh_1^2h_2^2}{3\pi a^4\nu(h_2^2 - h_1^2)}$$

Where a is the radius of the specimen, h_1 is the initial specimen thickness, h_2 is the specimen thickness after t s, F is the load (weight) and ν is the peeling rate.

The peeling rate was calculated by determining the elongation of resin paste per unit time obtained by using four or five peeling loads (100, 200, 300, 400 and 500 gr for all materials except Heliosit and Silux plus, 30, 50, 80 and 100 gr for Heliosit and 120, 150, 200 and 250 gr for Silux plus. All measurements were repeated three times at a temperature of $23 \pm 1.0^\circ\text{C}$.

Fig. 1 Schematic representation of apparatus used.

Discrimination test by touch by dentists using an instrument for resin filling

Each dentist performed an operation by filling each of 13 photo-activated composite resin pastes into a cavity of an epoxy resin tooth prepared in advance, according to standard techniques and then determined by touching to confirm whether the handling properties during filling were favorable (P) or not (W). The subjective measurements were determined by the following four characteristics and referred to the method indicated by Scott Blair^{10,11)}:

- A. Hardness of paste during operation
- B. Retention of shape
- C. Self-bonding properties of paste to cavity wall of epoxy resin tooth
- D. Release properties of paste from an instrument for filling

These items were performed using the recording paper shown in Fig. 2. These subjective measurements were performed by eighteen young dentists (each with more than four years clinical experience) and repeated four times for each dentist. The results of four subjective measurements were individually summarized with the ratio (PA, PB, PC, or PD) judged as being favorable for handling¹²⁻¹⁴⁾.

Sensory test recording paper on set tissue conditioner

Please mix each material 15 sec and spread on acrylic plate
using spatula immediately

Date:

Experience:

Examiner:

Occupation:

Code	Hardness		Retention property		Self-bonding		Release property	
	Good	Wrong	Good	Wrong	Good	Wrong	Good	Wrong
CL	☐	☐	☐	☐	☐	☐	☐	☐
DE	☐	☐	☐	☐	☐	☐	☐	☐
DU	☐	☐	☐	☐	☐	☐	☐	☐
EL	☐	☐	☐	☐	☐	☐	☐	☐
ES	☐	☐	☐	☐	☐	☐	☐	☐
HE	☐	☐	☐	☐	☐	☐	☐	☐
HS	☐	☐	☐	☐	☐	☐	☐	☐
LA	☐	☐	☐	☐	☐	☐	☐	☐
LI	☐	☐	☐	☐	☐	☐	☐	☐
PA	☐	☐	☐	☐	☐	☐	☐	☐
PL	☐	☐	☐	☐	☐	☐	☐	☐
PE	☐	☐	☐	☐	☐	☐	☐	☐
SI	☐	☐	☐	☐	☐	☐	☐	☐

Fig. 2 Recording paper used in sensory test.

Results

Peeling rate and apparent viscosity

The relationship between the external force ($\log F$) and the peeling rate ($\log \nu$) of 13 photo-activated composite resin pastes is shown in Fig. 3. There was a significant correlation ($r=0.950\sim0.997$) between $\log F$ and $\log \nu$ in all resin pastes, and the external force increased linearly with the increase of peeling rate. The coefficient of viscosity was determined using the results obtained from a load-elongation curve and Stefan's equation. The variation in coefficient of viscosity with peeling rate is shown in Fig. 4. The coefficient of viscosity in all materials except of HS and LI decreased slightly with increased peeling rate. There was a significant correlation between the log of the coefficient of viscosity ($\log \eta$) and the log of the peeling rate ($\log \nu$) ($r=0.870\sim0.995$) in all materials, except DE, HE, LI and SI. The variation of $\log \eta$ with $\log \nu$ for DE, HE, LI, and SI was markedly small.

Subjective measurement

In the subjective measurement C, there was no correlation between the ratio (Pc %) judged a favorable property and the external force (F) or coefficient of viscosity (η). However, in the subjective measurement of A, B and D, there was significant correlation between the log of each ratio ($\log P_A$, $\log P_B$ and $\log P_D$) and $\log (\log F)$ or $\log (\log \eta)$.

Fig. 3 Variation of external force ($\log F$) with peeling rate ($\log \nu$) of thirteen photo-activated composite resin pastes.

Fig. 4 Variation of viscosity coefficient ($\log \eta$) with peeling rate ($\log \nu$) of thirteen photo-activated composite resin pastes.

Fig. 5 Relationship between $\log P$ (%) and $\log (\log F)$ in three discrimination tests (A, B and D).

Fig. 6 Relationship between $\log P$ (%) and $\log (\log \eta)$ in three discrimination tests (A, B and D).

Fig. 7 Variation of correlation coefficient (r) obtained between the results of discrimination test and external force (F) with peeling rate ($\log \nu$).

Fig. 8 Variation of correlation coefficient (r) obtained between the results of discrimination test and viscosity coefficient (η) with peeling rate ($\log \nu$).

