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Influence of Shrinkage and Viscosity of Flowable Composite Liners on Cervical Microleakage of Class II Restorations: A Micro-CT Analysis

J Nie • AU Yap • XY Wang

Clinical Relevance

Interfacial integrity of Class II restorations is improved by the use of flowable composite liners, particularly those with low filler content. Giomer restorations had significantly less microleakage than those restored with nano-filled composites.

SUMMARY

This study determined the influence of shrinkage and viscosity of flowable composite liners on the cervical microleakage of Class II restorations using micro-CT. Seven composites of varying viscosities were selected and included five giomers (Shofu Beautifil II [BF], Flow Plus F00 and F03 [F00 and F03], Flow F02 and F10 [F02 and F10]) and 2 nano-filled composites (3M-ESPE Filtek Z350 [Z350] and Filtek Z350 Flowable [Z350F]). Polymerization shrinkage (n=7) was assessed with the Acuvol volumetric shrinkage analyzer while complex viscosity was determined with the advanced rheometric expansion system at 25°C. Standardized Class II restorations incorporating 1-mm horizontal layers of different flowable liners and 3-mm oblique layers of BF or Z350 were subjected to a silver nitrate test for 24 hours and examined using micro-CT. Microleakage was determined at 0.1-mm intervals from the buccal to lingual surfaces providing 30 sites per specimen and scored accordingly. Statistical analysis was performed with the one-way ANOVA, Kruskal-Wallis test, and Spearman's rho correlation at a significance level of $p<0.05$. Mean volumetric shrinkage ranged from 5.33±0.17% to 2.35±0.02% for F02 to Z350, respectively. The flowable materials had significantly higher shrinkage than did their sculptable counterparts (BF and Z350). Complex viscosities

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ranged from 9.65 to 4.20 (Z350 and F10, respectively) at a frequency of 10 rad/s and from 8.16 to 3.28 (Z350 and F03, respectively) for 100 rad/s. Giomer restorations had significantly less leakage than did those restored with nanofilled composites. No microleakage was observed with restorations lined with F02 or F10. The use of flowable liners reduced cervical microleakage of Class II restorations. Interfacial integrity of Class II restorations was significantly correlated with liner viscosity, filler volume, and shrinkage.

INTRODUCTION

Flowable composites were introduced in the mid-1990s.[1] They were developed to simplify placement procedures, enhance adaption to the internal surfaces of cavity preparations, improve cavity seal, and expand the clinical applications of resin-based composites. To decrease viscosity and increase flowability, filler loading of flowable composites was substantially reduced. Filler volume of flowable composites ranged from 37% to 53% compared with 50% to 70% of sculptable composite. While this allows flowable materials to be dispensed through fine-gauge needles, the higher resin content results in reduced strength, wear resistance, rigidity, and increased polymerization shrinkage.[2] Polymerization shrinkage stress at the tooth-restoration interface can lead to enamel fractures, interfacial debonding, and microleakage.[3,4] Microleakage may well cause marginal staining, postoperative sensitivity, secondary caries, pulpal pathology, and restoration failure.[5]

Flowable composite liners have been advocated as the first increment at the cervical or gingival floor of Class II restorations. They adapt well to microstructural irregularities of cavity preparations preceding sculptable composite placement. Several studies have reported a trend toward decreased microleakage in teeth restored with this technique.[6-8] In addition, a thinner layer of flowable composite liner appears to provide better marginal seal and adaptation and fewer voids.[9-11] Nevertheless, microleakage data from other authors do not support the use of flowable composite liners.[12]

This incongruity can be attributed somewhat to the fact that flowable composites are not a homogeneous group of materials. They vary significantly in terms of formulation, handling, and viscosity due to differences in resin and filler compositions. When resin content increases, flowability and shrinkage stress is higher, while elastic modulus is reduced.[13] The reduced material rigidity together with enhanced cavity adaption may mitigate the greater shrinkage stress accompanying the lower filler-volume fractions of flowable composites.

