The Effects of Different Desensitizing Agents on the Shear Bond Strength of Adhesive Resin Cement to Dentin

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ABSTRACT

Statement of the Problem: The choice of desensitizing agent can affect the bond strength of adhesive resin cement to dentin.

Purpose: The purpose of this study was to evaluate the effects of different dentin desensitizing agents on the bond strength of adhesive resin cement to dentin.

Materials and Methods: Sixty specimen teeth were randomly divided into six groups ($n = 10$). Five groups of teeth were treated with a desensitizing agent containing sodium and calcium fluoride in cellulose alone, hydroxyethyl methacrylate (HEMA), benzalkonium chloride and sodium fluoride, HEMA and glutaraldehyde, an ormocer-based or a resin-based dentin desensitizer. The remaining 10 specimens served as controls. Adhesive resin cement was applied to the dentin surface. The shear bond strength was measured using a universal testing machine at a 0.5 mm/minute crosshead speed. The data were analyzed statistically with one-way analysis of variance and a Tukey's Honestly Significant Difference (HSD) test ($\alpha = 0.05$). In addition, dentin surfaces were examined by a scanning electron microscope.

Results: The lowest bond strength was in the group treated with desensitizing agent containing sodium and calcium fluoride and the highest bond strength was from the group treated with desensitizing agent containing HEMA and sodium fluoride.

Conclusions: Desensitizing agents containing sodium and calcium fluoride reduced the bond strength of adhesive resin cement.

CLINICAL SIGNIFICANCE

The type of desensitizer used is an important factor regarding the bond strength of adhesive resin cements to dentin.

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INTRODUCTION

Dentinal hypersensitivity occurs after tooth preparation because of the exposure of dentinal tubules.[1,2] Brannström's hydrodynamic theory[3–8] proposes that dentinal hypersensitivity is attributable to chemical, thermal, or osmotic stimuli that cause the fluid within the tubules to flow inward or outward. The movement of the fluid creates a mechanical disturbance, which can excite nerve fibers in the pulp and induce pain.[1,9–11] Microleakage and tooth sensitivity, which occur after cavity or abutment preparation, or during cementation, are common problems. In order to prevent, or at least decrease hypersensitivity, and to

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reduce dentin permeability, desensitizing agents are employed to occlude or seal the dentinal tubules.[1,12,13]

Various dentin desensitizing methods such as varnishes, anti-inflammatory agents, tubular obturating procedures, adhesives, and lasers (neodymium-doped:yttrium aluminium garnet (Nd:YAG) and CO_2) may be applied to the tooth after cavity and crown preparation.[3,13–19] Desensitizing agents which decrease hypersensitivity by occluding the dentinal tubules can significantly reduce fluid infiltration across dentin.[2,20] It has also been reported that dentin permeability is reduced after application of desensitizing agent that precipitate fluoride or calcium oxalate crystals within the dentin.[20]

Current desensitizers include antibacterial components such as fluoride, triclosan, benzalkonium chloride, ethylene diaminetetraacetic acid, and glutaraldehyde.[21] The desensitizing effects of fluoride-containing solutions are related to precipitated fluoride compounds that mechanically block exposed dentinal tubules.[3,9,17] Primers containing glutaraldehyde and hydroxyethyl methacrylate (HEMA) (Gluma Desensitizer, Heraeus Kulzer, Hanau, Germany; Seal & Protect, Dentsply, York, PA, USA; HurriSeal, Beutlich Pharmaceuticals, Waukegan, IL, USA; D/Sense, Centrix Inc., Shelton, CT, USA; SuperSeal, Phoenix Dental, Fenton, MI, USA) can reduce hypersensitivity by sealing or occluding the exposed dentinal tubules by precipitating plasma proteins in the dentinal fluid.[1,3,20]

Durable bonding between dentin and luting material is one of the most important factors in avoiding detachment of restorations, as well as the prevention of microleakage, secondary dental caries and tooth fracture.[15,22,23] Various cements, such as zinc phosphate, zinc polycarboxylate, conventional glass-ionomer cements and resin-based cements, have been used as luting materials.[24–26]

