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PUSH-OUT BOND STRENGTH OF GLASS FIBER POST TO COMPOSITE RESIN

AND TO RESIN CEMENT/INTRARADICULAR DENTIN

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ABSTRACT

This in vitro study aimed at evaluating the effect of glass-fiber post (GFP) surface treatment with a silane-containing universal adhesive and/or a silane coupling agent on push-out bond strength to dual cure resin cement/dentin (Experiment 1) and to resin composite (Experiment 2). Methods: For Experiment 1, sixty human premolar root canals were prepared to receive GFP cementation. Then an adhesive system was applied, according to the manufacturer's instructions. The GFPs (Reforpost no. 2, Angelus) were randomly divided into 2 groups, according to application or nonapplication of a silane coupling agent for 60 seconds (Prosil, FGM). Each group was further divided into 3 groups, according to the adhesive system to be applied to the GFP surface (n=10): 1. SBU - silane-containing universal adhesive (Adper Single Bond Universal Adhesive, 3M ESPE); 2. ASB2 – etch-and-rinse adhesive system not containing silane in its composition (Adper Single Bond 2, 3M ESPE); 3. CG (control group) - no treatment. Cementation of GFPs to intraradicular dentin was performed with dual-cure conventional resin cement (Rely X ARC/ 3M ESPE). After 48 hours, the roots were cross-sectioned at three different depths, resulting in serial slices corresponding to the cervical, middle and apical root thirds. For Experiment 2, surface-treated GFPs (n=10) were centered in a plastic matrix in which the resin composite (Filtek Z250 XT, 3M ESPE) was incrementally inserted and light-cured. Slices were obtained, as in Experiment 1. Push-out bond strength testing was performed on a universal machine (0.5 mm/min crosshead speed). Results: In Experiment 1, three-way ANOVA (α =0.05) indicated that the bond strength of GFP to resin cement/intraradicular dentin was not affected by use or nonuse of a silane coupling agent, by application or non-application of an adhesive layer or by the root canal depth (p>0.05). For Experiment 2, two-way ANOVA and Tukey's test showed that when the GFP received silane pretreatment, bond strength to resin composite was not affected by the application of an adhesive layer to the post. However, when the post was not pretreated with silane, the bond strength was higher for the universal adhesive (p=0.021). The bond strength to post/resin cement/dentin was unaffected by post silanization or by the application of an adhesive system layer on the post. Bond strength of the post to resin composite was unaffected by adhesive system application, if the post was previously treated with a silane agent. In the absent of post silanization, a universal adhesive containing silane achieved higher bond strength of the post to resin composite.

Key Words: Dentine; Composites; Push-Out Bond Strength; Silane; Glass Fiber Posts.

1. INTRODUCTION

Restorative treatment of endodontically treated teeth usually requires the use of an intraradicular post to improve retention of the restoration [1,2]. The choice of post materials has shifted from rigid materials, such as metals, to materials that possess a mechanical behavior similar to that of dentin [1,3,4,5]. This has resulted in a decrease in catastrophic failures in radicular dentin.6 In this context, glass fiber posts (GFPs) have frequently been indicated due to their lower modulus of elasticity than cast posts, their more esthetic property, and their lower risk of staining the dental structure, since this kind of post does not undergo oxidation [4,7,8,9]. In cases of weakened roots, relining the glass fiber post with resin composite is an adequate option to improve post retention [4] and enhance tooth fracture resistance [10].

Despite the advantages of GFPs, post debonding has been frequently reported [11,12]. This has been attributed to many factors, such as the presence of a remaining coronal structure [6,13], method of cement application and post surface treatment [12,14], and problems during bonding procedures [11].

In an attempt to improve the bond strength of GFPs to resin cement or to resin composite, post silanization has been recommended to strengthen the bond between the inorganic fillers in the post and the organic matrix in the resin material [15,16,17,18]. Although the beneficial effect of silane on the bond strength of GFPs has been demonstrated [19], this effect seems to be valid only when associated with adequate post cleaning [14]. Investigations by other authors have reported no positive effect of silane on bond strength [20]. In addition, the application of an adhesive layer after silanization may enhance bond strength in middle and apical root thirds [21].

