

RESEARCH ARTICLE

Comparison of Film Thickness of Restorative Composites Considered for Thermo-Modified Cementation

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ABSTRACT

Objective: This study proposes an updated method for the film thickness of 21 resin composites used as cements.

Materials and Methods: A load of 30 N was subjected to two glass plates stacked on a heated base to simulate clinically relevant pressure and temperature during the seating of inlays/onlays/veneers. The axial position of the actuator was recorded before and after placing 22.15 mm³ of resin composite between the glass slides. The difference between the two measurements was calculated for each specimen. A total of 18 condensable restoratives, 1 injectable restorative, and 2 resin cements were tested, 5 specimens each ($n = 5$, $N = 105$). One-way ANOVA was performed for both variables and Games-Howell post hoc test was used for pairwise comparisons.

Results: The film thickness range was 6–200 μm, with the lowest values for restorative composites (AP-X, Z250, and Herculite XRV Incisal LT with 6–8 μm), followed by Gradia Direct (25 μm) and flowable/injectable composite resins, including designated cements (15–27 μm). Inspiro, Ultradent Transcend Universal, and ENA Hri dentin stood below 50 μm (range 28–49 μm). The other materials (range 81–200 μm) cannot be recommended for thermo-modified cementation.

Conclusions: The initial viscosity of restorative composites is not an indication of the film thickness when used for thermo-modified cementation. There is a wide choice of appropriate materials available on the market.

Clinical Significance: Some restorative composites are able to generate very thin films and can be used as cement with numerous advantages when compared to designated flowable or dual-cure cements.

1 | Introduction

Successful delivery of inlays/onlays and veneers implies that a perfect continuum has been established between the restoration and the tooth through material-specific (resin composite, polymer-infiltrated ceramic network, and various ceramics) and tissue-specific (enamel/dentin) conditioning [1, 2]. Traditional cementation implies that a certain resistance form (geometry) of the tooth preparation is present and that the restoration possesses a high intrinsic strength. The reason is that conventional cements have limited mechanical

and adhesive properties in addition to their poor resistance to dissolution compared to adhesive cements such as resin-modified glass ionomer and resin composites. The definition of the word cementation, however, also implies adherence/bond between the substrates involved. Hence, resin-based cements are recommended for the adhesive delivery of semi-direct CAD/CAM and indirect restorations such as inlays, onlays, and veneers because those restorations are intrinsically less resistant than traditional porcelain-fused-to-metal or zirconia crowns. The optimal adhesive and mechanical properties of resin-based cements can assure the long-term perennity

of totally nonretentive onlays, overlays, and new types of veneers made of brittle materials (Figure 1) [3].

Because of film thickness requirements to be able to seat the restoration, resin-based cements are usually flowable materials. To the authors' knowledge, Dr. Mark Friedman in Beverly Hills (CA) may have been the first author to propose using a restorative resin composite as a luting agent, in this case, Herculite XRV shade Incisal LT, to successfully deliver porcelain veneers in the 1990s [4, 5]. Simultaneously in Europe in 1995, a publication by Besek et al. explored the possibility of using a classic restorative resin composite to deliver ceramic inlays [6]. At that time, the author used the Cerec 1 CAD/CAM system. The poor fit of those original CAD/CAM ceramic inlays may have prompted them to compensate for the large, exposed gap at the occlusal surface with a luting agent that could better resist occlusal load and wear. A microhybrid restorative resin composite, Brilliant Lux Incisal (Coltene), was used and compared to traditional resin cement. It was concluded that dual-curing resin had no advantages versus light-curing with respect to polymerization rate when seating Cerec inlays. In addition, the overall handling of the light-curing composite resin was judged to be easier than that of the dual-cure material (unlimited time for seating and cleaning excesses before polymerization). As a result, the use of solely light polymerized restorative composites as cements gained popularity and significant changes in restorative concepts started to emerge (Figure 2). Because the 1990s were dominated by the use of microhybrid composites, with particle distribution ranging from 0.1 to a maximum of 10 μm , combining ultrasonic energy [7, 8] and/or preheating the material [9] would provide great assistance in seating the restorations

and still obtaining perfect seating [10], hence the term thermo-modified cementation. In the 2000s, however, new nanohybrid composite resin formulations prompted the inclusion of large clusters and prepolymerized filler complexes (PPFCs) with a particle size of up to 20–30 μm (Figure 3).

While all restorative resin composites demonstrate a significant reduction in viscosity when preheated, they remain generally more viscous than flowable materials and most of them provide film thickness that exceeds the 50- μm norm under ISO 4049 standard (150N between glass slides, room temperature) [11, 12]. Hence, there is a need for a new method for testing those materials under conditions that reflect their clinical applications, including realistic cementation forces [10], body temperature, and material preheating.

This study aims to investigate the film thickness of various types of preheated composite resins under a new method of better simulating clinical conditions (pressure and temperature) and provide the clinician with clear guidelines for the selection of the most appropriate luting agent. The null hypothesis is that the various types of resin composites will present similar film thicknesses regardless of their viscosity, composition, and filler type.

2 | Materials and Methods

As is the case with ISO 4049 standard for film thickness measurements [12], each material was tested between two flat square glass plates (22×22 mm). However, to better understand the behavior of the cement in more realistic clinical



FIGURE 1 | Successful bonded feldspathic porcelain restoration (A) on the left central incisor after 20 years (B and C). Note the perfect color match with the intact right central incisor despite the extremely nonretentive design and large incisal edge span of almost 5 mm at the distal aspect. In addition, this restoration happened to have cracked preoperatively (A, arrow). It was delivered regardless of the crack using a restorative resin composite as a luting agent, not a designated resin cement. Smartphone pictures reproduced with permission [2].