Recently, adhesive resin cements have been widely used for the cementation of many types of fixed partial dentures.[23] The sealing and bonding characteristics of the luting agents may be affected by the use of desensitizers, which have ingredients that induce chemical interaction with dentin's organic matrix.[15,27] There are various views on the loss of retention with the use of desensitizers before cementation of the restoration with conventional cements or resin cement. While some authors have reported the loss of crown retention with the use of desensitizers before cementation of the restoration,[2,14,23,28,29] other investigators have recorded no adverse effects on crown retention.[1,18] It has also been claimed that desensitizing agents containing oxalate reduce the bond strength of resin cement to dentin due to crystal penetration into the dentinal tubules, which creates an acid resistant dentinal surface.[2,30] The reduction in bond strength was attributed to poor resin infiltration due to crystal precipitation on the dentin surface.[23] Huh and colleagues reported that an oxalate-containing desensitizing agent did not affect the bond strength of the adhesive cement when a self-etching adhesive system was used.[23] Another study stated that the application of a desensitizing agent containing glutaraldehyde to a prepared dentin surface did not affect the bond strength of conventional and resin cements to dentin.[1,15,18] Glutaraldehyde reduces hypersensitivity by occluding dentinal tubules, possibly by precipitating plasma proteins in the dentinal fluid.[1,15,18] In addition, Itota and colleagues reported that application of fluoride varnish increased the bond strength of resin cement to demineralized dentin.[31] However, other investigations have reported that agents containing fluoride demonstrated lower bond strength to sound dentin than HEMA-containing desensitizing agents.[15,16,22]

In the context of the preceding uncertainty, this study aimed to evaluate the effects of five different desensitizing agents on the shear bond strength of dual-cure adhesive resin cement to dentin. Furthermore, this study also examined dentin surfaces under a field emission scanning electron microscope (SEM) after the application of desensitizing agents. This research tested the hypothesis that desensitizing agents reduce the bond strength of adhesive resin cement to dentin.

MATERIALS AND METHODS

Thirty, unrestored, caries-free, extracted human maxillary and mandibular third molars were used in the present study. The teeth were cleaned mechanically and stored in 0.5% chloramine at room temperature until used. The root of each tooth was removed from the crown by making a horizontal section at the cementoenamel junction. The crown of the tooth was divided into two equal halves, mesiodistally at the central fossa with a water-cooled diamond blade disc (Komet, Cebr Brasseler GmbH & Co. KG, Lemgo, Germany). The separated halves of each tooth were embedded, with the buccal or lingual surface facing upwards, in the centers of authopolymerizing acrylic resin blocks (Meliodent, Heraeus Kulzer, Armonk, NY, USA). These surfaces were ground flat with a standard-grit diamond rotary cutting instrument (105–125 μm, Diatech, Goltène AG, Altstatten, Switzerland) until the dentin surface was exposed. The surface preparation was finished with a fine-grit diamond rotary cutting instrument (45 μm, Diatech, Goltène AG) to ensure a smooth surface. Subsequently, specimens were divided into 6 groups of 10 specimens for the application of desensitizing agents. The desensitizing agents used in the present study are shown in Table 1. Group 1 served as a control and had no desensitizing agent. The desensitizing agents which contain sodium and calcium fluoride (Thermoline), HEMA and sodium fluoride (PrepEze), HEMA and gluteraldehyde (Gluma Desensitizer) were applied according to manufacturer instruction to the gently dried dentin surfaces of the specimens in groups 2, 3, and 4, respectively. While a light-curing ormocer-based desensitizing agent (Admira Protect) was used in group 5, a light curing resin-based desensitizing agent (Seal & Protect) was applied to the specimens of group 6.

In experimental groups, the desensitizing agents were applied with a cotton pellet by using gentle but firm rubbing motion. Desensitizing agents were applied as one layer for 30 seconds in groups 2, 3, and 4. Then, the specimens were air-dried. In group 5, the desensitizing agent was applied as one layer for 20 seconds, and then the excess solution was dispersed with a gentle stream of compressed air and was polymerized for 10 seconds. In group 6, desensitizing agent was applied as it was explained for group 5 and the specimens were polymerized for 20 seconds. Polymerization procedures in groups 5 and 6 were done with a light polymerization unit (Astralis 3, Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm^2. All procedures were performed by the same researcher to minimize variation in application technique.

