

RESEARCH AND EDUCATION

Effect of fiber post space irrigation with different peracetic acid formulations on the bond strength and penetration into the dentinal tubules of self-etching resin cement



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Fiber posts are indicated for the restoration of extensively damaged endodontically treated teeth because they have a satisfactory elastic modulus and can reduce the risk of root fracture.¹⁻³ Before cementation of the fiber post, the root canal must be prepared by partially removing the endodontic filling.⁴

The space corresponding to the cervical and middle thirds and the underlying dentin of the root canal is vulnerable to microbial contamination after the intraroot preparation.⁵ To provide local antiseptics, several irrigation solutions similar to those used during endodontic treatment have been recommended for the prepared space.^{6,7}

Sodium hypochlorite is the most recommended solution during endodontic treatment but does not satisfactorily remove the smear layer from the dentin surface and/or negatively interferes with the adhesive system of resin

ABSTRACT

Statement of problem. The post space must be irrigated with solutions that do not interfere with the bond strength and enhance penetration into the dentinal tubules of self-etching resin cement. Which solution is best is unclear. Peracetic acid with different formulations appears to be a good option.

Purpose. The purpose of this in vitro study was to evaluate the effect of irrigating the fiber post space by using 1% peracetic acid (PA) at low (LH) or high (HH) hydrogen peroxide concentration on the bond strength and penetration into the dentinal tubules of RelyX U200 self-etching resin cement.

Material and methods. After intraroot preparation for fiber posts in 60 endodontically treated teeth, the specimens were divided into 4 groups (n=15) according to the irrigation protocol: CG (control), distilled water; NA (NaOCl), 2.5% sodium hypochlorite; LHPA, PA with a low concentration of hydrogen peroxide; and HHPA, PA with a high concentration of hydrogen peroxide. The fiber posts were then cemented with self-etching resin cement labeled with rhodamine. Cross sections of the thirds were obtained by confocal scanning microscopy and submitted to the push-out and cement penetration tests. The data obtained in the push-out tests were evaluated by using ANOVA and the Tukey post hoc test, and those data obtained in the confocal screening were evaluated by using the Kruskal-Wallis and Dunn (Bonferroni correction) tests ($\alpha=.05$).

Results. Bond strength values in the cervical third of the CG were higher than those in NA ($P=.035$) but similar to those of the other groups ($P=.05$). In the other thirds, CG and LHPA values were similar to one another ($P=.05$) but higher than those of NA and HHPA ($P<.001$). The penetration rate assessed by confocal scanning microscopy revealed similar results in the cervical third for all groups ($P=.075$), except for NA which provided the lowest penetration. In the remaining thirds, CG and LHPA provided the highest penetration rate ($P=.015$).

Conclusions. The LHPA group had no negative effects on the bond strength and penetration into the dentinal tubules of the self-etching resin cement. (*J Prosthet Dent* 2019;122:46.e1-e7)

cements to root dentin.⁷⁻⁹ Other substances proposed for this purpose include chlorhexidine digluconate, phosphoric acid, and ethylenediaminetetraacetic acid

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Clinical Implications

The post space must be irrigated with solutions that do not interfere with the bond strength and penetration into the dentinal tubules of self-etching resin cement. Peracetic acid appears to be a good option, although the effects of different formulations on root dentin have yet to be determined.

(EDTA).^{7,10-14} However, their adverse effects and/or reduced antimicrobial properties are disadvantages.⁶

A consensus as to the ideal solution for fiber post space irrigation is lacking. Peracetic acid may be an option given its satisfactory antimicrobial activity and dentin surface cleaning properties and its effectiveness as a solution for root canal irrigation.^{10,15-18}

In aqueous solution, peracetic acid is in chemical equilibrium with hydrogen peroxide, acetic acid, and acetyl hydroperoxide.¹⁹ Its antimicrobial action comes from the release of free oxygen and hydroxyl radicals, which decompose into oxygen and acetic acid.^{16,20} Solutions of peracetic acid at different hydrogen peroxide concentrations are commercially available.^{21,22}

The presence of free radicals in a substance can adversely affect adhesive techniques, as oxidation interferes with the polymerization of resin monomers.⁷ Although the decomposition of the peracetic acid solution produces free radicals, its effects on the bonding of resin cements to root dentin are unclear, mainly because solutions are available at several different hydrogen peroxide concentrations.²³⁻³¹

