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Influence of Light-Curing Mode on Glass Fiber Post Cementation



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**Laiza Tatiana Poskus^a, Eduardo Moreira da Silva^a,
Luísa Chateaubriand Pegado^a, Viviane Hass^b, Glauco
Botelho dos Santos^a, Alice Gonçalves Penelas^{a*}, José
Guilherme Antunes Guimarães^a**

*a Analytical Laboratory of Restorative Biomaterials –
LABiom-R, Universidade Federal Fluminense / School
of Dentistry, Rua Mário Santos Braga, nº 30 - Campus
Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-
140.*

*b Departamento de Dentística, Universidade Estadual
de Ponta Grossa, Praça Santos Andrade, nº 01 - Centro,
Ponta Grossa, PR, Brazil – CEP 84010-330.*

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ABSTRACT

This study evaluated the Bond Strength (BS) of Glass Fiber Posts (GFP) to root dentin and the Degree of Conversion (DC%) of a resin cement under different Light-Curing Modes (LCM). For BS analysis, canals of seventy-eight decoronated bovine incisors were prepared and irrigated with 2.5% NaOCl. After root canal dentin hybridization and GFP conditioning (24% H₂O₂/1min) and silanization, specimens were distributed into 6 groups (n=10) according to LCM selected for resin cement polymerization: -CH (conventional/high irradiance) – 1050mW/cm²/23s; -RH (ramp soft-start/high irradiance) – 0-1050mW/cm²/5s + 1050mW/cm²/20s; -SH (step soft-start/high irradiance) – 100mW/cm²/5s + 1050mW/cm²/22s; CL (conventional/low irradiance) – 600mW/cm²/40s; RL (ramp soft-start/low irradiance) – 0-600mW/cm²/5s + 600mW/cm²/37s; SL (step soft-start/low irradiance) – 100mW/cm²/5s + 600mW/cm²/39s. BS was evaluated using push-out test according to root region: Cervical (C), Middle (M) and Apical (A). Micro-Raman spectroscopy was used to analyze DC% at the resin cement layer. Data were subjected to two-way ANOVA and Fisher LSD test ($\alpha=0.05$), and to Pearson correlation test. BS was affected by the LCM as follow: (SL>SH>CL=RL=RH=CH) and (C>M=A) ($p<0.05$). Adhesive failures were predominant at resin cement-dentin interfaces. DC% was also affected by LCM: RH=RL \geq CH=SH>CL=SL, and root third: C>M=A ($p<0.05$). No significant correlation was found between DC% and BS ($p=0.2214$). SL and SH groups presented the highest BS. The highest DC% was presented by the RH, RL and CH groups ($p<0.05$). Both step soft-start light-curing modes were able to improve the retention of the glass-fiber post. Even though the ramp soft-start light-curing modes led to the highest DC%, they did not influence GFP retention.

INTRODUCTION

Due to the esthetic appearance and the easy handling, the use of Glass Fiber Posts (GFP) for endodontically treated teeth rehabilitation became widespread in dentistry. These posts consist of a high amount of continuous reinforcing fibers embedded in a polymeric matrix composed of epoxy resins or other highly converted and cross-linked polymers. (1) This architecture guarantees suitable mechanical properties to the GFP, as well as elastic properties close to those of the dentin.

The cementation of GFP involves the use of resin-based cements, which allows better bond strength to the intraradicular dentin. As a polymeric restorative material, however, resin cements undergo shrinkage during their polymerization reaction. This phenomenon relies on the reduction of the intermolecular spaces between the dimethacrylate monomers of the polymeric matrix due to the conversion of C=C bonds (0.3-0.4 nm) into C-C bonds (0.15 nm). Clinically, this volumetric reduction is capable of developing stresses at the adhesive interface, which might jeopardize the interaction between resin cement and dentin (2). Among other aspects, the cavity configuration factor (C-factor) – the ratio of the bonded to the unbonded surface area – strongly influences the interfacial stresses generated during the resin-based materials polymerization (2). For root canals, this value may exceed 200, which, in turn, makes adhesion at this region highly critical (3). This is a clinical matter of concern.