A polytetrafluoroethylene mold (Isoflon, Diemoz, France) with a hole in the center (6 mm diameter × 2 mm deep) was used to apply the self-etching/self-adhesive resin cement (Panavia F 2.0, Kuraray Co. Ltd., Osaka, Japan) to the dentin surfaces. Before the application of resin cement, one drop each of primers A and B (ED Primer, Kuraray Co. Ltd., Osaka, Japan) were mixed for 5 seconds and applied to the dentin surface with a microbrush for 10 seconds. Excess primer solution was removed with cotton pellets, and the

TABLE 1. Desensitizing agents used in this study

Material	Composition	Batch number	Manufacturer
Thermoline	1% Sodium fluoride, 1% calcium fluoride ethyl acetate cellulose	490261	Voco, Cuxhaven, Germany
PrepEze	5% Benzalkonium chloride, 0.5% sodium fluoride, 35% HEMA	102920	Pentron, Wallingford, CT, USA
Gluma Desensitizer	35% HEMA, 5% glutaraldehyde, water	1010	Heraeus Kulzer, Hanau, Germany
Admira Protect	Mixture of different dimethacrylates (HEMA, HPMA, BisGMA), acetone, catalysts, ormocers, and additives	0300007 105	Voco, Cuxhaven, Germany
Seal & Protect	Methacrylate resins, PENTA, nanofillers, triclosan (a broad spectrum antibacterial agent), and acetone, photoinitiators, and stabilizers	B1N32	Dentsply, York, PA, USA

BisGMA = bis-phenol-A-diglycidylmethacrylate; HEMA = hydroxyethylmetacrylate; HPMA = 2-hydroxypropyl methacrylate; PENTA = diphentaerythritol pente-acrylateomonophosphate.

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primer was then gently air-dried. For cementation, equal amounts of a dual-polymerized resin luting agent base and catalyst paste were mixed and transferred to the hole with a plastic spatula. The resin cement was then light polymerized (Astralis 3) for 20 seconds at an output power of 600 mW/cm^2. After removal of the mold, the specimens were stored in distilled water at 37°C ± 2°C for 24 hours before testing.

Shear bond strength testing was performed with a universal testing machine (Lloyd LRX, Lloyd Instruments PIC., Fareham, Hampshire, England) at a crosshead speed of 0.5 mm/minute. The Kolmogorov–Simirnov test showed that the data had a normal distribution ($p > 0.05$). Homogeneity of variance test was done with Levene's test (F: 3.938, $p < 0.05$). Means and standard deviations of bond strengths were calculated and mean values were compared by one-way analysis of variance (ANOVA; SPSS 12.0, SPSS Inc., Chicago, IL, USA), followed by a multiple comparison test performed with a Tukey 's Honestly Significant Difference (HSD) test ($\alpha = 0.05$).

To evaluate changes in the dentin surface of the specimens after the application of the desensitizing agents, an additional specimen representing each group was air-dried overnight and gold sputtered with a sputter coater (S150B, Edwards, Crawley, England), and examined by means of a field emission SEM (JSM-6335F, JEOL Ltd., Tokyo, Japan) at 15.0 or 20.0 kV. No resin cement was applied to these specimens. The SEM photomicrographs were developed at 1,000× magnification for visual assessment.

TABLE 2. One-way ANOVA results

	Sum of squares	df	Mean square	*F*	Sig.
Between groups	486.82	5	97.36	54.79	0.0001
Within groups	95.96	54	1.79		
Total	582.78	59			

TABLE 3. Mean and SD values for shear bond strength (MPa)

Groups	Desensitizing agent	Mean ± SD
1	Untreated (control)	6.96 ± 1.06a
2	Thermoline	6.89 ± 0.49a
3	PrepEze	14.25 ± 1.19c
4	Gluma Desensitizer	9.36 ± 1.32b
5	Admira Protect	10.27 ± 1.65b
6	Seal & Protect	13.38 ± 1.84c

Values having same letter were not significantly different for Tukey test ($p < 0.05$).