Self-etching resin cement, which has similar dentin bond strength to conventional resin cements, has been used to optimize the cementation of fiber posts.³²⁻⁴¹ However, the effects of oxidizing agents on adhesive bonding are unclear, especially after the irrigation of the fiber post space with peracetic acid solutions. As the higher concentration of hydrogen peroxide in the peracetic acid solution also interferes with the incidence of smear layer and the presence of open dentinal tubules in the prepared post space, whether this phenomenon could also interfere with the adhesion of composite resin cements to the root dentin is unclear.⁴²

The purpose of this *in vitro* study was to evaluate the effect on the bond strength and penetration into the dentinal tubules of self-etching resin cement (RelyX U200) after cementation in the cervical, middle, and apical thirds of fiber post space irrigation by using 1% peracetic acid with either a low or high concentration of hydrogen peroxide. The null hypotheses were that peracetic acid solutions at different concentrations of hydrogen peroxide would not interfere with the bond

strength and penetration into the dentinal tubules in the different root thirds of the self-etching resin cement.

MATERIAL AND METHODS

The research project was reviewed and approved by the research ethics committee of the State University of Sao Paulo. Sixty human maxillary central incisors with similar root anatomy, recently extracted because of periodontal disease, were stored in 0.1% thymol solution at 4 °C as described by Belizário et al.⁴² After the teeth had been rinsed under running water, the teeth were sectioned transversely along the root axis by using a diamond disk (IsoMet 2000; Buehler) under refrigeration. The roots were sectioned at 17 mm, and the canals were instrumented to 16 mm from the root apex. The post spaces were prepared by using ProTaper instruments (Dentsply Sirona) up to F5, following the manufacturer's recommendations. At each instrument change, irrigation was repeated with 5 mL of 2.5% sodium hypochlorite solution (Asfer). The apical foramens were sealed with a composite resin to prevent the irrigation solution from escaping.

After a chemical-mechanical preparation, the root canals were irrigated with 5 mL of 17% EDTA (Bio-dinâmica) for 3 minutes and irrigated again with 5 mL of 2.5% sodium hypochlorite. After drying with an absorbent paper point, they were sealed by using the single-cone technique and epoxy resin-based cement (AH Plus; Dentsply Sirona), as described by Rodrigues et al.⁴³

After interim restoration of the root canal cervical opening by using interim restorative material (Coltosol; Coltène), the roots were immediately immersed in distilled water and stored at 37 °C for 7 days. The roots were then placed in plastic matrices, as described by Victorino et al.⁴⁴

After 24 hours, the fiber post space was prepared by using a #2 rotary instrument (White Post DC System; FGM) to 11 mm from the cervical root face. The roots were divided into 4 groups (n=15) according to the final irrigation solution used in the prepared intraroot space: CG (control, DW), distilled water; NA (NaOCl), 2.5% sodium hypochlorite solution (Asfer); LHPA, 1% peracetic acid solution (Sigma-Aldrich) with a low concentration of hydrogen peroxide (40% peracetic acid and 8% hydrogen peroxide); and HHPA, 1% peracetic acid solution (Peresal; Profilática) with a high concentration of hydrogen peroxide (4% peracetic acid and 26% hydrogen peroxide). An estimation of an appropriate sample size was obtained from the results of a pilot study, $\alpha=.05$ and $\beta=.02$.

The prepared root canal space was irrigated with 5 mL of the selected solution for 1 minute and held in place for 3 minutes without agitation. After irrigation, the root canal was aspirated by using a 0.48-mm-diameter

suction tube (Capillary Tips; Ultradent Products, Inc) and dried by using size F5 absorbent paper points (ProTaper; Dentsply Sirona). The external surface of the #2 fiber posts (White Post DC; FGM) was cleaned with 70% alcohol, and then silane (Prosil; FGM) was applied along the whole extension and immobilized for 1 minute.