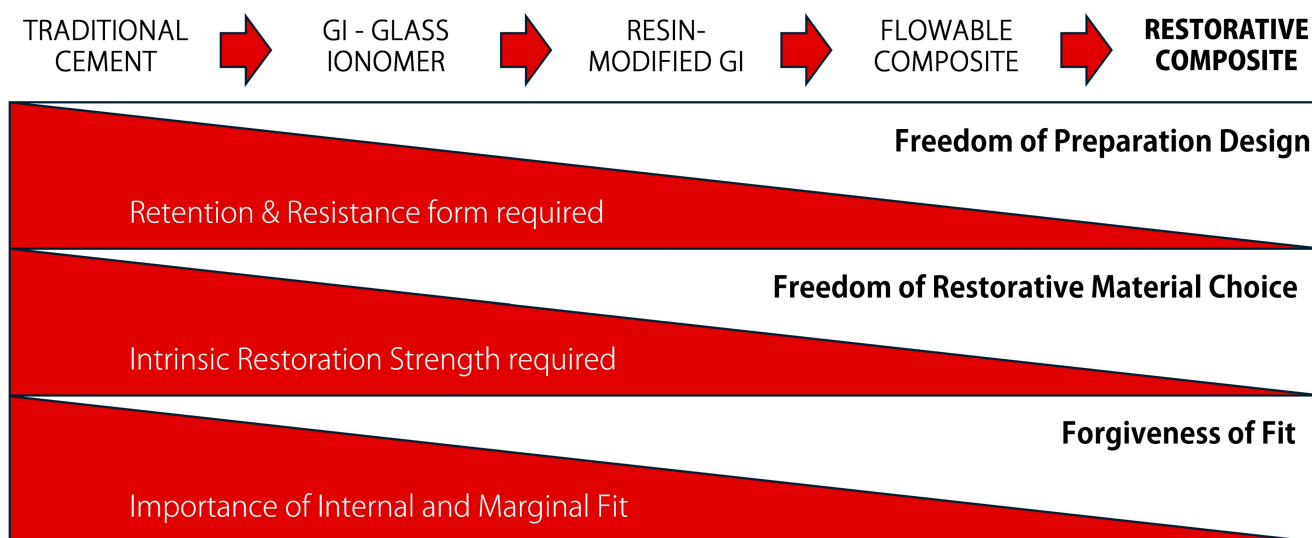


FIGURE 2 | Changing concepts about bonded indirect restorations in the 1990s.

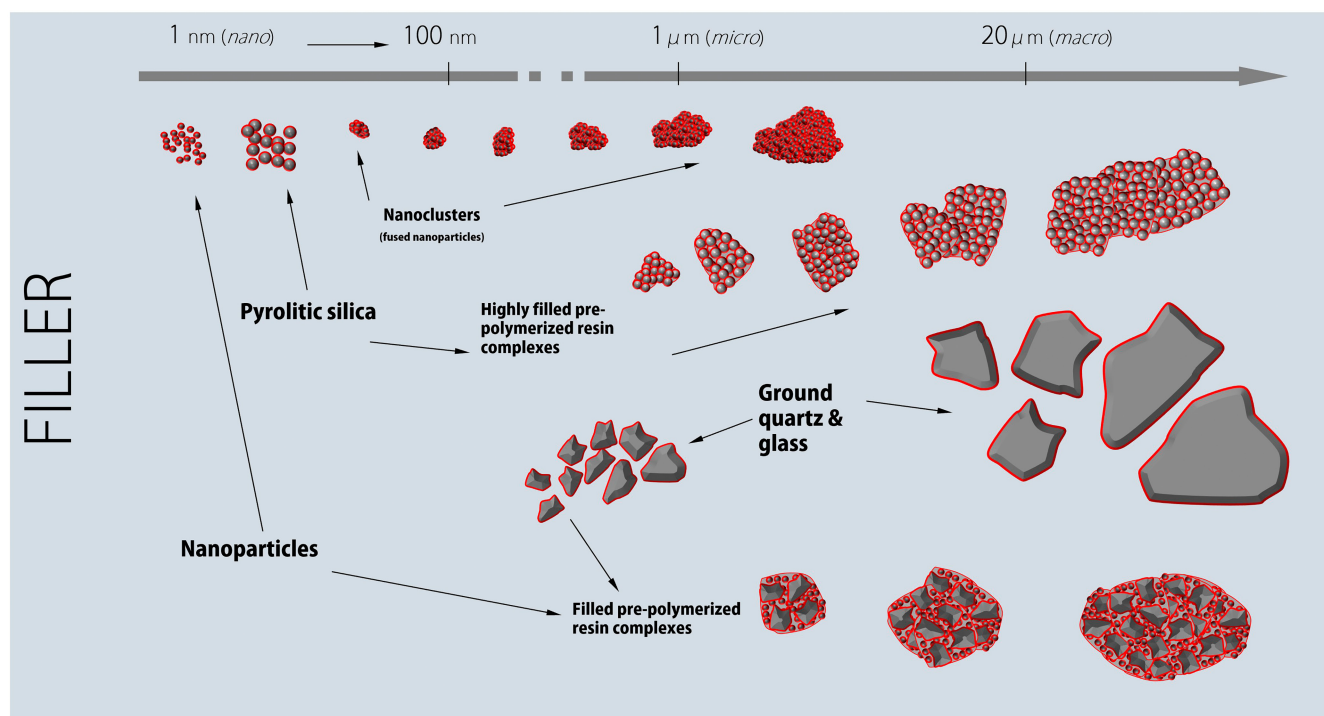


FIGURE 3 | Contemporary filler technology and a wide range of particle sizes (reproduced with permission) [2].