It has been shown that Light-Curing Mode (LCM) could influence the stresses generated at the resin-dentin interface (4). Slow rate LCM were extensively studied, as the lower irradiances would allow an extended pre-gel phase of polymerization, and, consequently, the monomer molecules would have more time to occupy new positions inside the polymeric matrix, thereby minimizing the stresses at the adhesive interface (5,6). Among these modes, the ramp soft-start starts with a low irradiance which increases exponentially, followed by a higher and constant irradiance until the end of the exposure. Differently, the step soft-start mode starts with a constant low irradiance for a specific time and abruptly changes to a higher irradiance to complete its cycle.

Although several studies have evaluated the influence of the LCM on bond strength and degree of conversion (DC%) of resin composites, (5,7-8) this subject has been poorly explored in the analysis of resin cements, mostly when these are used for the cementation of GFP (4,9-10). Therefore, the aim of this study was to evaluate the influence of slow rate

polymerization modes on the bond strength of GFP to the root canal dentin and on the DC% of the resin cement used for this purpose. The null hypothesis is that different LCM will not influence the bond strength of GFP, nor DC% of the dual resin cement.

MATERIALS AND METHODS

Specimen preparation

Seventy-eight sound bovine incisors were used for this study (60 for BS and 18 for DC% analyses). After cleaning and disinfection for 7 days in an aqueous solution of 0.5% chloramine, teeth were decoronated below the cement-enamel junction with a low speed diamond saw (IsoMet 1000, Buehler, Lake Bluff, IL, USA) for a root length of 14 mm and stored in distilled water at 37°C. Then, based on the following inclusion criteria, teeth were selected: absence of cracks (observed under a stereomicroscope / SZ61, Olympus, Tokyo, Japan), closed apices and canal coronary diameter less than 2 mm. Measurements were taken with a digital caliper (500-196-20B, Mitutoyo, São Paulo, SP, Brazil).

Canals were prepared under irrigation with 2.5% sodium hypochlorite solution up to the limit of 12 mm length, with a low speed #3 drill indicated by the GFP manufacturer (White Post DC #3, FGM, Joinville, SC, Brazil). Each drill was used to prepare 5 root canals, then discarded and replaced and a resin composite (Oppalis, FGM, Joinville, SC, Brazil) was used to externally seal the root canals apices. Specimens had their external root surfaces covered with black nail varnish to avoid external light transmission. To keep the post parallel to the testing machine longitudinal axis, a dental surveyor (EDG, São Carlos, SP, Brazil) was used to help root embedment in PVC cylinders. Figure 1 shows a schematic representation of the embedded specimen.

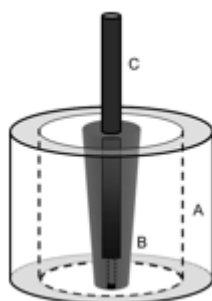


Figure No. 1: Schematic representation of the specimen. In A, the PVC cylinder; In B, the dental root; in C, GFP.

Each GFP was ultrasonically cleaned (Unique, Indaiatuba, SP, Brazil) with distilled water for 10 mins and air dried. After surface treatment with 24% hydrogen peroxide for 1 min, a silane coupling agent (Prosil, FGM, Joinville, SC, Brazil) was applied on the GFP surface with a micro brush (Cavibrush regular, FGM, Joinville, SC, Brazil). The root canal walls were conditioned with 37% phosphoric acid (Condac 37; FGM, Joinville, SC, Brazil) for 15 s, rinsed with distilled water for 30 s and blot-dried with three absorbent paper points (#80, Dentsply Maillefer, Petropolis, RJ, Brazil). A self-curing adhesive (Adper Scotchbond Multi-Purpose Plus, 3M ESPE, St. Paul, MN, USA) was applied with a micro brush (Cavibrush #1, FGM, Joinville, SC, Brazil), following manufacturer instructions.

