

Microtensile Bond Strength and Failure Modes of Flowable Composites on Primary Dentin with Application of Different Adhesive Strategies

Abstract

Background: Resin composite is an option for the restoration of primary teeth, and new materials with simplified procedures are increasingly being suggested. **Aims:** This study aims to evaluate the microtensile bond strengths and fracture modes of flowable composites on primary dentin with application of different adhesive strategies. **Materials and Methods:** Sixty extracted noncarious primary molars were abraded from buccal surfaces to expose dentin surface. The teeth were randomly divided into three groups as follows: Group 1, Vertise™ Flow (Kerr) (self-adhering flowable composite); Group 2, G-aenial Universal Flo® (GC Europe) (used with one-step self-etch system); Group 3, Tetric® N-Flow (Ivoclar/Vivadent) (used with two-step total etch system). Then, the flowable composites were applied to buccal dentin surfaces with the help of guide mold. Samples were embedded in acrylic blocks and sectioned to form dentin-composite sticks with a surface area of approximately 1 mm². Finally, a total of 180 sticks were obtained to give each group of 60 sticks. Microtensile bond strengths were measured using a universal testing machine (1 mm/min). Fracture modes were evaluated with scanning electron microscopy. **Statistical Analysis:** Microtensile bond strengths data were analyzed by Kruskal–Wallis nonparametric test. **Results:** The microtensile bond strengths of G-aenial (15.5 megapascals [Mpa]) and Tetric (13.0 MPa) were statistically significant higher than Vertise (2.3 MPa). It was recorded that most of fractures in G-aenial was 40% cohesive, Tetric was 53.3% mixed, and Vertise was 83.3% adhesive. **Conclusions:** The self-adhering flowable composite Vertise™ Flow had the lowest and G-aenial Universal Flo® had the highest microtensile bond values.

Keywords: Bond strength, flowable composite, primary dentin, self-adhering

Introduction

Flowable composites are created by reducing the filler content and increasing the diluent monomers in the formulation to reduce the viscosity of the material.^[1] The handling properties and injectable delivery system of flowable composites have improved the material placement in preparations and have increased the range of clinical applications.^[2,3] Flowable composites have a good wetting ability; therefore, they are expected to be adapted to the internal cavity wall better than the conventional hybrid composites which are more viscous.^[4]

However, due to their reduced filler content, especially first-generation flowable composites, they show increased polymerization shrinkage and lower mechanical properties when compared with conventional hybrid composites.^[1,4,5] On account of this, they have been suggested

to be used in areas of low occlusal loading and requiring good penetration such as restorative dental materials margin repairs, pit and fissure sealing, cavity lining, enamel defects, for small Class III and Class V restorations.^[3,6,7] The latest generations of flowable composites have higher filler content. Hence, according to the manufacturers, physical and mechanical properties of the flowable composites have increased and they are comparable to the conventional hybrid composites also with the same flow behavior. Therefore, they are now also recommended for larger or deeper posterior cavities and in higher thicknesses, similar to the conventional hybrid composites.^[2-4]

On the other hand, researchers have worked on reducing the sensitivity of the adhesion technique in the last decade. “Self-adhering composite resin” is a remarkable progress in adhesive dentistry, which combines an all-in-one bonding system and a flowable

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composite. Eliminating the need for a separate adhesive application provides advantages to saving chair time and minimizing handling errors. These self-adhering flowable composites are even more useful when a patient may be uncooperative during treatment, especially in pediatric dentistry. However, in previous studies, bonding effectiveness data are still limited on permanent and primary teeth.^[8-14]

Resin composite is an option for the restoration of primary teeth, and new materials with simplified procedures are increasingly being suggested. However, chemical, physiological, and micromorphological differences between primary and permanent teeth such as small size and lower concentration of dentinal tubules, more reactivity to acidic conditioner, decreased mineralization, and permeability are thought to be responsible for lower bond strength and sealing ability in primary dentin.^[15,16] Especially due to the accumulation of plaque in children with poor oral hygiene, cervical caries are common, and Class V restorations are more affected from microleakage than other types of restorations.^[7] Hence, a low-viscosity composite is recommended for cervical restorations to reduce polymerization shrinkage and microleakage.^[17]

The aim of this *in vitro* study was to evaluate the microtensile bond strengths and failure modes of a self-adhering flowable composite (step-less), a high filler content universal flowable composite (one-step self-etch system), and a conventional flowable composite (two-step total-etch system) for the control group on primary dentin. The null hypothesis of this study was that there are no differences between the experimental and control groups.