While studies have investigated the influence of polymerization shrinkage on microleakage of dental composites,[14-16] little is known about the impact of flowable composite viscosity on cervical microleakage. Nor has the efficacy of flowable giomers as liners been established. Giomers, offered in a wide range of viscosities, are based on prereacted glass ionomer (PRG) technology in which acid-reactive fluorosilicate glass is reacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and used as fillers. The objectives of this study were to determine the influence of shrinkage and viscosity of flowable composite liners on the cervical microleakage of Class II restorations using micro-CT. The relationships between polymerization shrinkage, viscosity, and microleakage were also established. The null hypotheses were as follows: 1) polymerization shrinkage and viscosity of flowable composite liners do not affect the cervical microleakage of Class II restorations and 2) there are no correlations between shrinkage, viscosity, and microleakage.

METHODS AND MATERIALS

Seven composite materials of varying viscosities were selected for this study including one sculptable giomer (Beautifil II [BF]) and four flowable giomers (Beautifil Flow Plus F00 [F00], Beautifil Flow Plus F03 [F03], Beautifil Flow F02 [F02], and Beautifil Flow F10 [F10]) in addition to a sculptable composite (Filtek Z350 [Z350]) and a flowable nano-filled composite (Filtek Z350 Flowable [Z350F]). The composite materials were used in conjunction with their corresponding conditioning/adhesive systems. The technical profiles of the materials evaluated are listed in Table 1. Information pertaining to material composition and filler volume were supplied by the manufacturers.

Volumetric Polymerization Shrinkage

The volumetic shrinkage of the composites was determined with the Acuvol volumetric shrinkage analyzer (X-81100P, Bisco, Schaumburg, IL, USA) at 25°C using the single-view mode. Seven specimens of each composite (n=7) were fabricated and tested. Flowable materials were syringed onto the Teflon pedestal and shaped into a hemisphere; sculptable materials were rolled into a ball and placed on the pedestal. The specimens were allowed to stand for 5

Table 1: *Technical Profiles of the Various Materials Evaluated*

Materials	Batch Number	Composition	Filler Volume (%)
Beautifil Flow Plus F00 (F00)	091013	Bis-GMA[a]	47.0
		TEGDMA	
		Aluminofluoro-borosilicate glass	
		Al_2O_3, DL-camphorquinone	
Beautifil Flow F02 (F02)	041156	Bis-GMA	34.6
		TEGDMA	
		Aluminofluoro-borosilicate glass	
		Al_2O_3, DL-camphorquinone	
Beautifil Flow Plus F03 (F03)	041004	Bis-GMA	46.3
		TEGDMA	
		Aluminofluoro-borosilicate glass	
		Al_2O_3, DL-camphorquinone	
Beautifil Flow F10 (F10)	041125	Bis-GMA	33.3
		TEGDMA	
		Aluminofluoro-borosilicate glass	
		Al_2O_3, DL-camphorquinone	
Beautifil II (BF)	061139	Bis-GMA	68.6
		TEGDMA	
		Aluminofluoro-borosilicate glass	
		Al_2O_3, DL-camphorquinone	
Filtek Z350 Flowable (Z350F)	N313982	Bis-GMA	55.0
		TEGDMA	
		Bis-EMA	
		Zirconia/silica filler	
Filtek Z350 (Z350)	N142553	Bis-GMA	59.5
		TEGDMA	
		Bis-EMA	
		UDMA	
		Zirconia/silica filler	

[a] Bis-GMA, bisphenylglycidyl dimethacrylate; TEGDMA, triethylenglycol dimethacrylate; Bis-EMA, bisphenol A ethoxylated dimethacrylate; UDMA, urethanedimethacrylate.

minutes before the initial precure volume (V1) was recorded. The specimens were then cured for 20 seconds using a LED curing light with an intensity of 500 mW/cm^2 (Bluphase, Ivoclar Vivadent, Shaan, Liechtenstein). The light cure tip was positioned 1 mm above the top of the specimens. Postcure volume (V2) was recorded 2 minutes after removal of the light source. The volumetric polymerization shrinkage of the materials was calculated according to the equation: polymerization shrinkage = (V1–V2)/V1×100%. Shrinkage data was subjected to normality testing with the Kolmogorov-Smirnov test. As data was normally distributed, it was consequently analyzed using the one-way ANOVA/LSD post hoc test at a significance level of α=0.05. Correlation between filler volume and shrinkage was analyzed using Pearson's correlation test (2-tailed, α=0.05).