Recently, the use of universal adhesives has been expanded in clinical applications [22], including the possibility of treating GFP or ceramic surfaces, because the silane molecule is often incorporated in universal adhesives. However, the literature is scant in revealing whether the application of silane, or a layer of silane-containing adhesive, or the combination of both materials, could enhance the bond strength of a GFP to resin cement/dentin or to resin composite, in cases of post relining.

The aim of the present study was to evaluate the effect of a silane agent and a silane-containing universal adhesive on the push-out bond strength of GFPs to resin cement/intraradicular dentin or to resin composite. The null hypothesis to be tested is that the application of a silane coupling agent, or a universal adhesive containing silane, or the association of both, does not affect the push-out bond strength to resin cement/dentin or to resin composite.

2. MATERIALS AND METHODS

2.1 Experiment 1 – Push-out bond strength of a glass-fiber post to resin cement/dentin

The present study was approved by the Research Ethics Committee (CAAE #54910516.9.0000.5374).

2.1.1 Root canals: endodontic obturation and preparation for post cementation

Sixty premolars were selected and kept in an aqueous 0.1% thymol solution by dissolving thymol in distilled and deionized water. The teeth were cleaned with periodontal curettes and their crowns were sectioned horizontally at the cervical level, near the cementoenamel junction, using a double-sided diamond disc (Microdont

Micro Usinagem de Precisão Ltda., São Paulo, SP, Brazil) coupled to a metallographic saw, to standardize the roots at 13 mm in length. The roots that did not conform to this criterion of length were discarded.

The root canals were debrided conventionally with #2, #3, #4 Gates Glidden drills, and irrigated with 1% sodium hypochlorite (Biodinâmica, Londrina, PR, Brazil). After instrumentation, the root canals were washed with saline solution (NaCl 0.9%). Excess solution was aspirated with a cannula, and the root canals were gently dried with absorbent paper points. Next, they were filled with gutta-percha and a calcium-hydroxide-based and eugenol-free endodontic cement (Sealer 26, Dentsply, Rio de Janeiro, RJ, Brazil), using the lateral condensation technique. The roots were then placed in a 21-mm diameter x 34-mm high acrylic matrix filled with heavy condensation silicone putty.

After 24 hours at relative humidity (37°C), root canal preparation was carried out using rotatory #2 Largo burrs (9 mm long). The root canals of all the groups were prepared and then rinsed using distilled water aspirated with cannulas attached to the suction unit, after which the irrigant residue was removed with absorbent paper cones, keeping the dentin moist.

2.1.2 Post cementation

The universal adhesive system (Single Bond Universal, 3M ESPE, St. Paul, MN, USA) was applied to the root canals, according to the manufacturer's instructions. Then the adhesive system was actively applied to intraradicular dentin for 20 seconds with a disposable brush. A brief air spray was applied for 5 seconds to evaporate the

solvate, excess adhesive was removed with an absorbent paper cone, and the adhesive was light-cured for 10 seconds.

The fiber posts were cleaned with 70% ethyl alcohol for 30 seconds, and gently air-dried for 5 seconds, as recommended by the post manufacturer.

In groups 1, 2 and 3, one layer of silane (Silane, Angelus, Londrina, PR, Brazil) was applied to the GFP surface for 1 minute, and gently air-dried.

In groups 2 and 4, one coat of the universal adhesive system containing silane (Adper Single Bond Universal, 3M ESPE, St. Paul, MN, USA) was applied to the GFP surface, but was not light-cured.

In groups 3 and 5, one coat of the adhesive system not containing silane in its composition (Adper Single Bond 2, 3M ESPE) was applied to the GFP surface, but not light-cured.

Post cementation was performed with the conventional dual-cure resin cement (Rely X Ultimate, 3M ESPE, St. Paul, MN, USA), following the manufacturer's instructions: small, equal amounts of base paste and catalyst paste were dispensed from a self-mixing syringe onto a block. The Endo tip was attached to the wide mixing tip for deepest possible insertion into the root canal, after which the resin cement was applied, beginning apically. The post was placed in the root canal filled with cement, and moderate pressure was applied to hold it in position. The excess was removed with a resin spatula before performing polymerization, and the resin cement was light-cured for 40 seconds. The specimens were kept at relative humidity and 37°C, for 48 hours.