conditions, the measurements were carried out at body temperature with the glass plates seated on a heated base to simulate body temperature (Calset device set at 37°C/98°F, Addent). A load of 30 N was chosen, instead of 150 N (ISO 4049), to better simulate the pressure used when seating inlays, onlays, or veneers [10]. The Acumen III (MTS Systems) was used to subject a maximum load of 30 N to the two glass plates stacked on the heated base and the axial position of the actuator was recorded (baseline). The load was repeated after placing a calibrated volume of the various cement candidates between the glass slides. Beforehand, each material was pre-heated in another Calset device for 3 min at 68°C/155°F before being dispensed into a cylindrical silicon mold for calibration

of the volume used (22.15 mm [3]) then placed between the glass slides for compression at 30 N (Figure 4). The difference between the axial position at baseline (without resin composite) and during the experimental measurement was calculated to obtain the film thickness for each material. A total of 21 products were tested (Table 1) with 5 specimens each ($n = 5$, $N = 105$), including 18 condensable restoratives, 1 injectable restorative, and 2 resin cements (Table 1). The circular spread (diameter) of the material was also measured by recording the width and height of the resin composite surface with a stainless steel digital caliper, and averaging the two measurements. The measurement for the spread was maxed at 22 mm (limits of the glass slide).

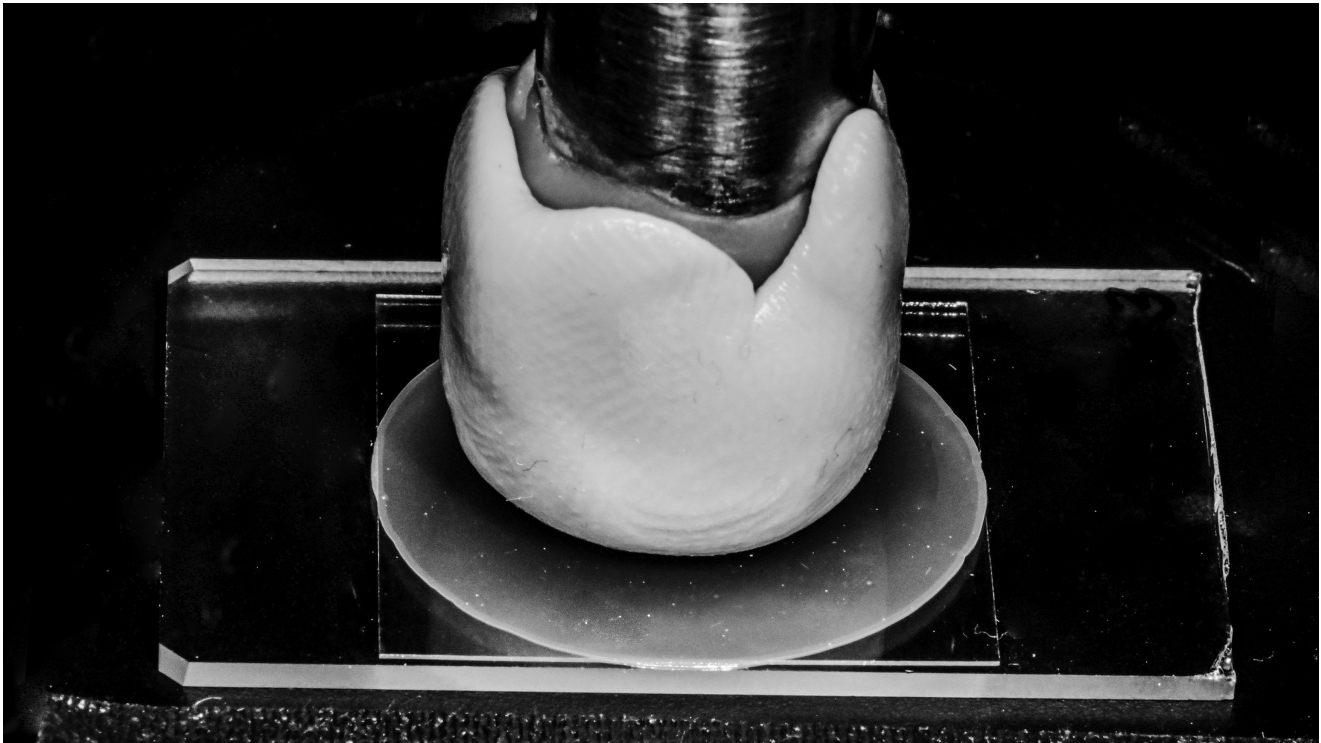


FIGURE 4 | Experimental setup with actuator applying 30N of load to the glass plates and resin composite Z250 in-between. Note that the material is so thin that it spreads to the edge of the 22-mm glass slide. The actuator tip was covered with polyvinilsiloxane to simulate finger pressure and prevent fracture of the glass slides.

Since the sample size was small ($n=5$), determining the distribution of the film thickness data was important for choosing an appropriate statistical method. Therefore, a Shapiro–Wilk test was performed and did not show evidence of nonnormality ($W>0.776$, p value >0.05). Based on this outcome, and after visual examination of the histogram and QQ plot, it was decided to run a parametric test. Levene's test was conducted to assess the equality of variance for the film thickness and spread across the different groups. The results of Levene's test indicate that the assumption of homogeneity of variances was violated: $F(20,84)=5.451$, $p=0.00$ for film thickness and $F(20,84)=4.282$, $p=0.00$ for spread. Consequently, it opted for the Games-Howell post hoc test, which does not assume equal variances, to conduct pairwise comparisons after one-way ANOVA. Also, the mean and standard deviation were used to summarize the film thickness and spread.