Rhodamine B (Sigma-Aldrich) was incorporated into the self-etching resin cement (RelyX U200; 3M ESPE) in the proportion of 0.01% (mass/mass) to allow visualization by confocal laser scanning microscopy. The cement was inserted into the space prepared for the post by using a Rhein tip (SS White Duflex), and the fiber post was then positioned and stabilized in the root canal. After the removal of excess cement in the cervical root, the material was photoactivated by using a light-emitting diode (LED) laser system (Bluephase G2, 1200 mW/cm²; Ivoclar Vivadent AG) for 40 seconds. The specimens were kept in distilled water at 37 °C for 7 days.

After 7 days, the roots were sectioned transversely along the root axis by using a diamond disk in a sectioning machine (IsoMet 2000; Buehler) under water cooling. Three sections were obtained from the apical, middle, and cervical thirds of each root, with a thickness of 2.0 ±0.1 mm. The cervical, middle, and apical sections were obtained at 1.0 mm, 5.0 mm, and 8.0 mm, respectively, from the cervical root face. The irregularities of the sections were removed with 1200-grit silicon carbide paper (Norton), and the sections were then cleaned by using a brush and compressed air.

Push-out tests were performed in each of the sections by using an electromechanical test machine (EMIC DL 2000; EMIC Equipment System and Test Ltd) with a load cell of 5 kN and operating at a speed of 0.5 mm/min. In all sections, the apical face was positioned to contact the tip of the device for displacement, with diameters of 1.30, 0.9, and 0.5 for cervical, middle, and apical thirds, respectively, so that the force was always applied from the apical to the cervical direction. The maximum bond strength was obtained by measuring the displacement of the fiber post and resin cement from the root canal. The data obtained in Newtons (N) were converted into megapascals (MPa), as described by Magro et al.⁴⁵

After the push-out tests, images of cement penetration into the root dentin were obtained by laser confocal scanning microscopy (Leica) at ×100 magnification. Each image was imported into the Image J program to calculate the percentage of cement penetration around the root canal, and the circumference of the root canal was measured. The area of the dentin in which the cement penetration occurred was then obtained by using the same procedure as previously described. The percentage of cement penetration in relation to the diameter of the root canal was obtained by following the method described by Ordinola-Zapata et al.⁴⁶

Table 1. Mean and standard deviation of bond strength values (MPa) of self-etching resin cement (U200) in root dentin in relation to different irrigating solutions and root thirds

Location	CG	NA	LHPA	HHPA	P
Cervical	3.38 ±1.48 ^a	1.75 ±1.16 ^b	3.01 ±1.58 ^{ab}	2.77 ±1.87 ^{ab}	.035
Middle	4.90 ±1.69 ^a	1.70 ±1.23 ^b	4.08 ±1.63 ^a	1.80 ±1.03 ^b	<.001
Apical	3.99 ±0.81 ^a	2.12 ±0.63 ^b	4.12 ±0.68 ^a	1.97 ±0.52 ^b	<.001

CG, distilled water; HHPA, peracetic acid solution at high concentration of hydrogen peroxide; LHPA, peracetic acid solution at low hydrogen peroxide concentration; NA, 2.5% sodium hypochlorite. Different letters in same row show statistically significant difference.

Each specimen was analyzed under a stereomicroscope at ×20 magnification to determine the fracture pattern following the categories described by Elnaghy⁴⁷: type 1, adhesive between fiber post and resin cement; type 2, cohesive within the resin cement; type 3, adhesive between the dentin and resin cement; and type 4, mixed. The data obtained in the push-out tests were submitted for ANOVA and Tukey post hoc tests. The data obtained in the cement penetration test in dentin were submitted for the Kruskal-Wallis and Dunn tests ($\alpha=.05$ for all tests).

RESULTS

Table 1 shows the mean and standard deviation of the bond strength values (in MPa) in the cervical, middle, and apical thirds. Of the self-etching resin cements, only CG showed higher mean bond strength to root dentin in the cervical third than NA ($P=.035$). The other groups were similar to one another ($P=.06$). In the middle third and apical root, CG and LHPA showed similar values ($P=.051$), and these were higher than those of NA and HH ($P=.001$), which, in turn, were similar to one another ($P=.070$).