When the value of $\log \nu$ was -0.75 , these results were shown in Fig. 5 and 6. The coefficient of correlation (r) between each $\log P$ ($\log P_A$, $\log P_B$ and $\log P_D$) in the measurement of A, B and D (except C) and $\log F$ was 0.90. Furthermore, a moderate correlation was estimated between each $\log P$ and $\log \eta$, and the respective coefficient of correlation (r) was 0.87 for A and 0.86 for B and D. The variation of the coefficient of correlation (r) between each $\log P$ obtained by subjective measurements of A, B and D and $\log F$ or $\log \eta$ with $\log \nu$ is shown in Fig. 7 and 8. In the measurements of A, B and D, the coefficient of correlation (r) and $\log \nu$ held a good approximation with a quadratic equation ($Y = -0.175X^2 - 0.277 + 0.828$ for $\log P_A$ with $\log F$, $Y = -0.175X^2 - 0.331X + 0.782$ for $\log P_B$ with $\log F$ and $Y = -0.127X^2 - 0.217X + 0.728$ for $\log P_C$ with $\log F$) and reached a peak of $\log \nu$ ranging from 0.79 to 0.95. A similar tendency was also obtained with the apparent viscosity ($\log \eta$), and a good approximation with a quadratic equation ($Y = -0.141X^2 - 0.213X + 0.814$ for $\log P_A$, $Y = -0.145X^2 - 0.256X + 0.768$ for $\log P_B$ and $Y = 0.118X^2 - 0.17X + 0.96$ for $\log P_C$) was observed. The peak coefficient of correlation (r) was at a value of $\log \nu$ ranging from 0.72 to 0.88.

Discussion

The external force (F) increased with increased peeling rate (ν) and a significant correlation between the two factors ($\log F$ & $\log \nu$) was obtained (Fig. 3). Furthermore, this relationship also held between the $\log$ of the apparent viscosity ($\log \eta$) and $\log \nu$ (Fig. 4). The $\log \eta$ of all materials, except HS and LI, decreased with increased $\log \nu$. It is thought that these materials possess Non-Newtonian flow property as the apparent viscosity decreases with an increasing rate of load. The $\log \eta$ of two materials (HS and LI) increased slightly with increased $\log \nu$. It is thought that these materials have the flow properties as a dilatant fluid. Almost no variation of $\log \eta$ with $\log \nu$ was seen for three materials (DE, HE and SI). From this fact and from the behavior shown in Fig. 3, it is estimated that these materials show Newtonian flow properties. These three flow properties have some influence on handling property. Therefore, it is required that the change of viscosity with increasing rate of load is small.

There is a law of S. S. Stevens¹⁵⁻¹⁷⁾ as to the relationship between the magnitude of a sensation and the intensity of a stimulus. The law indicates that a linear relationship is established between the $\log$ of the magnitude of a sensation and the $\log$ of the intensity of a stimulus.

In this investigation, the respective ratio of the subjective measurements ($P\%$) judged as being handling properties favorable in the measurements of A, B, C and D were examined. It can be seen that this ratio ($P\%$) obtained in the subjective measurements is equivalent to the magnitude of sensation and the values obtained in the objective measurements, such as external force (F), apparent viscosity (η) and peeling rate (ν), and corresponds to the intensity of stimulus. The relationship between the results of the objective measurements and those of the subjective measurements is shown in Fig. 5 and 6. There was a sufficient correlation between each $\log P$ and $\log (\log F)$ or $\log (\log \eta)$ in all characteristics except

C. It is presumed that dentists may have a view on favorable handling properties of composite resin paste through their own clinical experience. The values of $\log F$ for nine arbitrary points of $\log \nu$ were determined by using a regression line obtained between $\log F$ and $\log \nu$, and the correlation between each $\log F$ and $\log P$ was also examined. The coefficient of correlation (r) and $\log \nu$ held a good approximation with the quadratic equation as shown in Fig. 7. When the value of $\log \nu$ was -0.79 for A, -0.95 for B and -0.86 for D, the respective correlation coefficient reached its maximum value. A similar relationship was obtained using $\log \eta$ instead of $\log F$ as well (Fig. 8). When the value of $\log \nu$ was -0.75 for the A, -0.88 for B and -0.72 for D, the respective correlation coefficient reached its maximum value. The times obtained using this arrangement are 0.18s for A, 0.13s for B and 0.19s for D, because the peeling rate (ν) is replaced by $1/t$. This fact indicates that many dentists can judge by their finger touch, whether the handling properties of a photo-activated composite resin paste is favorable or not. It can be seen that the magnitude of tension force or the test speed is exceedingly important when discussing the handling property of paste.

Conclusion

Commercially available photo-activated composite resin pastes were classified into three flow properties (Non-Newtonian flow, flow of dilatant fluid and Newtonian flow). These properties affect the handling property of composite resin paste. Therefore, the materials which indicated the small change in viscosity might be preferable. From the result of sensory test, it became clear that the best speed is exceedingly important when discussing the property of paste material.

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光重合型コンポジットレジンペーストの操作性に関する臨床的評価

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抄 録

本研究の目的は、光重合型コンポジットレジンペーストの粘性率、引き剥ぎ速度、外力のような測定可能な値（客観的テスト、レオロジー的性質）と歯科医師の指感覚による計測値（主観的テスト）間の関係を調べ、コンポジットレジンペーストの好ましい性質を決定することである。レオロジー的性質は天秤式ブラストメーターを用いて計測した。主観的テストは、4年以上の経験をもつ18名の歯科医師で4回繰り返して行った。主観的テストは次の4種類である。A. ペーストの硬さ、B. 形態の保持力、C. 粘着性、D. 充填時の器具からのリリース特性。対数目盛で表示した外力（log F）と引き剥ぎ速度（log ν ）との間には全材料で十分な相関関係（ $r=0.950\sim0.997$ ）が認められた。デグフィルウルトラ、ヘリオモーター、ライトフィルII A、シラックスプラスを除いて他の全ての材料においても粘性率（log η ）とlog ν の間に十分な相関関係（ $r=0.870\sim0.995$ ）が認められた、主観的テストで好ましいとした性質、log P_A, log P_B, log P_Dとlog F また log η との関係はそれぞれ二次相関で近似できた。

キーワード：操作性/コンポジットレジンペースト/引き剥ぎ速度/レオロジー的性質/主観的テスト