After specimens had been divided into six experimental groups according to the LCM (Table 1), a dual resin cement (Rely-X ARC, 3M ESPE, St. Paul, MN, USA) was mixed according to the manufacturer instructions and inserted into the root canal with a syringe (Centrix, DFL, Rio de Janeiro, RJ, Brazil). The post was inserted into the canal and, after excess removal, the resin cement was light-polymerized (Emitter D, Schuster, Santa Maria, RS, Brazil). For all experimental groups, the radiant exposure was standardized at 24 J/cm². Irradiance was monitored using a radiometer (RD-7, Ecel, Ribeirão Preto, SP, Brazil) every 5 specimens.

Table No. 1: Experimental Groups

Light-Curing Mode	Protocol
CH (conventional / high irradiance)	1050mW/cm ² /23s
RH (ramp soft-start / high irradiance)	0-1050mW/cm ² /5s + 1050mW/cm ² /20s
SH (step soft-start / high irradiance)	100mW/cm ² /5s + 1050mW/cm ² /22s
CL (conventional / low irradiance)	600mW/cm ² /40s
RL (ramp soft-start / low irradiance)	0-600mW/cm ² /5s + 600mW/cm ² /37s
SL (step soft-start / low irradiance)	100mW/cm ² /5s + 600mW/cm ² /39s

Bond strength analysis

After posts cementation, the specimens (n=10) were immersed in distilled water and stored for 24h at 37°C. Specimens were then sectioned using a diamond saw, under water-cooling, perpendicularly to the post long axis, yielding six 1 mm thick discs. Thus, BS could be assessed in the three root canal regions (cervical, middle and apical). Before the push-out test, the apical and cervical diameters of the post/cement were measured using a stereomicroscope

and a digital caliper. A compressive load was applied, towards apical to cervical, by means of a cylindrical tip attached to a mechanical testing machine (DL2000, EMIC, São Paulo, SP, Brazil) at a 1 mm/min crosshead speed, only on the post area. Failure load for each specimen was recorded (N) and BS (MPa) calculated by the formula: $RU = F/A$, where F was the failure load and A was the adhesive interface area calculated by the formula:

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

Where

$\pi = 3,1416$,

R = cervical diameter of the post/cement,

r = apical diameter of the post/cement

and h = disk thickness.

Failure Pattern

After push-out test, all debonded specimens were examined under a stereomicroscope (40X) to determine the failure patterns, which were classified as: adhesive between cement and root dentin (ACD); adhesive between cement and post (ACP); cohesive (C – within the cement or the dentin or the post); and mixed (M – when more than one failure pattern could be observed)(11).

Degree of Conversion

After post cementation procedure, three specimens of each experimental group were selected and sectioned, as previously described, in two disks for each root third. The disks were wet polished using 1500 and 2000-grit SiC paper for 20 s and ultrasonically cleaned for 10 mins in distilled water. Afterwards, the *in-situ* DC% at the resin cement layer was evaluated using a dispersive micro-Raman spectrometer/microscope (Horiba Scientific Xplora, Villeneuve d'Ascq, France) (12) with a 785 nm diode laser through a X50/0.9 NA air objective. The Raman signal was acquired using a 600-lines/mm grating centered between 500 and 1800 cm^{-1} , and the employed parameters were 100 mW, spatial resolution of 3 μm , spectral resolution 5 cm^{-1} , accumulation time of 20 s with 4 co-additions. Spectra were obtained at the

middle of cement layer, at two random sites *per* disk. Spectra of unpolymerized cement were taken as references. The ratio of C=C bond content of monomer to polymer in the cement was calculated according to the following formula:

$$DC (\%) = \left[1 - \left(\frac{R - \text{cured}}{R - \text{uncured}} \right) \right] \times 100$$

Where ‘R’ is the ratio of aliphatic and aromatic peak intensities at 1639 cm⁻¹ and 1609 cm⁻¹ in polymerized and unpolymerized cement.