Table 2 shows the median, maximum, and minimum values of the percentage in which the resin cement penetrated the dentinal tubules. The groups presented similar values in the cervical third ($P=.072$). However, in the middle and apical thirds, the penetration of the resin cement into the dentinal tubules was similar between CG and LHPA ($P=.015$), and both were higher than that demonstrated in HHPA and NA ($P=.015$), which were similar to one another ($P=.015$). Figures 1-3 illustrate the cement penetration pattern in the dentinal tubules for each evaluated group in the cervical, middle, and apical thirds. Table 3 shows the percentage of fracture occurrence for each group evaluated.

DISCUSSION

The null hypothesis was rejected because the solution with a high concentration of hydrogen peroxide had a negative effect on the parameters evaluated in the middle and apical thirds. In contrast, the 1% peracetic acid solution at a low concentration of hydrogen peroxide

Table 2. Median and maximum and minimum values of penetration area (in percentage) of self-etching resin cement (U200) into dentinal tubules in relation to root canal area and different irrigating solutions and root thirds

Location	CG	NA	LHPA	HHPA	P
Cervical	8.1 ±25.3-1.01 ^a	9.2 ±23.1-0.0 ^a	12.1 ±22.0-0.0 ^a	9.02 ±90.0-0.1 ^a	.72
Middle	4.3 ±8.0-3.1 ^a	1.4 ±4.2-0.2 ^b	3.7 ±7.9-1.5 ^a	1.50 ±3.9-0.1 ^b	.015
Apical	3.9 ±5.7-2.9 ^a	2.3 ±3.5-1.1 ^b	4.4 ±4.9-2.7 ^a	1.9 ±2.9-1.2 ^b	.015

CG, distilled water; HHPA, peracetic acid solution at high concentration of hydrogen peroxide; LHPA, peracetic acid solution at low hydrogen peroxide concentration; NA, 2.5% sodium hypochlorite. Different letters in same row show statistically significant difference.

showed no effect on the bond strength in the dentin and penetration into the dentinal tubule of the self-etching resin cement (U200), no matter which third was evaluated.

The bond strength of adhesive resin cement to root dentin involves chemical and mechanical processes.^{26,27} A chemical interaction occurs between the adhesive system and dentin substrate, and mechanical penetration of the resin cement and/or adhesive system into the dentin tubules leads to the formation of tags.^{26,36} In vitro studies have demonstrated the superiority of self-adhesive cements (absence of classical hybrid layer) when compared with conventional dual-polymerized resin cements (requires a hybrid layer).²⁹⁻³¹ This superiority may be related to the bond mechanism of self-adhesive resin cements, which have a strong chemical interaction with the dentin hydroxyapatite,³⁵ because low demineralization³² and the absence of a hybrid layer have been described.^{35,40} Additionally, the self-adhesive luting cements are considered moisture tolerant because the phosphoric acid ester needs a wet dentin to create the chemical interaction with hydroxyapatite.³⁹ The push-out test and confocal scanning microscopy are the most commonly used methods for evaluating these phenomena.

The solution used to irrigate the post space had no effect on the bond strength of the resin cement (U200) in the dentin of the cervical third of the root. The anatomy and/or proximity to the photoactivation source showed little change at different concentrations of hydrogen peroxide, which is consistent with the findings of Matsumoto et al.³⁸ The dentinal tubules have a larger diameter⁴⁶ and a higher density³⁴ in the cervical third than in the other thirds; therefore, increased resin cement penetration is expected, improving the bond.

The proximity of the photoactivation source in the cervical third would also be expected to improve the bond, with increased polymerization of the resin.^{33,41} The complex shape of the root canal hinders apical light propagation, as described by Lindberg et al.³⁷

Bonding in the middle and apical thirds may have been reduced because the high concentration of