Viscosity

Viscosity measurements were performed using an Advanced Rheometric Expansion System (ARES, TA Instruments, New Castle, DE, USA) at a temperature of 25°C. The parallel plates viscometer module with a diameter of 25 mm was utilized to determine the viscosity of the composites. The gap between the plates was set at 1 mm. Strain sweep tests were performed to check the range of uniform output signals to ensure that all measurements were carried out within the linear limit of each material's deformation. A time sweep test was then conducted to destroy the internal structure of the specimens and to reveal any potential thixotropic behavior. A frequency sweep test was subsequently performed from 0.01 to 100 rad/s to determine the variation of the complex viscosity (η*) as a function of frequency.[17]

Table 2: *Restorative Protocol for the Class II Restorations*

Group	Bonding system	Material Used in Each Layer	
		Horizontal Layer (1 mm)	Oblique Layer (3 mm)
F00-BF	FL-Bond II	F00	BF
F02-BF	FL-Bond II	F02	BF
F03-BF	FL-Bond II	F03	BF
F10-BF	FL-Bond II	F10	BF
BF-BF	FL-Bond II	BF	BF
Z350F-Z350	Adper Easy One	Z350F	Z350
Z350-Z350	Adper Easy One	Z350	Z350

Microleakage

Twenty-one caries-free human premolars freshly extracted for orthodontic reasons were randomly divided into seven groups. Standardized Class II cavities (3 mm buccolingually, 4 mm occlusogingivally, and 2 mm deep [width of the gingival floor]) were prepared in each tooth using cylindrical diamond burs (Dia-burs, Mani, Tochigi, Japan) with water spray. The cervical cavity margins of the preparations were all in enamel. The teeth were then fixed in resin blocks (denture base resin type II, Shanhai Medical Instruments, Shanghai, China). A typodont model was established to simulate the interproximal relationship between two adjacent premolars. Sectional matrices (Palodent System, Dentsply, Milford, DE, USA) were placed, secured with wooden wedges (Hawe-Sycamore interdental wedges, Kerr, Bioggio, Switzerland), and checked with a hand instrument (Silver probe, Hu-Friedy, Chicago, IL, USA) to ensure that no detectable space existed between the matrices and cavity margins.

The Class II preparations were restored with different material combinations as shown in Table 2. Approximately 1-mm thick horizontal layers of composites were placed at the cervical margins and light cured for 20 seconds from the occlusal. Two oblique layers were subsequently applied in less than 2-mm increments and cured for 20 seconds each. After removal of the matrices, the restorations were cured for an additional 20 seconds from the buccal and lingual surfaces. The restored teeth were then polished (Super-snap polishing system, Shofu, Kyoto, Japan) and checked under a stereomicroscope (Zoom-630E, Shanghai Changfang Optical Instrument, Shanghai, China) at 40× magnification to prevent any detectable overhangs or microgaps along the margins. The teeth were then stored in distilled water at 25°C for 24 hours prior to microleakage testing.

