

Relation between Handling Characteristics and Application Time of Four Photo-polymerized Resin Composites

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Objective: To investigate the relation between handling characteristics and application time of four composite materials with subjectively different viscosities.

Methods: Eight experienced faculty members placed one Class II and one Class IV restoration in a random sequence into pre-prepared plastic teeth mounted on a typodont model, each using 4 types of composites (Herculite Précis (M1), Kerr; Tetric N-Ceram (M2), Ivoclar Vivadent; Filtek Z350 (M3), 3M-ESPE; Charisma Opal (M4), HareausKulzer), resulting in a total of 64 restorations. The application process was filmed with a high definition video camera. Two evaluators watched the recordings in a random sequence as well, timed the composite application and wrote down their observations, which were dichotomised into positive and negative ones. Application times were analysed with a two-way Kruskal Wallis test (time x dentist) and the observation data were analysed with a chi-square test ($P < 0.05$).

Results: Materials did not differ in their application time ($P > 0.05$). The mean application time was 12 ¼ minutes for the Class II and 9 ¾ minutes for Class IV restorations. However, there were statistically significant differences between the dentists in terms of application time. The observation data showed no significant difference between Class II and Class IV restorations but there were significant material differences ($P < 0.05$). M2 yielded 6% negative observations, while the other materials were between 35% and 38%.

Conclusion: There was no association between the handling characteristics of the tested composite resins and the speed of application. However, one of the tested materials (M2) showed significantly less problems in the application process.

Keywords: Composite direct application, handling, observation study, in vitro, viscosity

Before polymerisation, resin-based composite materials (hereon: composites) could be considered as very viscous liquids. Due to physical interactions between the filler particles and the resinous

matrix, they also show more or less thixotropic behaviour. Therefore, their rheological properties are of paramount importance for easy handling. In the early days with auto-polymerised composites, researchers were concerned about the mixing properties¹ or the setting time². With modern rheological measuring equipment they can be characterised quite well and differences between the different products can be shown³⁻⁵. Opdam et al⁶ measured the consistency of composites using the ISO test for elastomeric impression materials (ISO 4823) and were able to discriminate between the different composite products. The same was possible by forcing a rod at a high constant speed into the material and measuring necessary load⁷. However, it is difficult to deduce some clinical handling properties from these data since the testing methodologies do not reflect clinical situations. Furthermore, when photo-polymerised composites emerged on the dental markets

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with the option for clinicians to determine the timing of polymerisation, it was realised that the rheological properties of these materials were influencing their handling characteristics. Yet, there were no tests for determining the handling characteristics. One way to do this is the laboratory approach. Al-Sharaa & Watts⁸ and Kaleem et al⁹ used the stickiness as the measurable property to determine clinical handling by contacting the surface with a flat rod, then separating it from that surface and measuring either the height to that point or the force needed for separation. Other researchers measured the slumping of the composite after being shaped into a specific form with laser scans by comparing the initial shape with the resulting shape as a function of time^{10,11}.

Another way to look at handling of materials is to have the materials being used by dentists and ask their opinion with questionnaires. This is easy, but due to the large variation in subjective opinions of dentists, it is difficult to get reliable data. It is better to have measurable parameters, as done by Degrange M (Battle of the Bonds, unpublished data) where dentists had to bond composites to bovine dentin and subsequently the immediate shear bond strength was determined. However, they found that the variation between dentists was much larger than the variation between the different adhesives. Therefore, one can conclude that the dentist's knowledge and skills are very important, and will translate in a better quality of the restorations.

It is believed that a good initial quality of restorations will be mirrored in their longevity. Since the good quality is the least sum of errors, it is worth looking at the ease of application of composite materials that can be influenced by the handling characteristics of the materials.

The objective of this study therefore was to investigate the application behaviour of four composite materials with subjectively different viscosities. It was hypothesised that the more difficult the application technique or the material property, the longer it would take to place a restoration. Therefore, timing the application could reveal some differences between the composite materials. Furthermore, the sum of positive/negative observations is a measure of the handling properties. Analysing anonymous observation protocols of restoration placements in a free format could reveal some differences between composite materials tested. The null hypothesis tested was that handling characteristics and application duration of four resin composite materials would not show significant difference.