3 | Results

The mean and standard deviations for film thickness and circular spread are presented in Table 1 and Figures 5 and 6, and reflect a wide range of results, from $>200\mu\text{m}$ down to few micrometers for film thickness and 12 to 22mm for the circular spread. Examples of specimens are presented in Figure 7. The one-way ANOVA revealed statistical differences for film thickness and circular spread between groups: $F(20,84)=227.34$, $p=0.000$ for film thickness and $F(20,84)=47.19$, $p=0.000$ for spread. The Games-Howell post hoc test was used for pairwise comparison and statistical differences ($p<0.05$) are presented in different letters between groups in Figures 5

and 6. Considering the $50\text{-}\mu\text{m}$ threshold from ISO 4049 standard [12], with the adapted method of this study, 12 pre-heated restorative resin composites and the 2 resin composite cements from this study would be suitable for cementation: ENA HRi (UD1), Transcend Universal (A1D), Gradia Direct Anterior (A2), Inspiro Enamel (WR), Inspiro Dentin (Bi3), Transcend Universal (UB), Gradia Direct X (X-A1), Variolink Esthetic LC (Neutral), Gradia Direct X (X-BW), G-aenial Universal Injectable (BW), G-CEM Veneer (Bleach), Herculite XRV- INCISAL (LT), Z-250 (A1), and Clearfil AP-X (XL). The resin composites Tetric Powerfill (IVB) and Evanesce Silk (B1UX) provided means of film thickness just above the $50\text{-}\mu\text{m}$ threshold. The other 6 restorative resin composites are not suitable for cementation as their mean film thickness ranged from 81.2 to $200.6\mu\text{m}$, which may imply improper seating of the restoration.

The circular spread correlated with the film thickness, as the groups with more spread provided the thinner film thickness.

4 | Discussion

The mean film thickness of a wide range of resin-based restorative composites, injectable composite, and composite cements was investigated. The null hypothesis can be rejected because significant differences were observed.

Materials were presented in Table 1 according to their composition even though such data are extremely difficult to obtain. The authors did a considerable amount of research to obtain

TABLE 1 | Resin composites tested, their composition (manufacturer's data unless in parenthesis), and experimental results for film thickness and circular spread.

Resin-based composite (color)	Manufacturer	Resin type	Filler type	Filler content (%)	Average particle size (µm)	Average circular spread in mm (SD)*	Average film thickness in µm (SD)*
Herculite XRV-Enamel (A2)	Kerr	Microhybrid	Quartz and glass Barium glass Silica	79 weight	0.6 aver. 0.5–1 0.04	12.0 (1.5)	200.6 (21.9)
G-aenial A'CHORD (AO1)	GC	Ultrafine hybrid (heterogeneous)	Barium glass Silicon dioxide PPFC	70 vol. 82 weight	0.01–3.2	15.35 (2.1)	167.2 (6.9)
Simplishade (LT)	Kerr	Nanohybrid heterogeneous	Mixed oxides Ytterbium fluoride Barium glass Silica PPFCs	64.5 vol. 81.5 weight	0.04–6.0	17.2 (1.5)	102.4 (5.5)
G-aenial Sculpt (JE)	GC	Nanohybrid	Barium glass Silicone dioxide PPFCs	69 vol.	0.02–1.0	17.9 (1.3)	94.4 (20.3)
Essentia (MD)	GC	Micro-HDR hybrid (heterogeneous)	Barium glass Fluoroaluminosilicate glass Silicone dioxide PPFCs	63 vol. 76 weight	0.02–2.9 10	18.4 (0.4)	81.2 (4.1)
Tetric Powerfill (IVB)	Ivoclar Vivadent	Nanohybrid	Barium glass Ytterbium trifluoride. Si-Zr mixed oxide, and copolymers	54 vol. 79 weight	0.6 aver. 0.11–15.5	17.9 (0.5)	60 (5.5)
Evanescence Silk (B1UX)	Clinician's choice—	Nanohybrid	Proprietary nanofillers PPFCs	66 vol. 81 weight	0.6 aver. 3–20	20.1 (0.7)	54.6 (5.0)
ENA HRi (UD1)	Micerium	Microhybrid	Microparticles Silicone dioxide	53 vol. 75 weight	0.7 aver. 0.04	18.8 (0.7)	48.8 (2.2)
Transcend Universal (A1D)	Ultradent	Microhybrid	Silica Silane-treated silica	61 vol. 79 weight	0.9 aver.	20.7 (0.8)	43.6 (2.2)
Gradia Direct Anterior (A2)	GC	Microhybrid	Feldspar Silicone dioxide	64 vol. 73 weight	0.85 aver 0.02–2.9	21.2 (0.2)	38.8 (2.3)

(Continues)

TABLE 1 | (Continued)