After 48 hours at relative humidity, the specimens were fixed on acrylic plates with sticky wax, so that the root stayed parallel to the long axis of the plate surface.

The plates were then fixed to a precision metallographic saw equipped with a high concentration diamond disc (Extec Corp., Enfield, CT, USA). Parallel cuts were performed to obtain approximately 1.0-mm thick slices of each root third: cervical, middle and apical. Two slices of each root third were obtained for push-out bond strength testing.

The materials and their composition are described in Table 1.

2.1.3 Push-out bond strength testing

The specimens were subjected to the push-out test on a universal testing machine (EZ test) with a 50N load cell at a speed of 0.5 mm/min. The value in kgf was converted to MPa according to the following formula:

$$MPa = (kgf \ 9.8) \ area \tag{1}$$

The area (A) was calculated after measuring each slice individually with a digital caliper. The following formula was used:

$$A = \{ [\rho \quad (R+r)] \quad [h^2 + (R-r)^2]^{0.5}$$
(2)

where: π = 3.1416, R = fiber post radius (including resin cement thickness) measured on the cervical side of the slice, r = fiber post radius (including resin cement thickness) measured on the apical side of the slice, and h = height of the root slice. Since two slices were obtained from each root third, the mean value of these two slices was considered as the value of its corresponding third.

2.1.4 Failure mode analysis

After performing the push-out test, the specimens from each group were assessed under a light microscope at 40x magnification to establish the failure types. The failure modes were classified as: 1) adhesive failure between resin cement and fiber post (ARP), 2) adhesive failure between dentin and resin cement (ADC), 3) resin cement cohesion failure (CRCem); 4) dentin cohesion failure (DC); 5) post cohesion failure (PC); and 6) mixed failure (MF).

2.2 Experiment 2

The GFP received the same experimental treatments, as described in Experiment 1. Each GFP was placed individually in a transparent plastic matrix designed to keep the GFP in a vertical position, as suggested by Silva *et al.*[23] The matrix was then filled incrementally with resin composite (Filtek Z250 XT, 3M ESPE, St. Paul, MN, USA). Each 2-mm increment was light-cured for 20 seconds with a halogen light unit. The specimens were removed from the plastic matrix with a scalpel blade and kept at relative humidity for 48 hours.

The bonding area calculation and push-out bond strength testing were performed as described in Experiment 1. However, the comparison of thirds was not performed in Experiment 2, because the GFP/resin composite block was light-cured outside of the root canal, according to the anatomic post technique simulated in Experiment 2.

The failure modes in Experiment 2 were classified as: ARP - adhesive failure between resin composite and GFP; CRComp – cohesive in resin composite; and MF – mixed failure.

2.3 Statistical Analysis

Both experiments 1 and 2 presented normal distribution of the data. The data of Experiment 1 (push-out bond strength of glass fiber post to resin cement/dentin) was analyzed by applying three-way analysis of variance (ANOVA) for randomized blocks and Tukey's test. The data of Experiment 2 (push-out bond strength of resin composite to glass fiber post) was submitted to two-way ANOVA and Tukey's test. Statistical calculations were performed with SPSS 23 (SPSS Inc., Chicago, IL, USA). The significance level was set at 5%. The failure pattern was described with descriptive statistics (percentage).

3. RESULTS

3.1 Experiment 1 (Table 2, Figure 1) – ANOVA for randomized blocks revealed that the triple interaction of use of silane vs. type of adhesive vs. root third was not significant (p=0.435). Double interactions were not significant (use of silane vs type of adhesive, p=0.109; use of silane vs. root third, p=0.700; and type of adhesive vs. root third, p=0.924). It was also observed that the main factors under study had no significant effect on bond strength to intraradicular dentin (use of silane, p=0.658; type of adhesive, p=0.918; and root third, p=0.322). These results indicate that the bond strength of the GFP to resin cement/intraradicular dentin was not affected by using a silane coupling agent, by applying an adhesive layer (regardless of whether the adhesive contained silane), or by the root canal depth (cervical, middle and apical).