Materials and Methods

The 60 extracted noncarious human primary molars, collected with patients' informed consent under a protocol reviewed and approved by the institutional review board of Ankara University, Faculty of Dentistry, were used in the present study. The teeth were cleaned of tissue remnants and stored in distilled water at room temperature. Each

of the buccal cervical enamel surfaces was ground with a series of SiC papers (numbers 600, 800, 1000, 1200) under water cooling until the flat and homogeneous dentin surfaces were exposed. The teeth were randomly divided into three groups, resulting in 20 teeth per group, and the materials that have been used are listed in Table 1. All materials were used according to manufacturer's instructions:

- Group 1: Dentin surfaces were rinsed with water and gently air-dried for 5 s. A self-adhering flowable composite, VF (Vertise Flow Dental Restorative Materials, Kerr Corporation, Orange, CA, USA), was applied to the whole dentin surfaces with a dispensing tip and was brushed a thin layer (<0.5 mm) with moderate pressure for 15–20 s, then was light cured for 20 s with a visible light curing unit (Hilux, Ledmax-550, Benlioglu, Turkey). Finally, VF was incrementally placed to remaining buccal surfaces with using a silicon mold, and each 1.5 mm increment was light cured for 20 s.
- Group 2: Dentin surfaces were rinsed with water and gently air-dried for 5 s. A one-step self-etch adhesive, G-aenial Bond® (GC Corporation, Tokyo, Japan), was applied to the whole dentin surfaces and was light cured for 20 s with the same visible light curing unit. Finally, a universal and high filler content flowable composite, GUF (G-aenial Universal Flo®, GC Corporation, Tokyo, Japan), was incrementally placed to remaining buccal surfaces with using a silicon mold and each 1.5 mm increment was light cured for 20 s.
- Group 3: Dentin surfaces were rinsed with water and gently air-dried for 5 s. The dentin was etched with a 37% phosphoric acid (Total Etch®, Ivoclar-Vivadent, Schaan, Liechtenstein) for 15 s, rinsed for 15 s, and gently air-dried for 10 s. After that, the bonding agent Tetric® N-Bond (Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied to the whole dentin surfaces, gently air-dried for 10 s and was light cured for 10 s with the same visible light curing unit. Finally, a conventional flowable composite, TNF (Tetric® N-Flow,

Table 1: Manufacturer and content of the test materials

Materials/manufacturers	Type	General composition
Vertise™ flow (Kerr Corporation, Orange, CA, USA)	Self-adhering flowable composite	GPDM, methacrylate comonomers, prepolymerized filler, barium glass filler, nano-sized colloidal silica, nano-sized ytterbium fluoride
G-aenial Universal Flo® (GC Corporation, Tokyo, JAPAN)	High filler content universal flowable composite	Urethane dimethacrylate, BIS-MEPP, TEGDMA, strontium glass, SiO ₂ , photoinitiator, pigment
Tetric® N-flow (Ivoclar Vivadent, Schaan, Liechtenstein)	Traditional flowable composite	Paste of dimethacrylates, inorganic fillers, ytterbium trifluoride, initiators, stabilizers, and pigments
G-aenial Bond® (GC Corporation, Tokyo, JAPAN)	One-step self-etch adhesive	Dimethacrylate, 4-META, phosphoric acid ester monomers, distilled water, acetone, SiO ₂ , photoinitiator
Tetric® N-Bond (Ivoclar Vivadent, Schaan, Liechtenstein)	Two-step total-etch adhesive	Mixture of dimethacrylates, alcohol, phosphoric acid acrylate, HEMA, SiO ₂ , initiators, and stabilizers

TEGDM: Triethylene glycol dimethacrylate; GPDM: Glycerol phosphate dimethacrylate; 4-META: 4-Methacryloxyethyltrimellitic acid; HEMA: Hydroxyethyl methacrylate; SiO₂: Silicon dioxide; BIS-MEPP: 2,2-bis(4-methacryloxyethoxyphenyl) propane

Ivoclar Vivadent AG, Schaan, Liechtenstein), was incrementally placed to remaining buccal surfaces with using a silicon mold and each 1.5 mm increment was light cured for 20 s.