Resin-based composite (color)	Manufacturer	Resin type	Filler type	Filler content (%)	Average particle size (µm)	Average circular spread in mm (SD)*	Average film thickness in µm (SD)*
Inspiro Enamel (WR)	EdelweissDR	Nanohybrid Homogeneous	Radiopaque glass filler Nonagglomerated silica nanofillers	65 vol. 83 weight	0.02–3.0	20.0 (0.7)	38.4 (4.5)
Inspiro Dentin (Bi3)	EdelweissDR	Nanohybrid Homogeneous	Radiopaque glass filler Silica nanofillers	65 vol. 83 weight	0.02–3.0	18.8 (0.3)	38 (5.9)
Transcend Universal (UB)	Ultradent	Microhybrid	Silica Silane-treated silica	61 vol. 79 weight	0.9 aver.	20.8 (0.3)	37.6 (7.5)
Gradia Direct X (X-A1)	GC	Microhybrid	Fluoroaluminosilicate glass Feldspar Silicene dioxide	65 vol. 77 weight	0.02–2.9	21.2 (0.4)	28.4 (2.1)
Variolink Esthetic LC (Neutral)	Ivoclar Vivadent	Light-cured cement	Ytterbium trifluoride. Si-Zr mixed oxide	40 vol.	0.15–15.5	22.0 (0.0)**	27 (2.9)
Gradia Direct X (X-BW)	GC	Microhybrid	Feldspar Silicene dioxide	64 vol. 77 weight	0.02–2.9	21.8 (0.4)	25 (1.9)
G-aenial Universal Injectable (BW)	GC	Flowable resin composite	Barium glass Silicene dioxide	46 vol. 69 weight	0.01–0.5	21.1 (0.9)	18.4 (1.5)
G-CEM Veneer (Bleach)	GC	Light-cured cement	Barium glass Silicene dioxide	46 vol. 69 weight	0.01–0.5	22.0 (0.0)**	15.2 (0.8)
Herculite XRV-INCISAL (LT)	Kerr	Microhybrid	Quartz and glass Pyrolytic silica	70 weight	0.02–1.0	22.0 (0.0)**	8.4 (1.1)
Z-250 (A1)	3M ESPE	Small-particle spheroidal (3 M)	Patented milled spheroidal zirconia-silica particles	85 weight	0.6 aver.	22.0 (0.0)**	7.6 (1.1)
Clearfil AP-X (XL)	Kuraray	Microhybrid	Barium glass. Pyrolytic silica	71 vol. 85 weight	0.02–17	22.0 (0.0)**	6.6 (1.8)

*Data obtained in this study.
**Reached upper limit of spread.

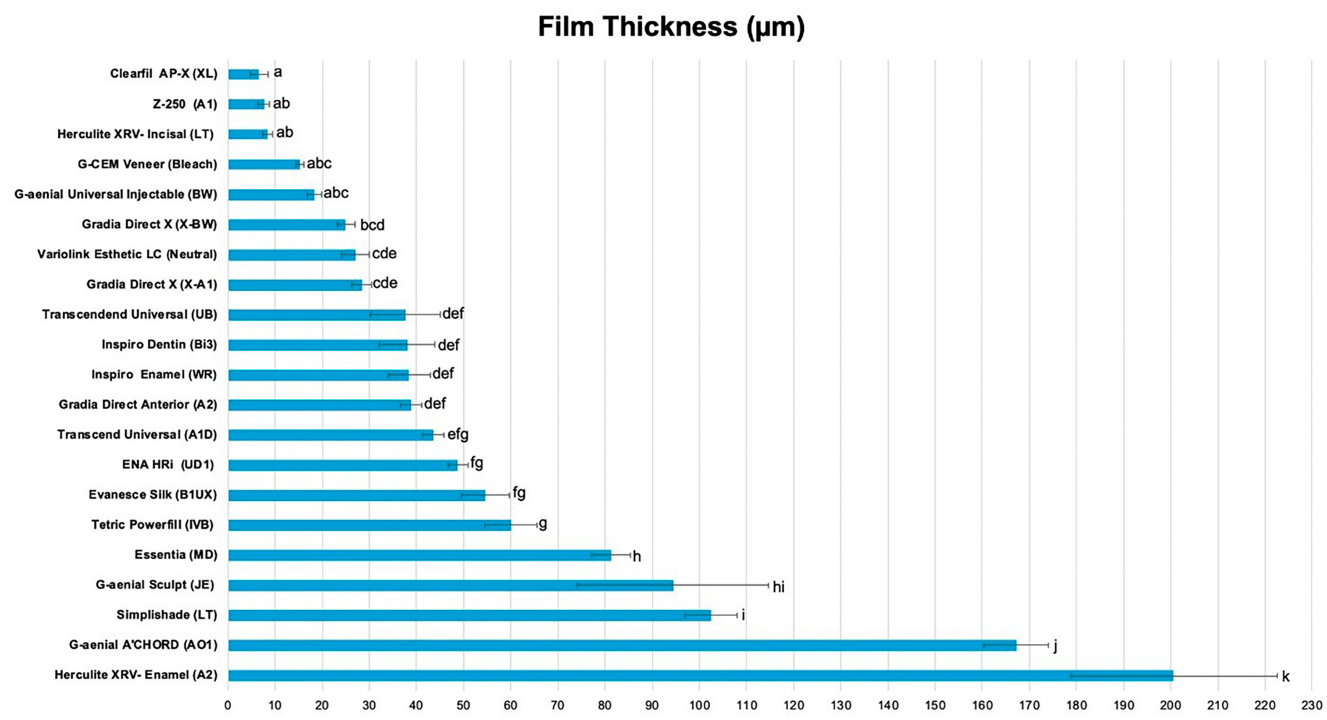


FIGURE 5 | Mean film thickness in micrometers for each material tested. Different letters indicate statistical differences between materials.