RESULTS

The results of one-way ANOVA revealed a significant difference among the groups ($p < 0.05$; Table 2). The mean shear bond strength values and the differences between groups are shown in Table 3. PrepEze, Seal & Protect, Gluma Desensitizer, and Admira Protect all showed a significantly higher bond strength than the control and Thermoline ($p < 0.05$). Application of the HEMA and sodium fluoride containing desensitizing agent (PrepEze) showed the highest bond strength value (14.25 ± 1.19 MPa), but no significant difference was found between it and the resin-based desensitizing agent Seal & Protect (13.38 ± 1.84 MPa) ($p > 0.05$). Lower bond strength values were obtained for the desensitizing agent containing sodium and calcium fluoride (Thermoline) (6.89 ± 0.49 MPa) and for the control group (6.96 ± 1.06 MPa), and no significant difference was found between these groups ($p > 0.05$). There were no significant differences between Gluma Desensitizer (9.36 ± 1.32) and the light-cured ormocer-based desensitizing agent Admira Protect (10.27 ± 1.65 MPa) ($p > 0.05$).

The microstructures of the five dentin surfaces treated with different types of desensitizing agent are shown in Figure 1. Groups are labeled A–F as follows. In the control specimen (A), exposed dentin tubules were observed. In the other specimens, SEM evaluation revealed that the dentin tubules were covered with desensitizing agent (B–F). Dentinal tubule surfaces were sealed with precipitants of calcium and sodium fluoride

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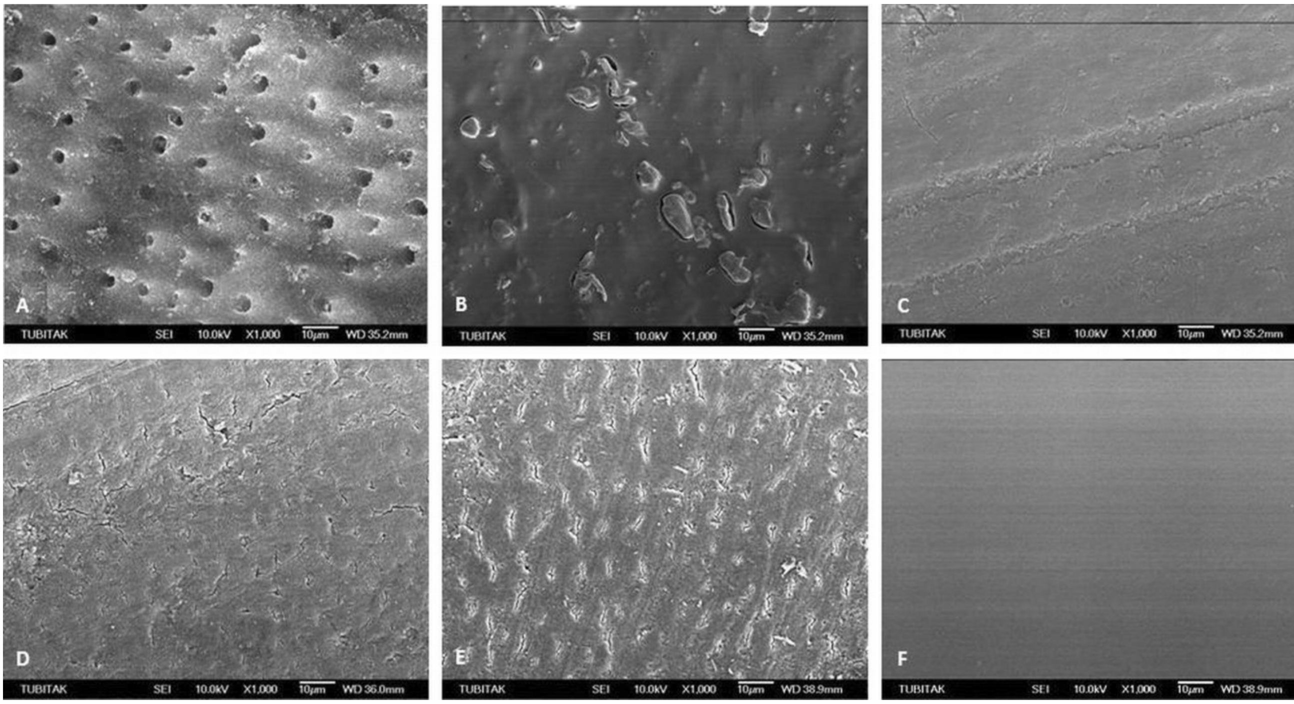