After all composite blocks in 3 mm heights were obtained on the buccal surfaces of the teeth and stored in distilled water at 37°C for 24 h, the specimens were then embedded in acrylic blocks, horizontally to the long axis of the tooth, leaving the buccal surfaces facing up and sectioned to form dentin-composite sticks with a surface area of approximately 1 mm². Finally 60 dentin-composite sticks were obtained for the each group. The dentin-composite sticks were then fixed with cyanoacrylate glue onto a testing apparatus, and a tensile load was applied with a microtensile tester (Micro Tensile Tester T-61010K Bisco, US) at a crosshead of 1.0 mm/min until fracture occurred. Fracture surfaces were observed using a stereomicroscope (Leica CLS100 Stereomicroscope, Leica Microsystems, Wetzlar, Germany) at a magnification of ×35 to determine the failure modes, which were classified as adhesive (between composite resin and dentin), cohesive within the substrate (dentin or composite resin), or mixed (if adhesive and cohesive fractures occurred simultaneously). Three different failure mode samples were selected from the each group, and the sections were then coated with gold for scanning electron microscopy (SEM) examinations. The observations were carried out by a single operator.

All specimens were maintained moist throughout the whole preparation and test procedure. Results were expressed in megapascals (MPa), and the microtensile bond strength (μ TBS) data were analyzed by Kruskal–Wallis nonparametric test. Pretesting failures were recorded as 0 MPa values and were included in the statistical analyses. For all of the analyses, the level of significance was set at $P < 0.05$. The Bonferroni test was applied for multiple comparisons.

Results

For each material, 60 dentin-composite sticks were used to calculate the mean and the standard deviation of the μ TBS to dentin. The median values of μ TBS recorded for each of the 3 groups are reported in Table 2. The results are presented in Figure 1 in order from highest to lowest bond strength. The μ TBS of GUF (15.5 MPa) and TNF (13.0 MPa) was statistically significant higher than VF (2.3 MPa) ($P < 0.001$). The μ TBS of GUF was greater than TNF although the results were not statistically significant ($P < 0.001$).

The distribution of failure modes is presented in Table 3 and Figure 2. It was recorded that most of failure modes in GUF was 40% cohesive, TNF was 53.3% mixed, and VF was 83.3% adhesive. All differences between the groups were found statistically significant ($P < 0.05$). Surface analysis revealed that pretesting failures occurred predominantly adhesive. All of the cohesive failures in the

study were found to occur in restorative materials. The SEM photomicrographs of μ TBS-failed surfaces are presented in Figure 3.

In Figure 3a and b, there were any composite material on the dentin surface in accordance with adhesive failure of VF samples. Fewer open dentin tubules and clogged with smear layer of dentinal tubules were seen in Figure 3c. The observations supported the lower bond strength of VF with adhesive failure mode.

In Figure 3d and e, the dentin surface was seen completely closed with composite material in accordance with cohesive failure of GUF samples. There were any open dentin tubules in Figure 3f and this supported the highest bond strength of GUF.

In Figure 3g and h, the dentin surface was seen partially closed with composite material in accordance with mix failure of TNF samples. Fewer open dentin tubules and composite material remaining on the dentin surface were seen in Figure 3i. The observations supported the acceptable bond strength of TNF with mix failure mode.

Discussion

Adhesion permanence is a very important factor for the success of a composite restoration. The bond strength of resin materials to the tooth structure is affected by several determinants such as the type of tooth, adhesive system, and restorative materials.^[18] Only a few data are available about the adhesive properties of this simplified, self-adhering flowable composite on primary teeth.^[11-13] Otherwise, there is only one study in literature regarding the bond strength of recently developed high filler content universal flowable composite on primary teeth.^[19] With this study, we have tested the bonding effectiveness of the new flowable composites to primary dentin.