Statistical Analysis

The software Statgraphics Centurion XVI (StatPoint Technologies, Warrenton, VA, USA) was used to perform the statistical analysis. Data of the push-out BS and DC% were separately submitted to Levene test to verify homogeneity of variances. Subsequently, data were subjected to two-way ANOVA to determine the significance of the two factors – (1) light-curing mode - LCM and (2) root region (cervical, middle and apical) – and their interaction, and to the Fisher LSD multiple comparison test ($\alpha=0.05$). Additionally, the Pearson test was used to check the correlation between BS and DC%.

RESULTS AND DISCUSSION

Concerning to BS, two-way ANOVA demonstrated statistically significant differences for the two factors – “LCM” and “root region” ($p < 0.05$), but not for their interaction. Table 2 presents the push-out BS means and standard deviations for the experimental groups. It can be noted that -SL exhibited the highest BS values, followed by -SH. The groups -CH, -RH, -CL and -RL showed the lowest BS values and were not statistically different from each other. On Table 3, it can be noticed that the cervical third exhibited significantly higher BS than the middle and the apical regions, which were not statistically different from each other.

The failure pattern distribution is described in Figure 2. Although adhesive failures between the cement and the dentin (ACD) were highly predominant, in SL group the percentage of mixed failures was comparable to ACD’s.

Table No. 2: BS and DC% means and standard deviation for the experimental groups

Experimental Groups	BS means ± SD (MPa)	DC% means ± SD (%)
CH	1.96 ± 1.52 ^c	46.20 ± 2.52 ^{ab}
RH	2.55 ± 1.32 ^c	47.70 ± 2.14 ^a
SH	3.61 ± 1.72 ^b	44.67 ± 2.58 ^b
CL	2.73 ± 0.99 ^c	42.46 ± 0.62 ^c
RL	2.69 ± 1.48 ^c	46.86 ± 2.18 ^a
SL	5.20 ± 1.88 ^a	42.10 ± 1.99 ^c

Different superscript letters in columns indicate statistically significant differences

Table No. 3: BS and DC% means ± standard deviations for root regions

Root Region	BS ± SD (MPa)	DC% ± SD (%)
Cervical	3.99 ± 1.84 ^a	46.27 ± 3.00 ^a
Middle	2.54 ± 1.54 ^b	44.96 ± 2.81 ^b
Apical	2.84 ± 1.75 ^b	43.76 ± 2.59 ^b

Different superscript letters in columns indicate statistically significant differences

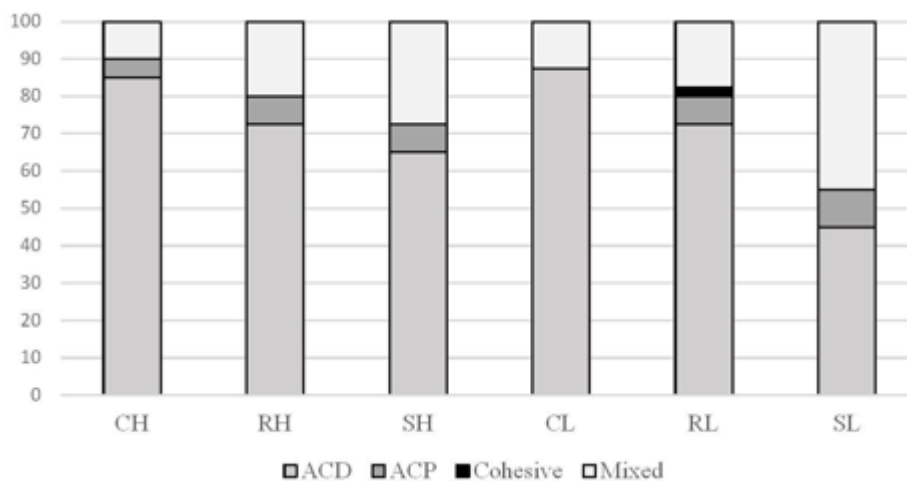


Figure No. 2: Failure pattern (%) for the experimental groups

Table 2 also presents the DC% means and standard deviations for the experimental groups. Two-way ANOVA demonstrated statistically significant differences ($p < 0.05$) for the two factors – “LCM” and “root region”, but not for their interaction. It can be noted that the -RH,

-RL and -CH exhibited the highest DC%, whereas -CI and -SL showed the lowest DC% without statistical difference from each other. Similar to BS results, DC% of the cervical third was statistically higher than those of the middle and apical thirds, which did not present statistically significant difference between them (Table 3). No significant correlation was found between DC% and BS ($r = -0.3 / p = 0.2214$).