FIGURE 1. Scanning electron microscopic views of the dentin surface after treatment with the desensitizing agents: A, control; B, Thermoline; C, PrepEze; D, Gluma Desensitizer; E, Admira Protect; and F, Seal & Protect.

salts in the specimen treated with Thermoline (B). In the specimen treated with Gluma Desensitizer (D), dentinal tubules were completely occluded with amorphous material. A smoother, resin-coated dentin surface was obtained with the use of a resin-based desensitizing agent (F).

DISCUSSION

The present study hypothesized that desensitizing agents reduce the bond strength of adhesive resin cement to dentin. On the contrary, this study demonstrated that bond strength values increased significantly with the use of most of the desensitizing agents ($p < 0.001$), with the exception of Thermoline, which had the highest fluoride content. Also in the present study, the group treated with Thermoline desensitizing agent showed the lowest bond strength value (6.89 MPa), but no significant difference was found with the control group (6.96 MPa). In self-etching/self-adhesive systems, it is essential that the monomers infiltrate into dentin for the creation of a resin-infiltrated layer.[15] A possible explanation for the Thermoline result is that sodium and calcium fluoride crystals were deposited in the dentin tubules and that they inhibited resin monomer infiltration. These crystals are acid resistant and may chemically and physically prevent complete penetration of the resin components of adhesive resin cement.[31]

Earlier studies showed that the application of fluoride-containing solutions increased[31] or did not affect[29] the bond strength of self-etching adhesive resin cement to demineralized dentin, when compared with nontreated control specimens.[29,31] However, more recent studies have reported lower bond strength values when the fluoride-containing agent was applied to sound dentin.[15,32] Sarac and colleagues demonstrated that an increase in the amount of fluoride in desensitizing agents decreased the bond strength of adhesive resin cement to dentin. They stated that an increase in precipitated crystals on the dentin surface because of a higher amount of fluoride resulted in lower bond strength.[32]

Swift and colleagues[1] reported that the use of the resin primer (Gluma Desensitizer) or the adhesive system

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(One-Step) had no effect on the retentive properties of zinc phosphate, glass ionomer and resin-modified glass ionomer cement.[1] In another study, it was stated that treatment with AD Gel (Kuraray) (10% sodium hypochlorite) before application of luting agents (Panavia F, Super-Bond G%B) produced optimum bond strength, irrespective of the presence of desensitizing agents (MS Coat, Sun Medical, Shiga, Japan; Saforide, Toyo Seiyaku Kasei Co., Osaka, Japan; Gluma GPS Desensitizer). It was thought that AD Gel conditioning is useful for removing the desensitizing agents from the dentin surface.[15]

Gluma Desensitizer consists of 5% glutaraldehyde and 35% HEMA.[33] The glutaraldehyde reacts with protein, producing precipitation on the dentin surface. The diffusion of monomers into dentin is likely to be accelerated by HEMA, despite such precipitation.[15,34,35] In contrast to previous studies,[1,15,18,36] the group treated with Gluma Desensitizer showed a higher bond strength than the control group ($p < 0.05$), but lower bond strength was obtained from the groups treated with PrepEze and Seal & Protect. There was no significant difference between the groups treated with Gluma Desensitizer and Admira Protect ($p > 0.05$). Admira Protect is a fluoride-releasing, one-component ormocer-based (ormocer = ORganically MOdified CERamic) product.[37] Ormocer materials contain inorganic-organic copolymers, in addition to silanized, inorganic filler particles. Admira Protect reduces hypersensitivity by sealing dentin tubules.[37] Bakes and colleagues found that the same agent reduced root dentin demineralization by forming precipitates in the tubules.[37]

In the present study, a self-etching/self-adhesive resin cement was used. It conditions and primes the enamel and dentin surfaces, without rinsing. For this reason, desensitizing agents can not be completely removed from the dentin surface and their remnants can decrease bond strength. Thermoline, Gluma Desensitizer, and Admira Protect appeared to prevent the primer and resin cement of self-etching/self-adhesive resin cement (Panavia F 2.0) from interacting with the dentin, resulting in lower bond strength relative to PrepEze and Seal & Protect.