The restored teeth were coated with two layers of nail varnish 1 mm short of the restoration margins and allowed to dry for 10 minutes. The primed teeth were then immersed in 50% $AgNO_3$ solution ($AgNO_3$, Sinopharm, Beijing, China) for 24 hours, washed under running water, stored in developer (RPX-OMAT, Kodak China, Shanghai, China) for 3 hours, and ultrasonically cleaned for 1 minute to eliminate silver particles from the tooth surfaces. A micro-CT system (Micro-CT, Institute of High Energy Physics, Chinese Academy of Science, Beijing, China) with an X-ray source of 70 kV/90 mA was used to scan the specimens. Each specimen was rotated 360° with a rotation step of 0.4°, and 900 projections were taken for each sample. The projections were reconstructed and converted to 2D bitmap format images. Resolution of the image was 1024*1024, with a pixel size of 11.5 μm. Cervical microleakage was scored according to the depth of dye penetration at the tooth-restoration interfaces using Mimics 10.01 software (Mimics, Materialise, Leuven, Belgium). Microleakage was recorded at 0.1-mm intervals from the buccal to lingual surfaces providing 30 sites per specimen and a total of 90 sites per group. Scoring for microleakage (Figure 1) was as follows: 0 = no dye penetration, 1 = dye penetration 0.0–0.5 mm, 2 = dye penetration 0.5–1.0 mm, 3 = dye penetration 1.0–1.5 mm, and 4 = dye penetration extending beyond 1.5 mm to the cavity wall. Microleakage data for the various material combinations were computed and analyzed using the Kruskal-Wallis test at significance level $p<0.05$. Correlations between microleakge, viscosity, shrinkage, and filler volume were performed with Spearman's rho correlation at significance level $\alpha=0.05$. Statistical analysis was carried out using SPSS version 20.0 (IBM, SPSS, Chicago, IL, USA).

RESULTS

Mean volumetric shrinkage for the flowable and sculptable composites is shown in Table 3. The

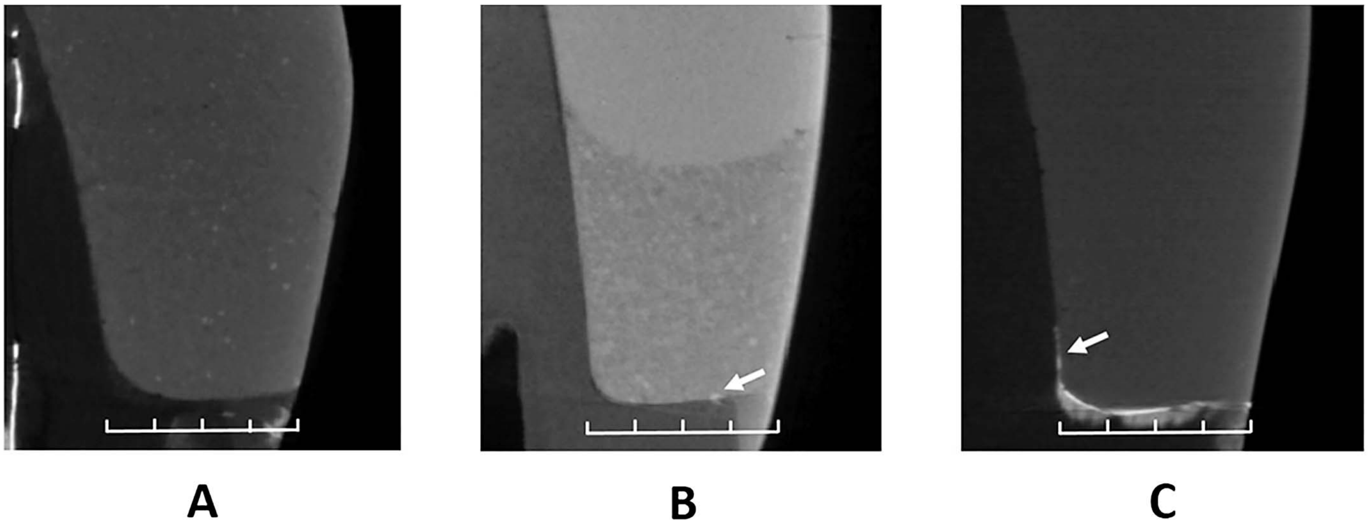

Figure 1. *Representative images depicting cervical microleakage scoring according to depth of dye penetration at the tooth-restoration interfaces using micro-CT. (A-C): Microleakage scores of 0, 2, and 4 according to the depth of silver penetration.*