Materials and methods

Materials used in this study are listed in Table 1. Eight experienced faculty members from the Department of Restorative Dental Sciences at the University of Florida placed one Class II and one Class IV restoration, each using 4 types of composites into a pre-prepared typo-

Table 1 Materials, batch numbers and manufacturers

Material	Composition (manufacturer data)	Batch no.	Manufacturer
Tetric N-Ceram A2	Urethane dimethacrylate, bis-GMA 15% Ethoxylated bis-EMA 3.8% Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide 63.5% Prepolymers 17.0% Additives, stabilizers, catalysts, pigments 0.7%	P72199	Ivoclar Vivadent
Tetric N-Bond	Urethane dimethacrylate, bis-GMA, dimethacrylate, hydroethyl methacrylate, phosphonic acid acrylate 80% Nano-Fillers (SiO ₂) <1% Ethanol < 20% Initiators and Stabilizers <2%	P75516	Ivoclar Vivadent
Charisma Opal A2	Bis-GMA and TEGDMA fillers 58%-vol (approx 78%-weight): Ba-Al-B-F-Si Glass (0.02-2µm) Pyrogenic SiO ₂ (0.02-0.07µm) Mean filler particle size: 0.7µm Stabiliser Photoinitiators (a.o. campherquinone) Pigments	010025 (8x)	Haraeus Kulzer

Material	Composition (manufacturer data)	Batch no.	Manufacturer
Charisma Opal A2	Same	010023 (5x)	Haraeus Kulzer
Charisma Opal A2	Same	010024 (2x)	Haraeus Kulzer
Gluma 2Bond	Ethanol / water Methacrylates (a.o. HEMA) Acidic monomers (a.o. 4-META) Glutarialdehyde Fillers Additives	010031	Haraeus Kulzer
Filtek Z350 XT A2 Body Shade	Diurethane Dimethacrylate (UDMA) 1–10% Bisphenol A Polyethylene Glycol Diether Dimethacrylate 1–10% Bisphenol A Diglycidyl Ether Dimethacrylate (bis-GMA) 1–10% Polyethylene Glycol Dimethacrylate 0.5% Triethylene Glycol Dimethacrylate (TEGDMA) <5% 2,6-Di-Tert-Butyl-P-Cresol <0.5% Silane treated Ceramic 60–80% Silane treated Silica 1–10% Silane treated Zirconia 1–10%	N321220	3M ESPE
Adper Single Bond2	Bisphenol A Diglycidyl Ether Dimethacrylate (bis-GMA) 15–25% 2-Hydroxyethyl Methacrylate 15–25% Decamethylene Dimethacrylate 5–15% Ethanol 10–15% Water 10–15% Silane treated Silica 5–15% 2 Propenoic Acid, 2Methyl-, reaction products with 1,10-Decanediol and Phosphorous Oxyde (P ₂ O ₅) 1–10% Copolymer of Acrylic and Itaconic Acid 1–5% Dimethylaminobenzoat(-4) <2 Champhorquinone <2 (Dimethylamino)Ethyl Methacrylate <2 Methyl Ethyl Ketone <0.5	N319974	3M ESPE
Herculite Précis A2e	Dimethacrylate resins Grounded barium aluminoborosilicate, 0.4 µm Prepolymerized filler Radio-opaque filler Fumed silica	3793312	Kerr Corporation
Optibond S	Alkyl dimethacrylate resin 55–60% Ba Al borosilicate glass 5–10% Fumed silica (silicon dioxide) 5–10% Sodium hexafluorosilicate 0.1–1% Ethyl alcohol 20–25%	4168405	Kerr Corporation
OptraPol Lens	Silicone Micro fine diamond crystallites 72%	NL 1789	Ivoclar Vivadent
OptraPol Cup	Same	NL 1788	Ivoclar Vivadent
OptraPol Lage Flame	Same	NL 1790	Ivoclar Vivadent
OptraPol Small Flame	Same	NL 1828	Ivoclar Vivadent



Fig 1 Setup with the Video camera in the Simulation Lab.



Fig 2 Affixed mounted typodont, aligned materials, and markers.

dont tooth mounted on a typodont model. This resulted in $2 \times 4 \times 8 = 64$ restorations placed. The entire application process was filmed with a video camera mounted in a fixed position in relation to the typodont model. Two stations were set up in the Simulation Laboratory, where electric hand pieces were available for the polishing process (Fig 1). For every dentist, a randomised sequence for the materials used was prescribed using a random number table¹². The dentists were instructed to use the centripetal technique for Class II restorations. They were given metal circular matrix bands, wooden wedges and Toffelmeier matrix band holders. For the Class IV restorations, silicon stents were provided. They were informed on the objective of the study and given an instruction sheet.