Huh and colleagues[23] evaluated the effects of four different desensitizing agents (SuperSeal; Copalite Varnish, Cooley and Cooley, Houston, TX, USA; MS Coat; and Gluma) on the bond strength of a self-etching adhesive resin cement to dentin. They found that the group treated with Gluma Desensitizer showed a lower bond strength value than the SuperSeal and control groups. SuperSeal desensitizing agent does not contain a resin component but it is acidic enough to remove the smear layer and replace it with a layer of calcium oxalate crystals.[23] However, Gluma Desensitizer contains HEMA and glutaraldehyde, which cause the coagulation of dentin fluid protein in the dentinal tubules and plugging of the tubules. The same authors also stated that crystal precipitation on the dentin surface could cause reduction in the bond strength.[23]

Mausner and colleagues[7] evaluated the effects of two different resin-based desensitizing agents on the bond strength of three conventional cements and a resin-based cement to dentin. Although the resin-based desensitizing agents reduced the bond strength of the conventional cements, it increased the bond strength of the resin-based cement to dentin. The authors stated that when polymeric resins are used as desensitizers, they seal the tubules and interact with the altered intertubular dentin. Primers A and B of All-Bond (Bisco, Schaumburg, IL, USA) desensitizing agent contain N-phenylglycine glycyl methacrylate, biphenyl dimethacrylate (BPDM) acetone, ethanol, and water. BPDM is the hydrophilic primer that penetrates the altered dentinal surfaces.[7] In the present study, the highest mean bond strength value was obtained from the group treated with PrepEze, but no significant difference was found with the Seal & Protect group. PrepEze desensitizing agent contains benzalkonium chloride, sodium fluoride, and HEMA. HEMA, as an example of a hydrophilic primer, is used to improve the infiltration of adhesive monomers into demineralized dentin by wetting the surface of collagen fibers, and it also maintains the collagen network in an expanded state by stiffening the collagen fibers.[16,22] Seal & Protect is a resin-based desensitizing agent and consists of methacrylate resins, dipentaerythritol pente-acrylateomonophosphate (PENTA), nanofillers, triclosan, acetone, photo initiators, and stabilizers.

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Malkoç and colleagues[21] evaluated the effects of three antimicrobial agents (MicroPrime, Danville Engineering, San Ramon, California, USA; Seal & Protect; Gluma Desensitizer) on the bond strength of an orthodontic composite resin to dentin after acid etching. Seal & Protect, which contains triclosan and PENTA, showed a higher bond strength value than Gluma Desensitizer and MicroPrime.[21]

One limitation of this study was that only one adhesive resin cement was used. In future studies, it would be advantageous to compare different adhesive resin cements. In this study, it is aimed to evaluate the effects of desensitizing agents, and to standardize the conditions, only one adhesive resin cement, which is generally used by most clinicians currently, was used. In a further study, it would be advantageous to use different adhesive resin cements. In addition, all desensitizing agents were applied according to manufacturers' recommendations. The thickness of desensitizing agents may play a role on the bond strength of adhesive resin cement. Also different results may have been obtained under different conditions such as aging or fatiguing of specimens or with different adhesive resin cements and desensitizing agents.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

1 Desensitizing agents affected the bond strength of a resin cement to dentin ($p < 0.001$).
2 All the desensitizing agents, except for one containing sodium and calcium fluoride (Thermoline), increased the bond strength of resin cements to dentin.
3 The resin-based desensitizing agent (Seal & Protect) and HEMA and benzalkonium chloride-based desensitizing agent (PrepEze) showed the highest bond strength values.

The authors of the current study conclude that the choice of desensitizing agent may have a significant effect on the strength of bonding between adhesive resin cement and dentin. It is therefore recommended that practitioners take into account their choice of desensitizing agent in cementation procedure.

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