The null hypothesis tested in this study was rejected, since the results indicated that BS of the GFPs to root canal dentin and the DC% of resin cement were significantly influenced by the studied LCM. The influence of the LCM on BS between GFP and dentin walls was investigated by means of push-out test. Although other methods have been described to evaluate BS, push-out has been described as more efficient and reliable (13-14). The variability of the present results was similar to that observed in other studies (13,15) and could be attributed to the unpredictability of the root canal morphology (15,16). Although the cervical diameter of root canal was standardized, subtle differences within the root canal anatomy could exist, once, regardless of the careful preparation and specimen selection, canals could have diverse conicity. Therefore, some variation on cement film thickness could not be predicted, and it has already been proven that cement thickness can influence the GFP retention (11).

Besides, endodontic treatment was not performed in this investigation, since materials like eugenol, gutta-percha and sealers remnants could interfere with the adhesive process and, consequently, harm the results (17-18). Also, in order to avoid inclusion of air bubbles, which could lead to a decrease on bond strength values and, consequently, predisposing post debonding, the resin cement was inserted into the root canal with a syringe (16).

The C-factor has been pointed out as an important concern in bonding procedures, as the higher the C-factor, the higher the stresses at the adhesive interface (6). Although the incremental technique is well established as an efficient method to minimize the effects of C-factor for resin composite restorations, it is not possible to apply this procedure while luting GFPs with resin cements. Besides, despite some studies have proposed soft-start LCM as an effective method to minimize polymerization stress on resin composite restorations (6), literature is still reduced when the focus is the GFP cementation (9).

An effective polymerization depends on a variety of factors, including the light-curing unit, its tip size and the employed wavelength, the radiant exposure, the amount of inorganic fillers

and photoinitiator present on the materials composition, and the LCM used to activate them (19). It is well known that resin-based materials should be submitted to a radiant exposure ranging from 21 to 24 J/cm² to ensure suitable monomer conversion and, consequently, appropriate mechanical properties (20). Although the experimental groups presented different LCM, all of them presented the final radiant exposure close to 24 J/cm². Concerning irradiance, when a high parameter is used, a much larger number of camphorquinone molecules will be excited, which would probably result in a greater extent of polymerization when compared to a photoactivation performed with low irradiance, since the last could lead to insufficient photon-density to initiate the free-radical reaction (21). However, the light emitted from a light-curing unit can be irradiated in different ways such as, continuous, ramp and step modes. The last two, the so-called soft-start LCM, start the photoactivation with low irradiances, allowing a prolonged pre-gel phase and, consequently, contributing to the decrease of the polymerization rate and the shrinkage stresses at the adhesive interface (5, 22). While the lower initial intensity stage favors the extended pre-gel phase, the posterior higher intensity step intends to complete the final radiant exposure (irradiance x time) to guarantee a suitable material polymerization (6). This fact could explain the highest BS found for the soft-start step groups (SI and SH), which combined a low irradiance (100 mW/cm²) during the initial 5 s with a higher final irradiance (SL, 600 mW/cm² and SH, 1050 mW/cm²). It is noteworthy that BS of the SL group was significantly higher than that of SH, probably due to the extended time with low irradiance which led to lower stresses at the adhesive interface. On the other hand, comparing to SL, the higher final irradiance of the SH group could have increased the shrinkage stresses, thereby reducing BS to the root canal walls.