Hypothesis that there are no differences between the experimental step-less self-adhering flowable composite (VF), one-step self-etch system with high filler content universal flowable composite (GUF), and the control group of the two-step total-etch system with a conventional flowable composite (TNF) should be partially rejected. One could accept that the very simple to use VF applied without a separate adhesive would bond less effectively to primary dentin than both of GUF and TNF, which have similar bond strength. In previous studies, VF showed significantly lower bond strength to primary and permanent enamel and dentin when compared with other flowable composites.^[8,10,18,20-23] Vichi *et al.*^[10] reported that VF showed the lowest bond strength values on permanent dentin and enamel and they associated the material's inadequate wettability. Bektas *et al.*^[14] reported that VF demonstrated the lowest bond strength to permanent dentin because of the addition fillers which might decrease the wettability by increasing the viscosity. Eliades *et al.*^[24] reported that VF has 70 wt% inorganic filler content and

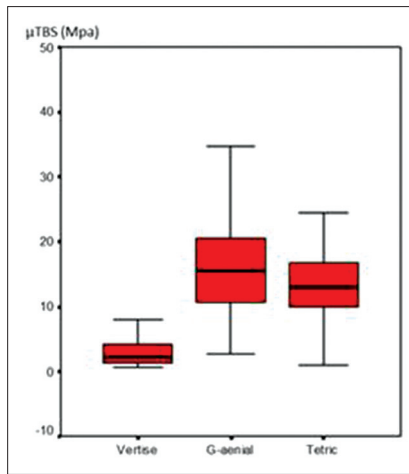


Figure 1: The results of microtensile bond strength

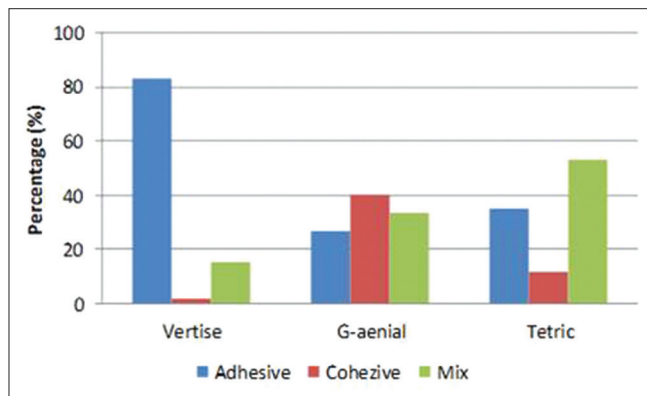


Figure 2: The distribution of failure modes

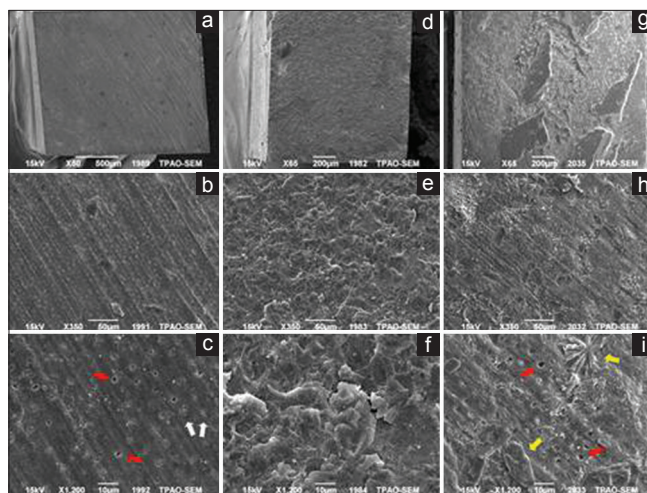


Figure 3: As failure modes and typical empty dentin tubules showed in scanning electron microscopy images. (a) Vertise Flow-adhesive failure scanning electron microscopy, $\times 50$. (b) Vertise Flow scanning electron microscopy, $\times 350$. (c) Vertise Flow scanning electron microscopy, $\times 1200$. (d) G-aenial Universal Flo[®]-cohesive failure scanning electron microscopy, $\times 65$. (e) G-aenial Universal Flo[®] scanning electron microscopy, $\times 350$. (f) G-aenial Universal Flo[®] scanning electron microscopy, $\times 1200$. (g) Tetric[®] N-Flow-mix failure scanning electron microscopy, $\times 65$. (h) Tetric[®] N-Flow scanning electron microscopy, $\times 350$. (i) Tetric[®] N-Flow scanning electron microscopy, $\times 1200$. Red arrow: Open dentin tubules. White arrow: Clogged with smear layer of dentinal tubules. Yellow arrow: Material remaining on dentin surface