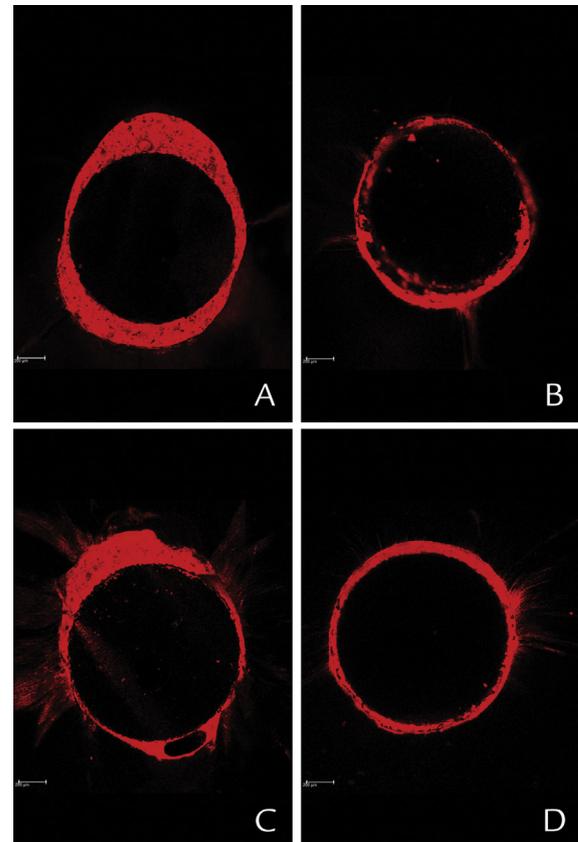


Figure 1. Representative image of resin self-etching cement penetration into dentin of root cervical third. A, CG: distilled water (control). B, NA: 2.5% sodium hypochlorite. C, LH: peracetic acid solution with low concentration of hydrogen peroxide. D, HH: peracetic acid solution with high concentration of hydrogen peroxide. Original magnification $\times 20$.

hydrogen peroxide solution interfered negatively with the results of the push-out test. This can be explained by the release of free oxygen from the chemical decomposition of the peracetic acid solution,¹⁹ interfering with resin polymerization and therefore bonding.²⁴ As observed in the present study, the higher the concentration of peroxides and free radicals in a solution, the greater the negative effect on bonding.^{22,25}

Acid solutions remove the smear layer from the dentin surface.¹⁴⁻¹⁶ Acetic acid, present in the peracetic acid solution, is a weak acid and cleans residue from the dentin surface.^{15,16} Although the peracetic acid solution with a low concentration of hydrogen peroxide consists of 40% acetic acid, approximately 10 times more concentrated than the other solution, it is unlikely that this concentration explains the results of the push-out test in the middle and apical thirds of the root. In the case of EDTA and citric acid, no significant differences were observed.^{11,12} The higher concentration of hydrogen peroxide probably interfered with smear layer removal, which is consistent with the results of Scelza

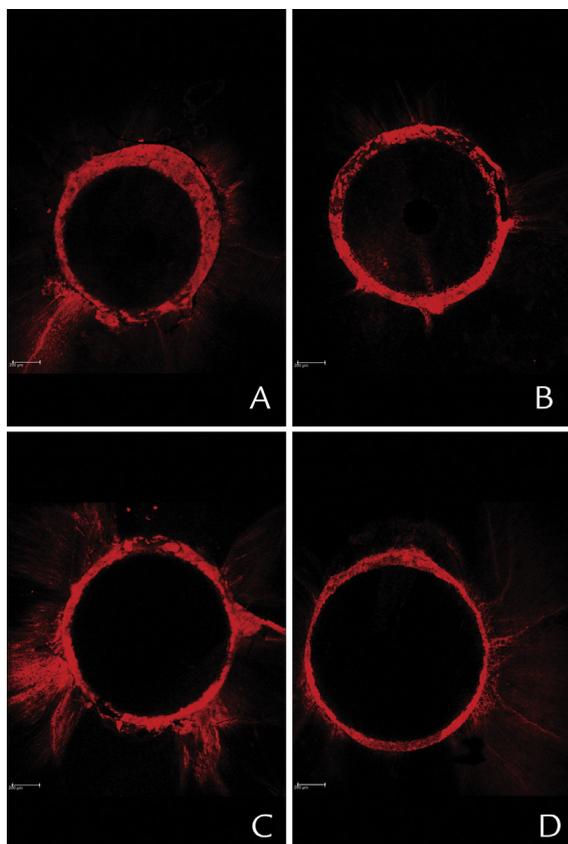


Figure 2. Representative image of resin self-etching cement penetration into dentin of middle third of root. A, CG: distilled water (control). B, NA: 2.5% sodium hypochlorite. C, LH: peracetic acid solution with low concentration of hydrogen peroxide. D, HH: peracetic acid solution with high concentration of hydrogen peroxide. Original magnification $\times 20$.

et al¹³ who reported that smear layer removal was lowest when hydrogen peroxide was used as an irrigation agent. Reduced smear layer removal negatively affects penetration into the dentinal tubules of the self-etching resin cement.