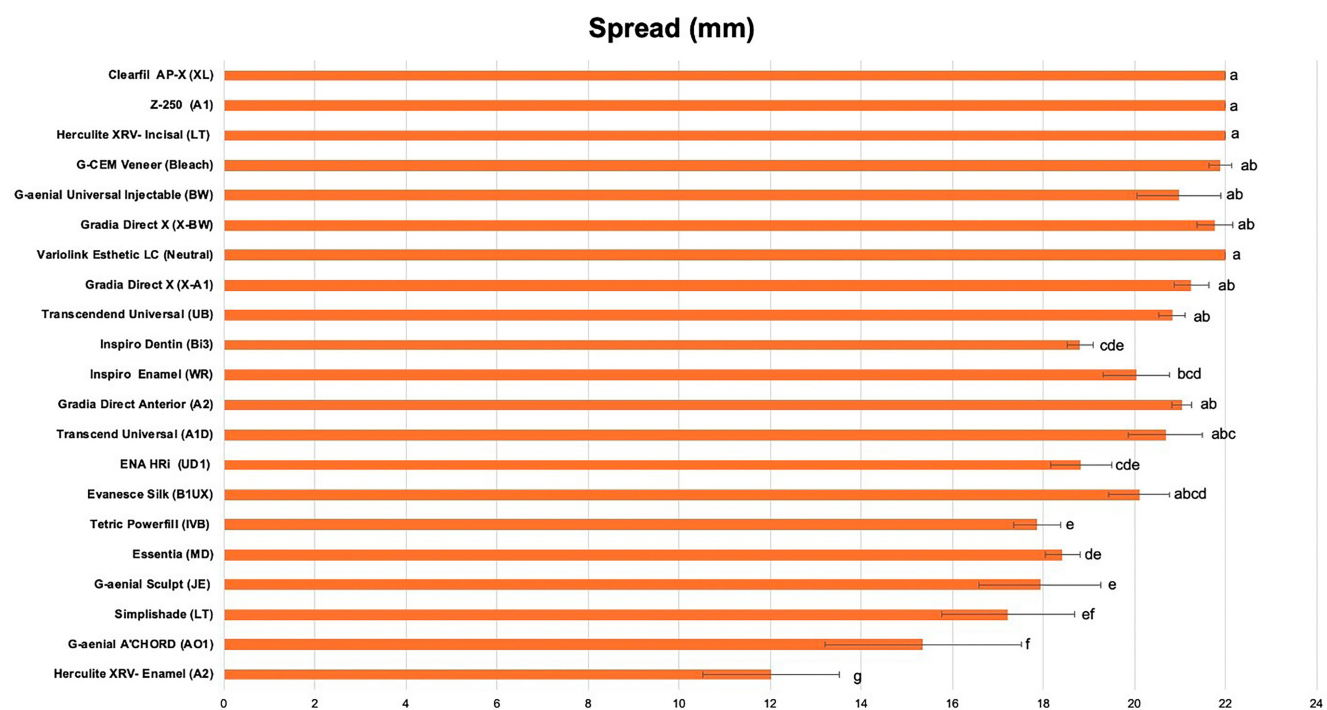


FIGURE 6 | Mean circular spread in millimeters for each material tested. Different letters indicate statistical differences between materials.

detailed information about each product, but there remains always a possibility of missing information or proprietary information not divulged by the manufacturers. Although the experiment did not include rheological measurements, the authors can ascertain that apparently more viscous materials, such as Inspiro Enamel WR, could easily produce film thicknesses of 38.4, while apparently less viscous Herculite

XRV Enamel A2 displayed 200.6 µm. In contrast for the same brand and product, Herculite XRV Incisal LT, which was recommended as a veneer cement by Mark Friedman in the 1990s [4], provided a film thickness of 8.4 µm. Unfortunately, this material, due to its filler composition, cannot be recommended as luting agent because it is not radiopaque enough. The same lack of radiopacity affects GC Gradia Anterior. On