The materials were aligned according to the randomisation and the typodont marked with numbers, visible in the film, identifying the materials used (Fig 2).

In the first step, the application time of the composite, from the appearance of the first increment on the screen to the end of the last polymerisation step was timed using the time code of the video film. The results were analysed with the Kruskal Wallis test (SAS 9.2, SAS Institute).

In the second step, two dentists watched the application and protocolled the observations for every increment applied in a free format. They were blinded regarding the materials. Both evaluators had to agree on the protocolled observations. In the instance of disputed results, the film was rewound and the scene was observed repeatedly until an agreement was reached. The sequence of the observation was randomised in a different way than the sequence of application of the restorations.

In the third step, each protocol of the 8 dentists was regrouped according to cavity class and material, and coloured with the electronic highlighter: no colour = descriptive facts, green: positive observations, red = negative observations.

Finally, the positive and negative observations per dentist and material were counted and analysed with the chi-square test (SAS 9.2, SAS Institute).

Results

The results of the composite application times for Class II and IV restorations are shown in Table 2 and 3 and in Figs 3 and 4. There were no significant differences between the materials ($P > 0.05$), but the application duration varied significantly between the operators (dentists) ($P > 0.05$). Since the dentists' behaviour was not a topic of the study, this was not further investigated with post hoc tests.

The pooled data (Class II and IV) of the evaluation of the observation protocols are presented in Table 4. A significant difference was observed between Material 2 and those of others ($P > 0.001$). This is obvious looking at the percentage of negative comments from the total sum of comments per material. Material 2 yielded 6% negative comments, while the others varied from 35% to 38% negative comments.

Discussion

The materials included in the study were selected based on their perceived consistency, since the thixotropic behaviour of the composites interferes with viscosity measurements. Material 1 was considered the least

Table 2 Mean application times of 8 dentists in minutes placing Class II and IV composites. Grouped according to materials

Composite	Class II	Class IV
	Mean ± SD	Mean ± SD
1	11.32 ± 4.24	9.21 ± 3.40
2	11.37 ± 4.72	10.86 ± 4.59
3	13.53 ± 3.76	9.66 ± 4.81
4	12.35 ± 3.58	9.22 ± 3.16
Mean	12.17 ± 1.02	9.74 ± 0.83
Kruskal Wallis rows and columns	NS ($P > 0.05$)	

Materials: 1 = Herculite Précis, 2 = Tetric N-Ceram, 3 = Filtek Z350 XT, 4 = Charisma Opal

Table 3 Application times of 8 dentists in minutes placing Class II and IV composites (Herculite Précis, Tetric N-Ceram, Filtek Z350 XT, Charisma Opal). Grouped according to dentists

Dentist	Class II	Class IV
	Mean ±SD	Mean ±SD
1	8.75 ± 0.93	6.38 ± 1.18
2	7.69 ± 0.83	8.88 ± 1.53
3	9.32 ± 2.83	11.36 ± 3.36
4	15.83 ± 2.69	13.63 ± 1.96
5	18.50 ± 0.46	6.53 ± 1.23
6	11.19 ± 3.10	9.75 ± 7.26
7	12.51 ± 3.2	13.75 ± 0.65
8	14.00 ± 1.46	7.63 ± 1.97
Mean	12.17 ± 1.94	9.74 ± 2.13
Kruskal Wallis	$P < 0.05$	$P < 0.05$

Fig 3 Application time (minutes) by dentist (x-axis) and composites for Class II composite restorations. Kruskal Wallis: Dentists $P < 0.05$, Material NS. (Materials: 1 = Herculite Précis, 2 = Tetric N-Ceram, 3 = Filtek Z350 XT, 4 = Charisma Opal).