flowable materials shrank more than their sculptable counterparts BF and Z350. F02 and F10 had significantly higher shrinkage than did F00, F03, or Z350F. Correlation coefficient between filler volume and volumetric shrinkage was −0.86 for all composites and −0.98 for the giomer materials. Complex viscosity is an objective rheologic index of flowability of materials. A lower complex viscosity stipulates easier flow. The flowability of all composites improved with increasing frequency (Figure 2). Table 4 indicates the complex viscosities, after natural logarithmic transformation, at frequencies $\omega = 100$ rad/s and 10 rad/s that represent the viscosities of the composites during and after extrusion through the syringe needle. At both 10 rad/s and 100 rad/s, the sculptable materials BF and Z350 had the highest complex viscosities. For the flowable composites, three-clustering of complex viscosities was observed as follows: Z350F and F02 > F00 > F03 and F10.

Distribution and mean rank of cervical microleakage scores are reflected in Table 5. The use of flowable and sculptable giomer materials resulted in less microleakage than that of their nano-filled counterparts. No microleakage was observed for restorations restored with F02 or F10 flowable giomer liners. For both composite types, the use of flowable liners resulted in significantly less microleakage. Table 6 displays the correlations between viscosity and microleakage as well as shrinkage and microleakage. Significant correlations were observed between shrinkage and microleakage (r=−0.78), also between filler volume and microleakage (r=0.88). Significant correlations were also noted between viscosity and microleakage at frequencies of 10 rad/s (r=0.81) and 100 rad/s (r=0.76). Although viscosity was negatively related to shrinkage (r=−0.61 and −0.54), the correlations were not significant.

DISCUSSION

The effect of flowable composite liner shrinkage and viscosity on the cervical microleakage of Class II restorations was evaluated using micro-CT. The null hypotheses were rejected as microleakage was significantly influenced by and correlated with polymerization shrinkage and viscosity of the flowable composite liners. Complex viscosities of the flowable composites were significantly lower than were their sculptable counterparts BF and Z350. The flowable materials evaluated were non-Newtonian in nature, exhibiting decreased viscosity with increased shear (frequency) rate. This "shear-thinning" behavior allows the flowable composites to be injected easily through fine gauge needles.[18] Complex viscosities of the flowable materials, which were shear-rate

Table 3: *Mean Volumetric Shrinkage of the Various Materials*

	F00	F02	F03	F10	BF	Z350F	Z350
Shrinkage (%)	4.63 (0.23)^a	5.33 (0.17)^b	4.72 (0.24)^a	5.20 (0.21)^b	2.55 (0.09)^c	4.81 (0.04)^a	2.35 (0.02)^c

$^{a-c}$ Letters indicate statistically significant differences. Results of one-way ANOVA and LSD post hoc test (p<0.05).