Confocal scanning microscopy detected a significant difference in cement penetration into the dentinal tubules only in the middle and apical thirds. In addition to the ability to remove the smear layer, the reduced flow of the resin cement may be a factor in this finding.³⁸ The shape of the dentinal tubules helps the resin cement to penetrate,⁴⁶ leading to similar bond strengths, no matter which irrigation solution is used in the post space.

The most frequent fracture pattern for NA and HHPA was type 3, at the interface between the dentin and self-etching resin cement. This is consistent with the push-out test results and suggests incomplete polymerization of the cement. In contrast, in CG and LHPA groups, a higher frequency of the mixed fracture pattern suggests lower interference of the peracetic acid solution at a low concentration of hydrogen peroxide in the bonding process of the self-etching resin cement than other solutions.

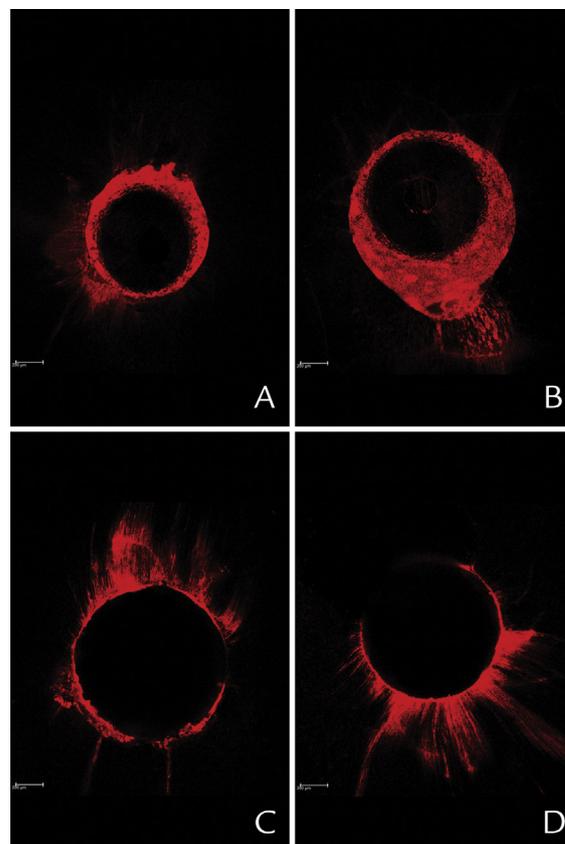


Figure 3. Representative image of self-etching resin cement penetration into dentin of apical third of root. A, CG: distilled water (control). B, NA: 2.5% sodium hypochlorite. C, LH: peracetic acid solution with low concentration of hydrogen peroxide. D, HH: peracetic acid solution with high concentration of hydrogen peroxide. Original magnification $\times 20$.

Table 3. Frequency of fracture pattern occurring as a result of irrigation solution used to prepare fiber post space

Pattern	CG	NA	LHPA	HHPA
Type 1	18.89	7.00	17.12	11.34
Type 2	28.88	27.00	15.55	15.56
Type 3	18.89	28.00	13.99	26.43
Type 4	33.35	38.00	53.33	46.66

CG, distilled water; HHPA, peracetic acid solution at high concentration of hydrogen peroxide; LHPA, peracetic acid solution at low hydrogen peroxide concentration; NA, 2.5% sodium hypochlorite. Type 1, adhesive between fiber post and resin cement; Type 2, cohesive within resin cement; Type 3, adhesive between the dentin and resin cement; Type 4, mixed.

The present study demonstrated the possibility of using peracetic acid as a post space irrigation agent because of its dentin-cleaning properties and antimicrobial activity. However, further studies should be conducted to elucidate possible interference in other types of resin cements and/or adhesive systems.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The 1% peracetic acid solution at a low hydrogen peroxide concentration did not interfere with the bond strength of the self-etching resin cement (U200) to dentin.
2. The solution with low hydrogen peroxide concentration did not interfere in the penetration into the dentinal tubules, regardless of the third of the root evaluated.

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