According to the general interpretation of failure modes (Figure 1), it was observed that, with or without applying a silane agent pretreatment or an adhesive layer to the post, the cohesive in dentin (CD) failure type was predominant in the cervical and middle root thirds, whereas the adhesive between resin cement and

dentin (ARD) failure mode was predominant in the apical third. An exception was found for the group that received no silane pretreatment, but did receive a layer of silane-containing adhesive, in which the apical root third presented equal frequency of ARD and CD failure modes. Another exception was found in the cervical and apical thirds of the groups that received no silane pretreatment or adhesive layers, in which there was high CRCem failure frequency.

3.2 Experiment 2 (Table 3, Figure 2) – Two-way analysis of variance demonstrated that there was a significant interaction between two factors: pretreatment of GFP with silane and the type of adhesive system applied to the post (p=0.021). Tukey's test indicated that when the GFP received silane pretreatment, the bond strength to resin composite was not affected by the application of an adhesive layer to the post, even if the adhesive had silane in its composition. However, when the pretreatment of the post with silane was not performed, bond strength was higher if the adhesive system had silane in its composition. Moreover, in the absence of silane pretreatment, the application of a layer of an adhesive system containing silane in its composition promoted higher bond strength than the application of a layer of adhesive without silane. Statistically, lower bond strength was observed in the group that received neither the application of silane pretreatment nor a layer of adhesive system.

Analysis of failure mode indicated that the cohesive in resin failures were predominant (60% to 95%) in most groups. An exception was found in cases in which the post received neither the application of silane pretreatment nor a layer of adhesive system, in which case most failures were observed between the post and the resin composite (ARP). The use of an adhesive containing silane in its composition

promoted no failures between resin composite and GFP, regardless of whether the post was or wasn't silanized with a silane coupling agent. The absence of ARP failures was also verified when silane pretreatment was associated with a layer of adhesive not containing silane in its composition. In the absence of GFP silane pretreatment, the application of a layer of adhesive without silane or no adhesive layer yielded an increase in frequency of mixed failures.

4. DISCUSSION

In theory, GFP pretreatment either with a silane coupling or with post silanization is indicated because the coupling action of the silane leads to the formation of covalent bonds from the reaction of the organofunctional group of the resin matrix and the inorganic glass fiber of the GFP [24].

However, the literature is conflicting in stating whether the use of a silanecoupling agent for GFP pretreatment is a determinant for optimizing adhesion between the post and resin material. This is especially the case when novel materials are considered, such as adhesives that already contain silane in their composition, the so-called universal adhesives. Meanwhile, many clinicians apply a layer of adhesive over the GFP after silanization to improve bond strength [21]. The doubt that still remains is whether post silanization is really necessary to improve the bond strength of GFP to resin cement or to resin composite when a universal adhesive is applied to the post.

The push-out bond strength of the GFP to the dual resin cement/intraradicular dentin, tested in Experiment 1, led to acceptance of the null hypothesis, since it indicated that there were no significant differences among the Experiment 1 groups (p>0.05, Table 2). This result corroborates previous reports [9,15,25]. Metanalysis

recently reported by Skupien et al.[14] indicates that the silanization of posts had no effect on bond strength to root dentin, but increased bond strength when associated to a cleaning procedure. In fact, Faria et al. [26] reported that ethyl alcohol improves the retention of glass fiber posts, removes grease residues that are stuck on the posts, and promotes better contact between the post and the silane agent or between the post and the adhesive material. In the present study, the posts were cleaned with ethyl alcohol, but in the groups submitted to post silanization after cleaning, bond strength values were not higher than those found in the groups without silane application. Perhaps, ethyl alcohol cleaning was sufficient to ensure adequate contact of post to resin cement. It is important to highlight that most failures occurred near the dentin substrate (cohesive in dentin or adhesive between dentin and resin cement), as demonstrated in Figure 1. It is hypothesized that because bond failure first occurred at the dentin-cement interface, it was difficult to observe the effect of experimental post treatments (silane pretreatment or adhesive layer). Braga et al. [27] reported that the cement-dentin interface is the critical issue for post cementation to root canal. Moreover, comparison of root thirds indicated no significant difference in bond strength, as also observed in previous reports [7,28]. It has been speculated that in vitro study conditions called for keeping the light-curing device very close to the cervical region of tooth during polymerization of resin cement, thus promoting high resin monomer conversion, regardless of the root third.