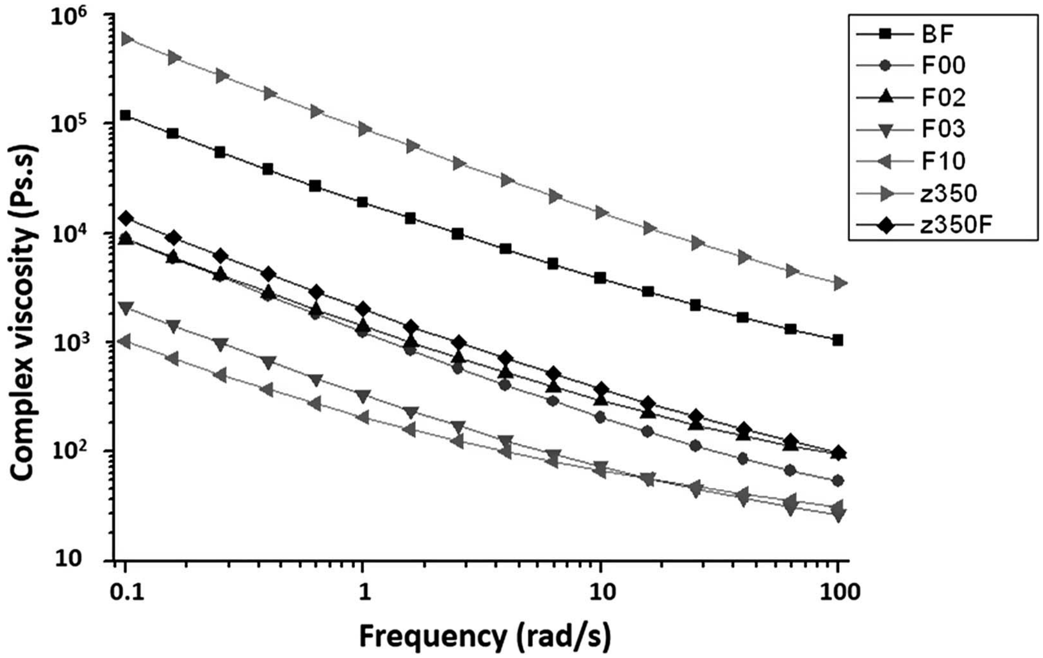

Figure 2. *Complex viscosities of the materials at different frequencies.*

dependent, varied markedly as with other commercially available flowable composites.[19] Dental composites consist primarily of an organic resin matrix and inorganic glass fillers. When shear stresses are applied, the arrangement of the resin monomer molecules and glass fillers are altered. Moreover, interactions between the resin matrix and fillers are also weakened, leading to decreased viscosity with increasing shear frequency (Figure 1). The variance in complex viscosities between flowable materials is an intricate phenomenon that can be attributed to disparities in resin composition as well as filler size, content, surface morphology, and treatment method.[20]

Although volumetric polymerization shrinkage can be assessed by a variety of methods, the optical video imaging technique was selected because of its relative simplicity and comparable dilatometry results.[21] Polymerization shrinkage is affected by the resin matrix composition, filler type, and content.[22] The PRG fillers in the giomer composites can release and recharge fluoride, depending on environmental fluoride concentrations, without compromising strength and stability.[23] The giomer materials evaluated contained the same resins and fillers but in varying quantities (Table 1). When filler content decreased from 68.6% to 33.3%, volumetric shrinkage increased from 2.55% to 5.20% accordingly. The association between filler volume and shrinkage for the giomer composites was significant and very strong ($r=-0.98$). When data for all composites were considered, filler volume was found to be the dominant factor affecting volumetric shrinkage. Our data corroborated those of other authors evaluating the polymerization shrinkage of commercial and experimental composites based on zirconia, silica, and other types of fillers.[24,25]

In addition to filler content, the relative proportion of Bis-GMA, TEGDMA, and other monomers were reported to affect volumetric shrinkage.[26] The resin matrix of most dental composites is a blend of BisGMA and TEGDMA. TEGDMA, which has lower molecular weight and viscosity than Bis-GMA, serves as a diluent and facilitates the incorporation of inorganic fillers. TEGDMA's low molecular weight, however, offers a large number of double bonds and provides a high degree of cross-linking leading to greater shrinkage.[18] The aforementioned

Table 4: *Complex Viscosity of the Materials at Frequencies of 10 rad/s and 100 rad/s*

	F00	F02	F03	F10	BF	Z350F	Z350
Complex viscosity frequency(10 rad/s)	5.33	5.68	4.29	4.20	8.88	5.93	9.65
Complex viscosity frequency(100 rad/s)	3.98	4.56	3.28	3.44	7.40	4.58	8.16

Table 5: *Distribution and Mean Ranks of the Cervical Microleakage Scores for the Various Materials*

