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Effect of the C-factor and Dentin Preparation Method in the Bond Strength of a Mild Self-etch Adhesive

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Clinical Relevance

A course cut carbide bur should be avoided prior to a mild self-etch adhesive, because it adversely affected bond strength. In contrast, a fine cut carbide bur provided the best combination: high bond strength with low variability, which suggests more reliable bond strength performance.

SUMMARY

This study evaluated the effect of the C-factor and dentin preparation method (DPM) in the bond strength (BS) of a mild self-etch adhesive; the study also observed the SEM superficial aspects of the corresponding smear layer. For purposes of this study, 25 molars (n=5) were used in a bond strength test. The molars were divided into two parts (buccal and lingual): one part received a Class V cavity (C-factor=3) and the other received a flat surface (C-factor=0) with the same bur type (coarse diamond or carbide bur and fine diamond or carbide bur), both within the same dentin depth. Five teeth were prepared with wet 60-grit and 600-grit SiC papers. After restoration with Clearfil SE Bond, microtensile beans (0.8 mm²) were prepared and tested after 24 hours in a universal testing machine (0.5 mm/minute). An additional two teeth for each DPM were prepared for SEM evaluation of the smear layer superficial aspects. The BS values were submitted to one-way ANOVA, considering only the DPM (flat surfaces) and two-way ANOVA (C-Factor x DPM, considering only burs) with *p*=0.05. Although the DPM in the flat surfaces was not significant, the standard deviations of carbide bur-prepared specimens were markedly

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lower. The BS was significantly lower in cavities. The fine carbide bur presented the most favorable smear layer aspect. It was concluded that different dentin preparation methods could not prevent the adverse effect in bond strength of a high C-factor. A coarse cut carbide bur should be avoided prior to a mild self-etch adhesive, because it adversely affected bond strength. In contrast, a fine cut carbide bur provided the best combination: high bond strength with low variability, which suggests a more reliable bond strength performance.

INTRODUCTION

Aiming to simplify the adhesive technique into a single step, self-etch adhesives incorporate acidic monomers to simultaneously demineralize and infiltrate dentin, forming the hybrid layer. This strategy was claimed to avoid discrepancies between depth of the etched and infiltrated dentin. Moreover, operator dependency and post-operative sensitivity were reported to be minimized when using this adhesive approach,[1] which explains its popularity.

However, among other factors, the efficacy of such adhesives depends on the demineralizing potential of the self-etch primer that must penetrate beyond the smear layer and remain acidic enough to etch and hybridize the underlying intact dentin. According to their pH, self-etch adhesives can be classified as mild, intermediary and strong.[2-3] Although the three types form hybrid layers, they were thinner for the mild type and the partially dissolved smear layer was incorporated into the adhesive interface.[2] There was concern regarding this complex interfacial structure, because, despite the initial bond strength being similar,[4] the three-step etch&rinse adhesive was more resistant to fatigue than the two-step mild self-etch adhesive.[5]

Some studies demonstrated a decrease in bond strength when a self-etch adhesive was applied to a thick smear layer.[6-9] This adverse effect was not confirmed in another study.[10] This controversy might be related to the manner in which the smear layer was produced. *In vitro* studies usually adopt SiC papers, because it is a practical procedure to standardize the smear layer; however, this limits comparison with clinical conditions. In addition, dentin preparation with burs apparently produced a more dense smear layer than that formed by abrasive papers as, with burs, a notable reduction in dentin tubule openness was observed in SEM (scanning electron microscopy), resulting from the self-etch primer application.[11-12]

Apart from the smear layer, it is important to consider that resin composite suffers polymerization shrinkage as it cures, generating stress at the adhesive interface, which might cause it to rupture.[13] Stress relief is possible via flow at the early stages of resin composite polymerization.[14] But stress relief by flow is limited by the different constraint levels determined by the cavity configuration or C-factor (ratio between bonded and unbonded areas).[15]

There are numerous bond strength studies in the literature involving dentin preparation with 600-grit SiC paper and flat surfaces (most favorable C-factor).[6,10,16] These studies actually make a quality control of the existing adhesive systems available in the market; however, they unfortunately do not relate to more critical and demanding clinical conditions, such as high C-factor cavities.

The C-factor influence in bond strength was assessed most often using etch&rinse adhesives that require a separate phosphoric acid etching step prior to application of the primer or adhesive.[17-19] This issue was also evaluated using self-etch adhesives,[20] but the bur-type effect in the performance of mild self-etch adhesives was likely to produce bond deficiencies with a thick smear layer, which was not directly addressed. The use of carbide burs seems promising,[11,21] but, to date, it is not known whether it would be sufficient to avoid the adverse effects of a high C-factor condition.