Apart from a situation in which the post is very well adapted to the root canal, rehabilitation of a widened canal space is usually done using the direct anatomical post technique (a resin composite combined with a prefabricated glass

fiber post), associated to crown restoration [29]. This technique may solve some of the problems associated with the cementation of a poorly adapted fiber post in a widened canal space, because it promotes increased bond strength to root dentin. Aiming at reproducing this procedure, Experiment 2 tested the bond strength of the resin composite to the fiber post among similar groups of Experiment 1 (Table 2). Using the methodology applied in Experiment 2 to simulate this technique, only one adhesive interface (between resin composite and post) was formed, and the effects of post treatment were more visible, since there were significant differences among the groups. Hence, the null hypothesis was rejected.

It was demonstrated that when a silane coupling agent was applied to the fiber post, the bond strength to resin composite was similar between the groups in which the universal adhesive (containing silane) and the etch-and-rinse adhesive (not containing silane) were applied, or in cases in which no adhesive was used. The effect of the silane agent on the post may have been sufficient to promote adequate bond strength [19] by enhancing the interaction between the organic (resin cement) and the inorganic (glass from the post) phases [21], especially because the posts were cleaned with ethyl alcohol before silanization [14,26]. The effects of a silane coupling agent were better evidenced when the groups not receiving the adhesive layer were compared: the group that received silane application had higher bond strength than the group that received no treatment. Similar results were reported by Goracci *et al.* [24] and were confirmed by the meta-analysis conducted by *Moraes et al.*[12].

However, the results of Experiment 2 demonstrated that, in the absence of post silanization, an adhesive containing silane in its composition promoted the

highest bond strength values in the Experiment 2 SBU group, statistically different from all other Experiment 2 groups. This result could be attributed to the silane agent contained in its composition. The lowest bond strength was observed in the groups with no post silanization pretreatment or adhesive application, regardless of whether the adhesive contained silane. In fact, Skupien et al.14 demonstrated no significant effect on bond strength of posts that were cleaned but not silanized.

The results of Experiment 2 also demonstrated that higher bond strength values were achieved when post silanization was associated to the application of a conventional etch-and-rinse adhesive than when the same adhesive was applied without post silanization (Table 3), a result corroborated by Machado *et al.* [21] It is believed that unfilled resin adhesive enabled the formation of a more compatible and stronger interaction between the silanized post and the heterogeneous composition of the resinous material, but it is important to highlight that Machado *et al.* [21] evaluated bond strength with resin cement, and not with resin composite, as done in Experiment 2.

Failure mode supports the findings of bond strength testing in Experiment 2 (Figure 2). When the posts receive silanization plus an adhesive layer, there were over 94% of cohesive failures, a result similar to that found by Silva *et al.* [23] Similarly, more than 70% of the failures of the posts that were not silanized but that received the application of adhesive layer were cohesive in resin composite. However, in the cases that no treatment was applied (neither silanization nor adhesive application), there was an increase in mixed failures, especially between resin composite and post. The high percentage of cohesive failures in the groups that received an adhesive layer, especially universal adhesive, can be attributed to the

adhesive application reducing the gaps in the adhesive interface, which is responsible for initiating mechanical failures and increasing the number of stress points inside the fiber post-resin composite sample [23].

The present study indicates that the experimental conditions of the study affected bond strength results and interpretation of data, considering that the results for Experiment 1 (post to resin cement and dentin) and Experiment 2 (post to resin composite) were very different. Experiment 1 (bond strength between post/resin cement and dentin) demonstrated that the weak link lies in the dentin/resin cement interface, and that the effects of post treatment were irrelevant, as confirmed in a recent meta-analysis[12]. In Experiment 2 (bond strength between post/resin composite simulating the anatomical post technique), post silanization can be an indicated procedure, and, in this case, there is no difference in the type of adhesive system applied. In the absence of post silanization, an adhesive system already containing silane in its composition should be preferred. By the results found in this manuscript, especially those in Experiment 2, further studies should be focused on evaluating the influence of silane agent (used as pretreatment agent or by applying an universal adhesive) on long-term bond strength of GFP to resin cement/dentin and to resin composite.