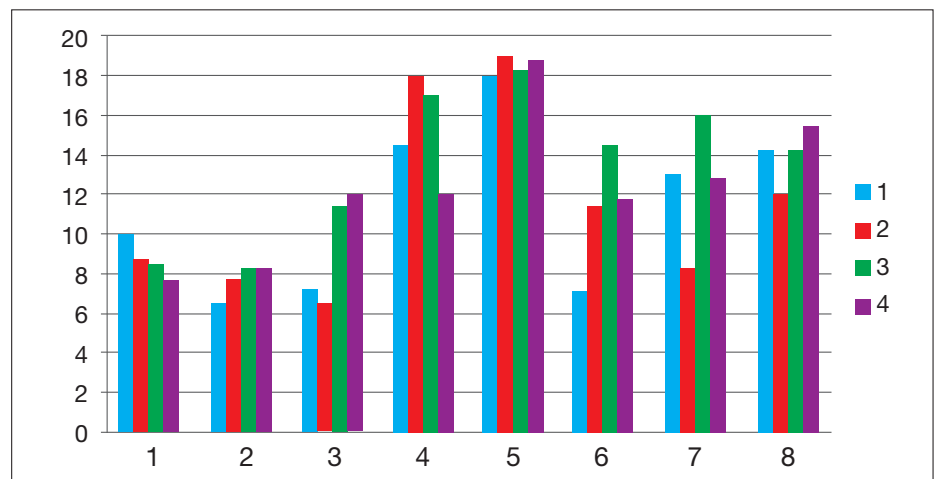


Fig 4 Application time (minutes) by dentist (x-axis) and material in Minutes for Class IV composite restorations. Kruskal Wallis: Dentists $P < 0.05$, Material NS. (Materials: 1 = Herculite Précis, 2 = Tetric N-Ceram, 3 = Filtek Z350 XT, 4 = Charisma Opal).

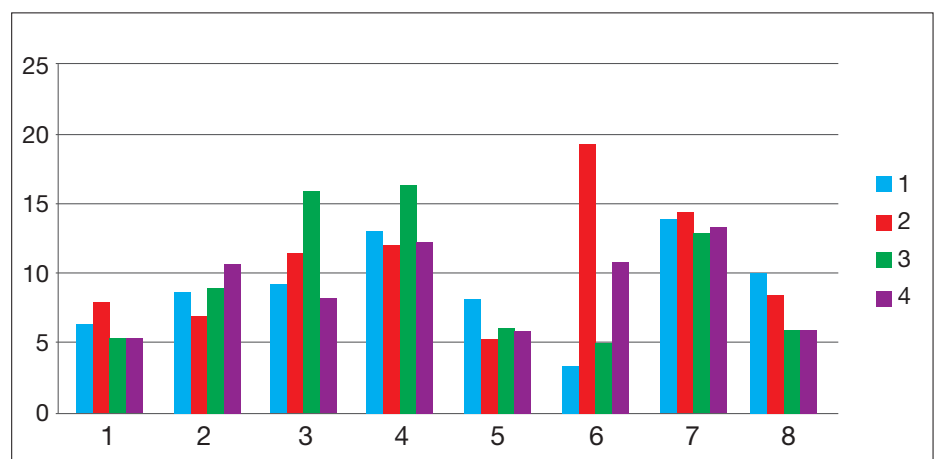




Table 4 Sum of positive and negative comments after dichotomization for the different materials

Composite	Sum of positive comments	Sum of negative comments	Total	Negative comments of total comments (%)
1	49	29	78	37%
2	98	6	104	6%
3	48	30	78	38%
4	60	33	93	35%
Chi square	$P < 0.0001$			

Materials: 1 = Herculite Précis, 2 = Tetric N-Ceram, 3 = Filtek Z350 XT, 4 = Charisma Opal

flowable and Material 4 the most flowable. The other two materials were positioned in between Material 1 and 4 in terms of ‘viscosity’. The composites tested have different filler loads and different monomer matrices (Table 1) that might have influenced their consistency^{4,7}. Observing with a video camera was thought to be more objective than asking the practitioners their opinion on the handling properties of the composites. By using standardised tooth preparations, offering standard instruments, giving them a ‘new’ material (the standard material in the clinic is Filtek Z 250) and requiring them to use the centripetal technique, all participating practitioners faced the same conditions, thus decreasing variability. We did not standardise the number of increments, since we wanted to be as close as possible to normal conditions in the dental office. We assumed that the dentists would know that the maximal thickness of an increment for a light curing composite would be 2 mm. Random assignments for the placement of the restorations, as well as the sequence of the evaluation, possibly eliminated the learning curves. One drawback of this *in vitro* study is that typodont teeth were used instead of human teeth. However, they were wetted with the corresponding adhesive system of the composites applied, thus mimicking the situation in real teeth. Another drawback is the ambient temperature in the simulation laboratory, which is less than the tooth temperature in the mouth. Since the temperature influences the rheological properties of composites⁹, this aspect could have affected the outcome.