Table 2: The median values of microtensile bond strengths recorded for each of the 3 Groups

Material	μ TBS (Mpa)
Vertise [™] Flow	2.3 (2.93) ^{a,b}
G-aenial Universal Flo [®]	15.5 (10.06) ^a
Tetric [®] N-Flow	13.0 (6.99) ^b
P	<0.001

^aStatistically significant differences in μ TBS median values among vertise and G-aenial ($P < 0.001$); ^bStatistically significant differences in μ TBS median values among vertise and Tetric ($P < 0.001$). Mpa: Megapascal; μ TBS: Microtensile bond strengths

Table 3: The distribution of failure modes

Failure mode	Vertise [™] flow (%)	G-aenial Universal Flo [®] (%)	Tetric [®] N-flow (%)
Adhesive	50 (83.3) ^{a,b}	16 (26.7) ^a	21 (35.0) ^b
Cohesive	1 (1.7) ^a	24 (40.0) ^{a,c}	7 (11.7) ^c
Mixed	9 (15.0) ^{a,b}	20 (33.3) ^{a,c}	32 (53.3) ^{b,c}

^aStatistically significant differences in failure mode among vertise and G-aenial ($P < 0.05$); ^bStatistically significant differences in failure mode among vertise and Tetric ($P < 0.001$); ^cStatistically significant differences in failure mode among G-aenial and Tetric ($P < 0.05$)

high viscosity with poor flow characteristics. The manufacturer recommends brushing the first layer of VF onto the entire cavity surface for 20 s to increase the effect of the acidic composite matrix with active application as suggested by Vichi *et al.*^[10] On this study, although performing active application, the bond strength of VF was found insufficient.

The glycerol phosphate dimethacrylate (GPDM) is the functional monomer of VF. This monomer is also used in Optibond FL (Kerr, Orange, CA, USA), which has been proved in both *in vitro* and *in vivo* studies to be a high-performing adhesive among the currently using adhesive systems.^[8,25] According to Poitevin *et al.*,^[8] GPDM “etches” rather than “bonds to” hydroxyapatite (HAp), because of unpublished observations of two-step adhesive that also contains GPDM, nevertheless revealed a 2 μ m deep hybrid layer free of HAp. Specifically, the phosphate group of GPDM is responsible for acid etching.^[10] Poitevin *et al.*^[8] believed that a flowable composite should contain a functional monomer that rather has an effective chemical bonding potential, as it cannot penetrate deeply to achieve self-adhesiveness. Further chemical interfacial analysis is definitely needed to clear up the bonding mechanisms of the self-adhering flowable composites.

The self-adhering flowable composite eliminates the main steps of etching, rinsing, priming, and bonding. Fu *et al.*^[26] have reported that vacant dentin tubules and dentin-resin gaps were observed in both the TEM and SEM examinations, which were associated with the poor adhesion of VF. Moreover, the nonrinse VF appeared to open the dentin tubules and exposed a microporous collagen fibrillar network, which is similar to the effect

of an etch-and-rinse approach using phosphoric acid. However, as known, in etch-and-rinse (three steps) systems, the rinsing process allows the calcium phosphates away, but in the case of nonrinse VF, these calcium phosphates are embedded. Therefore, it is thought to be poor adhesion and weakening the interfacial integrity.^[5]

Only a few data are available about the adhesive properties of self-adhering flowable composite on primary teeth, and they support the results of permanent teeth studies. Scaminaci *et al.*^[11] examined μ SBS of a self-adhering light-curing resin composite, a self-etch adhesive, and a glass-ionomer cement to primary dentin. They reported that the bond strength measured in self-adhering resin composite VF group was significantly lower than that one recorded in self-etch adhesive group. Tuloğlu *et al.*^[12] reported that SBS values of VF groups were lower than conventional flowable resin composite groups for primary and permanent teeth. Pacifici *et al.*^[13] evaluated the shear bond strength of VF to dentin of primary molars. The authors concluded that VF established on primary dentin bond strengths values similar to glass ionomer cements, not to composite resins. These findings are in line with the results of our investigation on primary dentin.