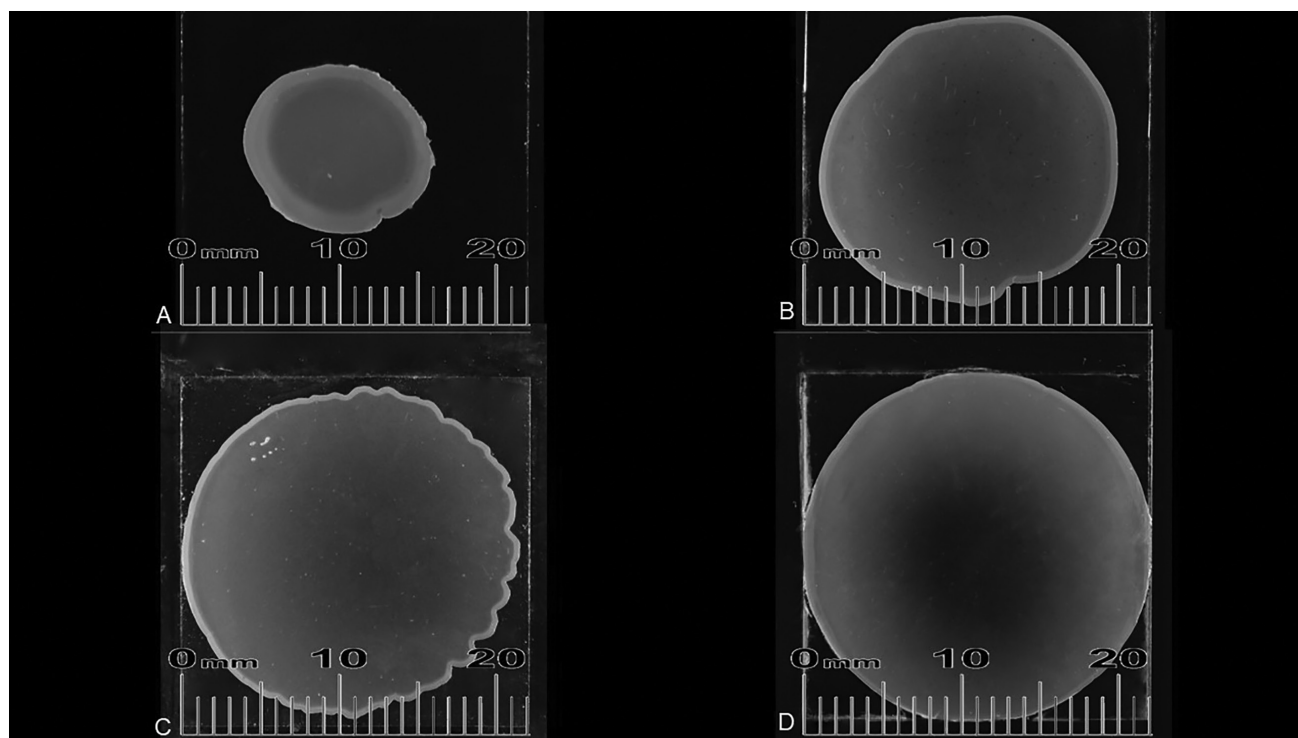


FIGURE 7 | Examples of specimens after the test (compression 30N at 37°C/98°F). (A) Herculite XRV shade Enamel A2-FT 200.6µm, circular spread 12.0mm. (B) ENA Hri shade UD1-FT 48.8µm, circular spread 18.86 mm. (C) Gradia Direct X shade X-A1-FT 28.4µm, circular spread 21.4mm. (D) Z250 shade A1-FT 7.6µm, circular spread 22mm.

the other hand, Clearfil AP-X shade XL (Kuraray) was able to generate the smallest thickness (6.6µm), similar to Filtek Z250 (7.6µm). Both these restorative materials were able to supersede actual flowable resin composite cements, G-CEM Veneer (GC) and Variolink Esthetics LC (Ivoclar) with 15.2 and 27µm, respectively, and the GC G-aenial Universal Injectable (18.4µm), demonstrating that viscosity should not be correlated to film thickness. This is in accordance with other studies [10, 11]. Specifically, one flowable resin composite cement (RelyX Ultimate, 3M) prevented the seating of CAD/CAM inlays, while a more viscous restorative resin composite of the same manufacturer (Z100, 3M) allowed better seating of the same inlays [10]. Hence, viscosity alone does not capture the full complexity of how resin composites behave under load during cementation. Other factors may influence film thickness beyond the material's initial viscosity such as material composition, filler distribution, particle type/size/shape, and size of monomers in the resin matrix. Manufacturers do not typically disclose such information.

A reference material that has been widely recommended by clinicians and successfully used as a luting agent for veneers (including clinical studies) is ENA Hri dentin [13, 14]. This product still averaged 48.8µm. Other good restorative candidates for cement include GC Gradia Direct X shades A1 and shade BW with 28.4 and 25.0µm, respectively. It seems reasonable to establish that the threshold of 50µm is a very safe limit and that clinicians should ideally focus on materials with no greater film thickness. Nevertheless, the 50-µm threshold used in the ISO 4049 norm [12] was also used in this study to suggest the suitability of a resin-based material for the thermo-modified cementation technique. This threshold could be questioned for two reasons.

First, the 150N of the ISO norm is too high to simulate clinical seating forces [10]. Second, clinical studies indicate that the average marginal and internal gaps in veneers and crowns vary between 100 and 315µm [15–17]. In this context, it could be justified to increase that threshold to 120µm as suggested by other authors [18–20]. This would disqualify only two products in the current study.

Limiting the spread to less than 22mm was a potential limitation of this work. Yet, another criticism of the modified ISO method used in this article could be that it does not mimic exactly the cementation of a restoration, since flat glass plates are used, instead of anatomical tooth preparations with an indirect restoration. To clarify that potential problem, the authors used the same experimental setup (including the baseline measurement without cement) to seat three restorations on a typodont tooth with a veneer preparation (tooth 2.1., P-Occlusal, Brazil) using Herculite XRV Enamel A1, ENA Hri UD1, and Z250 A1 (Figure 7). The incomplete seating was obvious when using Herculite XRV Enamel and generated 254µm of axial displacement (elevation), while the veneer seated more than acceptably using ENA Hri UD1 (21µm axial displacement) and Z250 A1 (100% seated, no axial displacement) (Figure 8).

It is difficult to correlate the composition and filler content with the results. Some materials such as Simplishade feature very small fillers (0.005–0.4µm) but large film thickness (102.4µm) but also include a rheological modifier, which acts as a stabilizing network if left undisturbed. Under this condition, the apparent viscosity of the material is higher, which prevents material creep, commonly known as “slump”. Simplishade, alike G-aenial Sculpt and GC Essentia, may also contain PPFCs [21],