Despite being soft-start methods, the ramp groups, RH and RL, employed higher radiant exposures during the pre-gel phase of the resin cement polymerization (2.63 J/cm² and 1.5 J/cm², respectively) than the step groups SL and SH (0.5 J/cm²). This aspect could explain the lower BS presented by the ramp groups, which could be related to a higher stress generation at the bonding interfaces produced by them. As well as the ramp groups, the conventional LCM groups (-CH and -CL presented lower bond strength than the step groups and were not different from each other. In addition, since the conventional LCM presented the same irradiance throughout the cycle, the shorter pre-gel phase could increase the stress at the bonding interface, leading to lower BS values.

Concerning BS outcomes for the radicular region, despite some authors had found no significant differences among different regions of the root canal (11,23), in the present study the cervical third presented higher values than the middle and apical ones (Table 3), this behavior is in agreement with other studies, independently of the employed LCM (14). A reasonable explanation could be the best access to the cervical third, favoring acid conditioning and adhesive system application (24), as well as allowing direct light transmission to canal walls (25). Besides that, the large concentration of dentinal tubules with great diameter, the presence of tubular resin tags and lateral branching at the cervical third, which decrease towards the apical region, could have also contributed to these results (26).

In general, the failure pattern was predominantly adhesive between resin cement and root dentin, results that agree with other previously published (11,27). These findings suggest that hydrogen peroxide and silane application as post surface treatment are effective and that hybridization procedures within the root canal are difficult to perform and still need to be improved. Nevertheless, the SL group showed an equal distribution of ACD and mixed failures, which could be attributed to the lowest stress development during the light-curing procedure.

Differently from earlier studies that showed similar DC% for resin cements light-activated under various LCM with similar radiant exposures (8,9,28), the current outcomes presented significant differences on the DC% among the experimental groups. In the same way, previous investigations have reported that, even with similar radiant exposures, LCM such as pulse delay (29,30) and step soft start (30) may lead to lower DC% values. In the present study, the step groups (SL and -SH) also presented lower DC% values, while the ramp groups (-RH and RL) showed the highest (Table 2). Although both step and ramp LCM are soft-start's and it could be expected similar DC% values, those results could be explained by the large variations in the dentin optical properties, which can be caused by differences in the dental hard tissues such as, tubule diameter and orientation, age and physiological changes (31-32), characteristics that could not be standardized for teeth selection. With regard to the radicular regions, the higher DC% at the cervical third than at the middle and apical ones clearly shows the influence of the light transmission on this response.

CONCLUSION

Within the limitations of this study, it could be concluded that both step LCM were able to improve the retention of the glass-fiber post. Even though the ramp soft-start light-curing modes led to the highest DC%, they did not influence GFP retention.

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<p><i>Image</i> <i>Author -1</i></p>	<p>Laiza Tatiana Poskus, Associate professor Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>
<p><i>Image</i> <i>Author -2</i></p>	<p>Eduardo Moreira da Silva, Full professor Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>
<p><i>Image</i> <i>Author -3</i></p>	<p>Luísa Chateaubriand Pegado, Associate Researcher Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>
<p><i>Image</i> <i>Author -4</i></p>	<p>Viviane Hass, Associate professor Departamento de Dentística, Universidade Estadual de Ponta Grossa, Praça Santos Andrade, nº 01 - Centro, Ponta Grossa, PR, Brazil – CEP 84010-330.</p>
<p><i>Image</i> <i>Author -5</i></p>	<p>Glauco Botelho dos Santos, Associate professor Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>
<p><i>Image</i> <i>Author -6</i></p>	<p>Alice Gonçalves Penelas, Adjunct professor*, Corresponding Author Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>
<p><i>Image</i> <i>Author -7</i></p>	<p>José Guilherme Antunes Guimarães, Associate professor Analytical Laboratory of Restorative Biomaterials – LABiom-R, Universidade Federal Fluminense / School of Dentistry, Rua Mário Santos Braga, nº 30 - Campus Valonguinho, Centro, Niterói, RJ, Brazil - CEP 24020-140.</p>