On the other hand, for comparison of the other testing materials in our study, we combined the adhesives with the flowable composite resin produced by the same manufacturer. According to De Munck *et al.*,^[25] the kind of adhesive and composite (and the actual brand) significantly influence the μ TBS. That's why we used GUF with G-aenial Bond® (GC Corporation) and TNF with Tetric® N-Bond (Ivoclar Vivadent).

The composition of universal flowable composite matrix and the pretreatment of the filler surface are modified to provide optimal viscosity and handling properties for restorations.^[27] GUF contains nanoparticles in its filler content. Beun *et al.*^[28] showed that it is possible to produce flowable materials with similar properties to conventional microhybrid composites by adding nanoparticles. Lazaridou *et al.*^[29] showed that new flowable materials with increased filler volume have better wear resistance than some conventional composites, showing best results for GUF, and they suggested that new flowable composite materials could be used now in a wider range of clinical application.

Poggio *et al.*^[30] reported that the carboxylic group of 4-META renders G-aenial Bond® more hydrophilic and more suitable for dentinal surfaces, which are rich in water. Juloski *et al.*^[31] evaluated that μ TBS to permanent molar dentin and they showed that μ TBS of GUF was relatively higher than critical bond strength. In previous studies reported that when permanent dentin is used as a bonding substrate to evaluate adhesive systems reaching critical bond strengths over 15 MPa.^[32,33] In general, acceptable minimum value of bond strength to primary dentin reported by Jumlongras and White^[34] is 17.6 MPa. In these values,

the VF (2.3 MPa) give unsatisfactory results; we see that the GUF (15.5 MPa) and TNF (13 MPa) produce more acceptable levels of results. The present results give an indication that universal flowable composite material could be used on primary dentin. The literature has only one study about the bond strength of recently developed high filler content universal flowable composite on primary teeth. The results of the study conducted by Sachdeva *et al.*^[19] demonstrated that μ TBS of GUF was significantly greater than self-adhering flowable composites. These results showed compliance with our study.

The μ TBS test has been considered as a very sensitive technique and one of the most reliable methods to evaluate the bonding performance of the materials *in vitro*.^[18,35] When materials or substrates with relatively low bond strength values are tested, specimens tend to fail prematurely during preparation.^[10,36] In this study, a relatively high proportion of the VF beam specimens failed before testing when bonded to dentin. Similarly, Poitevin *et al.*^[8] pointed out that pretesting failure score was high in VF group like that 16 failure in 24 specimens. If there is a pretesting failure, the bond strength value can be recorded 0 MPa for the specimen. This actually penalized the material seriously because there was always a certain bond strength above 0 MPa. However, if the specimens that failed before testing would have been excluded from the μ TBS calculation, distinctly a higher data would have been noted.^[37] Eventually, a high occurrence of pretesting failure in one experimental group generally goes together with comparatively low μ TBS values for the remaining specimens that did not fail before testing.^[8] In this study, the specimens that failed before testing were included in the calculation of the mean μ TBS.

The SEM observations in the present study support the findings of the microtensile bond strength values. Fracture analysis showed that higher bond strengths were often associated with “mixed” or “cohesive” failures. In groups where GUF was used, failures appeared completely “cohesive within the composite.” According to Van Ende *et al.*,^[4] this may sign that the exhibited stress had exceeded the tensile strength of the composite before the real bond strength was appeared. Hence, these failures were mostly associated with higher MPa values. On the contrary, pretesting failures always occurred “adhesively at the interface.” According to Oyama *et al.*,^[38] this may sign that debonding due to shrinkage may have occurred within the soft and sticky oxygen inhibition layer that was detached from the cured part of the adhesive layer when the composite retracted from the cavity bottom during polymerization.

Conclusion

Within the limitations of this study, it was concluded that among the current flowable composites, Vertise™ Flow had the lowest and G-aenial Universal Flo® had the highest microtensile bond values on primary dentin. Although the

newly developed adhesive systems are more user-friendly because of their simplified bonding procedures, the bonding strengths of self-adhering flowable composite Vertise Flow were found to be inadequate. More laboratory and clinical studies are needed on newly developed materials. With further developments in material technology, self-adhering materials could be promising materials, especially for pediatric dentistry.

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Conflicts of interest

There are no conflicts of interest.

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