The current study evaluated the effect of the C-factor and smear layer preparation (SiC paper or bur) in dentin bond strength of a mild self-etch adhesive. The null hypotheses tested were: 1) there is no influence of the C-factor or 2) there is no influence of the dentin preparation method in the bond strength of a mild self-etch adhesive. The superficial aspects of the different dentin preparation methods were also evaluated by SEM and a third null hypotheses was tested: 3) there is no difference in the superficial aspects of the smear layer produced by different dentin preparation methods.

METHODS AND MATERIALS

Teeth Preparation

In the current study, 25 third molars were used for the bond strength test and 12 third molars were used for SEM evaluation of the superficial aspects of the smear layer. All were stored in 0.5% chloramine solution until specimen preparation.[22] The molars were provided by the Human Teeth Bank of the University of São Paulo Dental School after the Ethics Committee approved the project (protocol number: 79/05).

Two factors were considered: the C-Factor (flat surface and cavity) and the Dentin Preparation Method (coarse diamond bur–**CD**, fine diamond bur–**FD**, coarse carbide bur–**CC** and fine carbide bur–**FC** and 60-grit + 600-grit SiC papers for flat surfaces only). For each experimental condition, five samples were prepared.

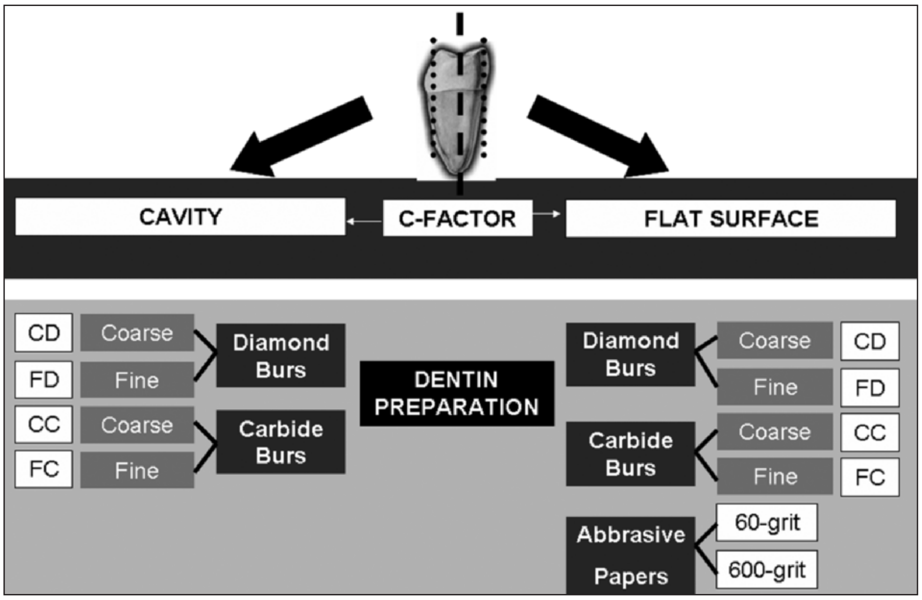

Figure 1. *Experimental design.*

As indicated in Figure 1, each tooth was sectioned into two parts in the mesio-distal direction (Labcut 1010, Extec Corp, Enfield, CT, USA). Likewise, the buccal and lingual faces were further sectioned with a minimum width of the extracted sections only to flatten the surfaces that were considered for dentin depth reference, as all the cavities and flat surfaces were prepared with a 1.5 mm of depth from this initial flattened surface.

Using a device for standardized cavity preparations, each tooth had its buccal or lingual portion randomly assigned to a Class V preparation 3 mm in length, 3 mm wide and 1.5 mm deep (C-Factor = 3) as measured with a digital caliper or a flat surface prepared at the same dentin depth, extending the cavity dimensions until the cavity walls were eliminated and only the flat surface remained. Therefore, in each tooth, one of the parts had a cavity prepared and the other part had a flat surface prepared with the same bur. Additionally, five teeth were similarly prepared to the same dentin depth, then, one of the parts received additional polishing for one minute with 60-grit SiC paper, while the other part was polished with 600-grit for thick and thin smear layer production.[2]

Bond Strength Test

As recommended by the manufacturer, the mild self-etch adhesive Clearfil SE Bond (Table 1) was applied by the same operator under controlled temperature and humidity conditions (24°C and 75% of relative humidity) using a light curing unit (Q-HL75-Dentsply Caulk, Milford, DE, USA) with an intensity of 600 mW/cm^2 previously measured in a radiometer.

After adhesive application, a resin composite buildup was made with Z250 (3M ESPE, St Paul, MN, USA) in three portions of approximately 1 mm, which were light cured for 30 seconds each in flat surface specimens. The cavities were restored in the bulk-filling technique using a Centrix syringe and light cured for 90 seconds.

After 24 hours of water storage at 37°C, the specimens were sectioned in the mesio-distal and occluso-gingival directions using a diamond disc (Labcut 1010, Extec Corp, Enfield, CT, USA) perpendicular to the adhesive interface of the flat surface specimens or the axial wall of the cavity specimens for microtensile beams, obtaining a cross-section area of approximately 0.8 mm^2.