	Score					n	Mean Ranks
	0 0	0<a≤0.5 1	0.5<a≤1 2	1<a≤1.5 3	1.5<a≤2 4		
F00	73	17	–	–	–	90	265.8^{a,b}
F02	90	–	–	–	–	90	218.5^{a}
F03	76	14	–	–	–	90	257.5^{a,b}
F10	90	–	–	–	–	90	218.5^{a}
BF	65	15	2	5	3	90	298.5^{b}
Z350F	41	12	17	4	16	90	391.7^{c}
Z350	1	7	5	50	27	90	558.0^{d}

$^{a\text{-}c}$ Letters indicate statistically significant differences. Results of Kruskal-Wallis test ($p<0.05$).

may explain in part the slightly higher shrinkage of BF (2.55%) compared with that of Z350 (2.35%) despite BF's higher filler content (Table 1). The flowable "Plus" giomers (ie, F00 and F03) also contain more TEGDMA than do their predecessors F02 and F10,[27] which may have negated some of the positive effects on polymerization shrinkage offered by the higher filler content of F00 and F03.

Micro-CT has been used to study dental composite shrinkage, gap formation, and microleakge.[28] This nondestructive technique requires a radiopaque dye such as silver nitrate for studying the interfacial integrity of composite restorations. Cross-sectional analysis was performed as 3D reconstruction software was not available for the micro-CT used. A 1-mm layer of flowable composite liner was used before placing the sculptable materials, as this has been shown to result in the least microleakage.[29] No microleakage was observed when F02 and F10 were used as flowable liners. Both these materials had the lowest filler volume and highest resin content.

Although higher resin content is associated with greater shrinkage stress,[22] a strong and negative association was observed between shrinkage and microleakage (r=–0.78). The greater shrinkage stress accompanying the higher resin content of flowable liners might be mitigated by their lower elastic modulus and viscosity.[30,31] Shrinkage stress of the sculptable composites is absorbed by the relatively elastic initial layer of liner, thereby preserving the cervical interfacial integrity of the Class II restorations.[31,32] Microleakage was significantly and strongly correlated with viscosity due to poorer cavity adaptations. Findings corroborated the results of previous studies on nongiomer materials.[6-8] The giomer restorations had significantly less microleakage than those restored with nano-filled composites. This, along with their fluoride release/recharge and antibacterial properties, accounts partly for their long-term, favorable performance.[33]

The current study had several limitations. First, the cervical cavity margins of the Class II preparations were located in enamel and not dentin. Bonding to enamel results in less microleakage and merely embodies a best-case scenario.[34] The ability of flowable composite liners to reduce microleakage at dentin margins is still contentious[35,36] and warrants additional investigation. Moreover, the aging and thermal cycling of restorations is known to adversely affect enamel microleakage.[34,37] These events are beyond the scope of the current study and should be considered for future work. Adhesive strategies and systems may possibly influence microleakage and bond strengths.[37-39] The aforementioned, together with clinical data from longevity studies, are merited to substantiate the clinical utility of flowable composite liners in Class II restorations.

CONCLUSIONS

The influence of polymerization shrinkage and viscosity of flowable composite liners on the cervical

Table 6: *Correlations Between Shrinkage, Microleakage, Resin Content, and Viscosity*

	Viscosity				Microleakage	
	Shrinkage		Microleakage		Filler Volume	Shrinkage
	10 rad/s	100 rad/s	10 rad/s	100 rad/s		
r	−0.61	−0.54	0.81*	0.76*	0.88*	−0.78*

* indicates significant correlation. Results of Spearman's rho correlation ($p<0.05$, 2-tailed).

microleakage of Class II restorations was evaluated using micro-CT. Within the limitations of this in-vitro study, the following conclusions can be made:

1. Flowable composite liners should be used to reduce cervical microleakge of Class II restorations.
2. Cervical microleakage is associated with the polymerization shrinkage and viscosity of the flowable composite liners employed.
3. When selecting composite liners, those with lower filler volume are encouraged despite their higher shrinkage.
4. Giomer restorations had significantly less cervical microleakage than those restored with nano-filled composites.

Regulatory Statement

This in vitro study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Department of Cariology and Endodontology at Peking University School and Hospital of Stomatology.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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