Since the placement of the adhesive resin was not within the scope of the study and no visible differences were observed, this step was neither evaluated for timing, nor for handling aspects. The polishing of the restorations, which should be a standard procedure, was not seen as such by the dentists. Although the operators were provided with Soflex discs (3M ESPE)

and OptraPol (Ivoclar) polishers, each operator used a different approach. Therefore, this procedure was not comparable anymore and it was decided to disregard these parts of the videos when considering the duration required for each restoration.

The null hypothesis could only partially be rejected, since, against the expectation, we could not find any application time effect between the composites that had definitely different consistencies as seen in the application videos. The dentists very obviously quickly changed their application modes when they encountered difficulties like slumping or stickiness. Despite using dentists from the same institution, their individual application techniques varied substantially, which is documented by the significant time differences for the same task. This was even more pronounced for the polishing process, which was the reason not to evaluate this part of the composite application.

Looking at the most often observed negative events (Fig 5), stickiness of the composite was the largest problem by far, being expressed by the observation ‘sticks to instrument/detaches from tooth’. This is by far the most difficult task for a composite manufacturer to solve. In principle, the material should be sticky in order to wet the primed tooth surface well since extended manipulation creates bad margin quality¹³, on the other hand, it should not stick to the instrument. Since the stickiness is temperature dependant⁸, being more pronounced at higher temperatures, one should expect even more problems *in vivo*, since teeth are known to have a temperature slightly below 37°C in contrast to the room temperature used in this experiment (approximately 20°C). In the clinical situation, the longer the composite is manipulated, the more problems are to be expected due to viscosity changes in the material as a function of time and temperature.

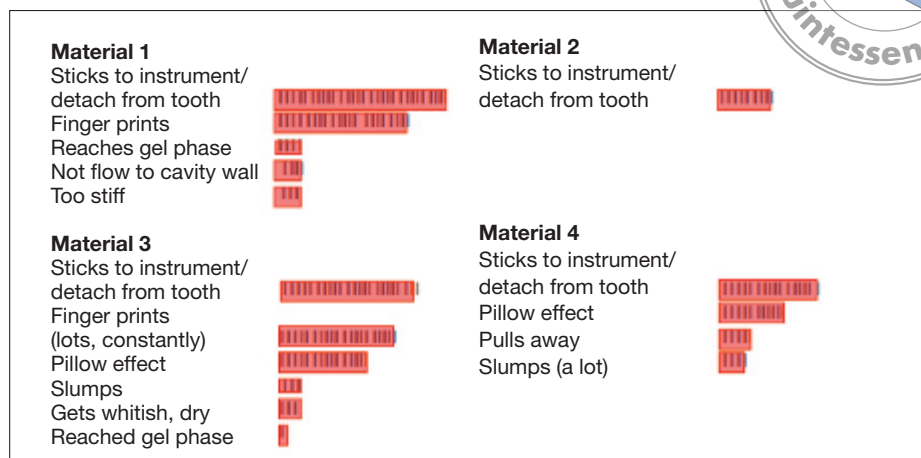


Fig 5 Frequency distribution for the most common negative observations.

‘Fingerprints’ were also quite often seen, especially in the more thixotropic Materials 1 and 3 (Fig 5). ‘Fingerprints’ means multiple small instrument marks that do not disappear after a short time but are maintained into the surface structure, which makes subsequent polishing more time consuming. In addition, this may lead to discrepancies or poor adaptation between resin composite layers. Again, these rheological properties depend on the resin/filler ratio^{4,7}, the filler composition and particle size distribution¹⁴ (Tetric N-Collection, Ivoclar Vivadent 2012), and the quality of the silanization^{14,15}.

Conclusion and clinical significance

Based on the results of this *in vitro* study, one can conclude that at least for the materials tested, there is no association between the handling characteristics and the speed of application. However, one of the tested materials showed significantly less problems in the application process. It can be hypothesised that due to the lower probability to make errors with a material that is easier to apply, a superior margin quality could be the result of this behaviour. *In vitro* margin analysis studies and *in vivo* longevity studies should be initiated to confirm this hypothesis.

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