5. CONCLUSIONS

It can be concluded that:

- Bond strength of post/resin cement/dentin was unaffected by post silanization or by the application of an adhesive system layer on the post.

- Bond strength of post to resin composite was unaffected by application of the adhesive system if the post was previously treated with a silane agent. In the absent of post silanization, a universal adhesive containing silane achieved the best bond strength performance.

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Material Trade Name Manufacturer	Composition
Universal Adhesive System Adper Single Bond Universal Adhesive 3M ESPE	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethyl alcohol, water initiators, silane .
Etch-and-rinse Adhesive System Adper Single Bond 2 Adhesive 3M ESPE	Bis-GMA, HEMA, copolymer of acrylic and itaconic acids, water, ethyl alcohol, glycerol 1,3-dimethacrylate, diurethane dimethacrylate, silane treated silica, water.
<i>Resin Cement</i> Rely X Ultimate 3M ESPE	Base paste: Methacrylate monomers, radiopaque silanated fillers, Initiator components, stabilizers and rheological additives. Catalyst paste: Methacrylate monomers, radiopaque alkaline (basic) fillers, initiator components, stabilizers, pigments, rheological additives, fluorescence dye, dark cure activator for Scotchbond Universal adhesive.
Glass Fiber Post Reforpost #2 Angelus	Glass fiber (80%); pigmented resin (19%), stainless steel filament (1%).
Silane Coupling Agent Silano Angelus	Silane and ethyl alcohol

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Bis-GMA, bisphenol-glycidyl methacrylate; HEMA: 2, hydroxy ethyl methacrylate; MDP, methacryloyloxydecyl dihydrogen phosphate.

Table 2 – Mean (standard deviation) of push-out bond strength (MPa) of glass fiber post to resin cement/intraradicular dentin, according to the experimental groups (Experiment 1).

Pretreatment of post with silane agent	Adhesive	Root Third	Push-out bond strength	
	Adper Single Bond Universal Adhesive (containing silane)	Cervical	11.01 (2.92)	
		Middle	10.92 (4.27)	
		Apical	11.14 (3.94)	
Present	Adper Single Bond 2	Cervical	10.98 (2.42)	
(Silane coupling		Middle	10.86 (3.34)	
agent)		Apical	9.66 (4.42)	
	Absent (without adhesive application)	Cervical	10.31 (3.49)	
		Middle	8.91 (3.55)	
		Apical	9.26 (3.52)	
Absent	Adper Single Bond Universal Adhesive (containing silane)	Cervical	11.39 (2.77)	
		Middle	9.89 (4.38)	
		Apical	9.08 (2.99)	
	Adper Single Bond 2 (not containing silane)	Cervical	10.07 (2.26)	
		Middle	10.42 (2.41)	
		Apical	10.37 (4.61)	
	Absent	Cervical	11.13 (3.11)	
	(without adhesive	Middle	12.71 (2.61)	
	application)	Apical	9.75 (3.31)	
RCCC				

Table 3 - Mean (standard deviation) of push-out bond strength (MPa) of glass fiber post to resin composite, according to the experimental groups (Experiment 2).

	Adhesive system			
Pretreatment of post with silane agent	Adper Single Bond	Adper Single Bond 2	Absent	
	Universal Adhesive (containing silane)	(not containing silane)	(without adhesive application)	
Present	13.62 (1.82) Ab	14.17 (2.99) Aa	13.87 (2.08) Aa	
Absent	15.73 (1.28) Aa	13.38 (2.70) Bb	11.38 (3.68) Cb	

, in oil Legend: Means followed by the different letters (uppercase in rows and lowercase in columns) are statistically

Figure 1. Graph representing the frequency (%) of failure modes after push-out bond strength testing in Experiment 1.



Legend: ARD – adhesive between resin cement and dentin; ARP – adhesive between resin cement and glass fiber post; CD – cohesive in dentin; CRCem – cohesive in resin cement; MF – mixed failure.

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Figure 2 - Graph representing the frequency (%) of failure modes after push-out bond strength testing in Experiment 2.



Legend: ARP – adhesive between resin cement and glass fiber post; CRComp – cohesive in resin composite, MF – mixed failure

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