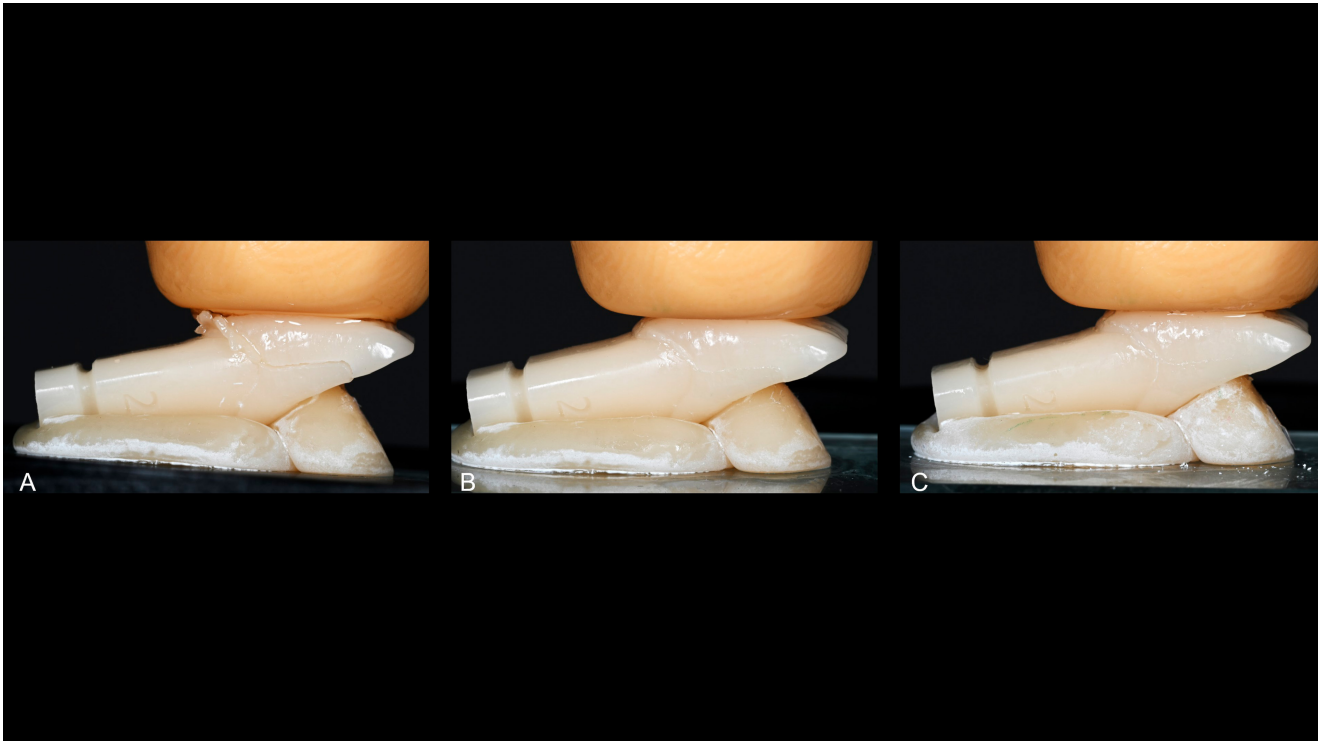


FIGURE 8 | Post hoc test using prefabricated veneers and typodont teeth with a veneer preparation and 30N seating load. (A) Herculite XRV shade Enamel A2—254µm axial displacement. (B) ENA HRi shade UD1—21µm elevation axial displacement. (C) Z250 shade A1—0µm, no axial displacement.

which appear to be absent from all materials with film thicknesses less than 50µm. It is therefore reasonable to conclude that restoratives with PFFCs should be avoided as cements, including those designated cements with nanofiller clusters, especially when cementing inlays [10].

When selected carefully, preheated restorative composites can be used as a cement, which will provide a multitude of advantages. The preheating of the material will initially facilitate the seating of the veneer/inlay/onlay, but it was demonstrated that the temperature of the composite resin will quickly drop [9, 22]. This is indirectly a benefit because the return to the original viscosity will facilitate the cleaning of the excesses. The unlimited working time is another reason for using solely light-polymerized materials, resulting in significantly less excesses and overhangs [23]. The use of restorative composites rather than resin cements also proved to increase the fatigue resistance of the bonded restorations such as laminate veneers [14] and limit the wear at the exposed interface (margin) [24], in addition to other positive effect on the marginal adaptation and strength [25, 26]. As previously mentioned, internal gaps in veneers and crowns can easily exceed 300µm. Such a gap justifies the use of cements with optimal physicochemical properties, which would logically favor restoratives composites over designated cements (flowable, dual-cure, etc.). In this context, color stability is another substantial advantage over dual-cure cements that are known for their color variation [27].

Using restorative composites as cements requires a specific workflow [28]. For instance, the etched/silanated ceramic must be wet with low-viscosity adhesive resin to assure proper bonding. This omission of this step does not appear to be as critical

when using designated cement/flowable composite resins [29]. Another useful clinical tip is to place the restoration preloaded with preheated composite into a heating device. Once the tooth preparation surface has been conditioned, it is ideal to seat the restoration within a delay of 15s, after which the temperature will decrease and the viscosity increase rapidly, stabilizing the restoration and facilitating the cleanup without the need for tack-curing [28]. One common lingering question when using solely light-polymerizing materials surrounds the depth of polymerization. This problem, however, has been demystified. In the presence of restorations thicker than 3mm, the polymerization time can be extended to 90s per surface. Using this approach, no difference in mechanical performance of thick onlays and overlays could be detected up to 7mm of thickness when comparing designated dual-cure cements and light-cure restorative materials [23, 30–33].

5 | Conclusions

A modified method was proposed to measure the film thickness of composite resin to be used as cements. The mean film thickness of direct composite resin restoratives, injectables, and cements ranged from 6 to 200µm. The lowest film thickness was generated by some restorative materials, followed by flowable/injectable composite resins, including designated cements. It is suggested that a restorative material must be in the 0–50µm range using the new norm. Some restorative materials, however, may be problematic for the thermo-modified cementation technique (range 50–200µm). Composite resin restorative materials need to be selected carefully to be used as cement. Their initial viscosity is not an indication of the film thickness when used for

the thermo-modified cementation. Materials with prepolymerized fillers should be avoided.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The authors have nothing to report.

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