The beans were individually fixed to a device[23] for microtensile testing, which was performed in a universal testing machine (Kratos Dinamometros, São Paulo, SP, Brazil) at a speed of 0.5 mm/minute. The rupture load and cross-section dimensions were measured for bond strength calculation (MPa) for each beam. The data was analyzed by ANOVA (p=0.05).

Table 1: *Microtensile Bond Strength (standard deviations) in MPa of the Two-way ANOVAs Performed Separately for Diamond, Carbide Burs and Abrasive Paper*

Dentin Preparation/C-Factor	Diamond	Carbide	Abrasive Paper
Fine/Flat	34.76(8.74)a	32.62(2.23)b	
Fine/Cavity	28.82(4.34)a	27.70(2.08)c	
Coarse/Flat	31.86(5.53)a	25.96(1.70)c	
Coarse/Cavity	28.9(6.01)a	20.21(2.38)d	
600-grit			33.26(9.59)E
60-grit			25.47(8.22)F

**Comparisons are valid only within the same statistical analysis (columns). The symbols indicate no statistically significant differences (p>0.05).*

Specimens Preparation for SEM

Twelve teeth were divided into two parts and the flat surfaces were prepared as previously described (n=4). The specimens were fixed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C, rinsed with 20 ml of 0.2 M sodium cacodylate buffer at pH 7.4 for one hour with three changes, followed by rinsing with distilled water (one minute) and dehydration in ascending grades of ethanol: 25% (20 minutes), 50% (20 minutes), 75% (20 minutes), 95% (30 minutes) and 100% (60 minutes). The specimens were then immersed in hexamethyldisilazane (HMDS) for 10 minutes[24] and gold-sputtered to be observed at SEM (Philips Electronics NV, Balzers, Liechtenstein).

For the smear layer surface evaluation, three regions, equally spaced for each specimen, were observed under original magnifications of 1000x, 3000x and 5000x and the following operation parameters: 20 kV, spot: 3.0 and WD 10.6, using secondary electrons. The representative micrographs were selected for each preparation method for posterior descriptive analysis.

RESULTS

Bond Strength

Preliminary data analysis demonstrated considerable differences between the standard deviation of diamond and carbide bur groups and the abrasive papers group (S^2 diamond bur/S^2 carbide bur = 9.067 - $p<0.001$; S^2 abrasive paper/S^2 diamond bur = 1.971 - $p>0.05$; S^2 abrasive paper/S^2 carbide bur = 17.8672 - $p<0.001$). These differences can be observed in Table 1. This fact led to a separate analysis of variance. As the same tooth provided the substrate for flat surfaces and Class V cavities for the burs group and the 60- and 600-grit SiC paper for the abrasive paper group, the burs data were subjected to a two-way ANOVA with the factor dentin preparation considered independent and the C-factor tied. The data with the abrasive papers were subjected to one-way ANOVA, considering the factor dentin preparation. As each tooth provided data for both the 60- and 600-grit SiC paper, the variability between repetitions was isolated (between teeth). The significance level was set at 5%. As there were no premature failures, no specific analysis was necessary.

Two-way ANOVA:

The analysis of variance with the diamond bur data did not demonstrate significant differences among the dentin preparation types. Table 1 shows that the coarse and fine diamond burs presented similar results. On the other hand, the C-Factor was significant and bond strength was lower in cavities (26.41 MPa) than in flat surfaces (31.30 MPa).

The analysis of variance with carbide bur data presented a significant effect of both main factors. This can be easily identified in Table 1. The fine cut bur produced higher bond strength values than the coarse cut bur independent of the C-factor condition.

The analysis of variance with abrasive papers showed that 600-grit SiC paper produced significantly higher bond strength than 60-grit SiC paper.

One-way ANOVA

Table 2 shows that there was no difference in bond strength between fine cut diamond burs and fine cut carbide burs for both C-factor conditions. On the other hand, for coarse cut burs, the carbide bur produced significantly lower values. However, carbide burs also presented markedly low standard deviations (Table 1).

Bond strength with 600-grit SiC paper was similar to fine cut burs. However, abrasive papers cannot be used in cavity preparations and their standard deviations were high.

These results (Tables 1 and 2) demonstrated that a favorable selection for dentin preparation would be the fine cut carbide bur for both *in vitro* studies and clinical trials, as high bond strength with low variability was achieved with carbide burs, which indicates better results reliability.

Surface Aspects

Considering the surfaces produced by coarse (A, C and E) and fine (B, D and F) preparation methods in Figure 2, there was a marked contrast between the 60- and 600-grit SiC paper. The fine abrasive paper produced a uniform surface, practically without thick deposits of the smear layer and with apparently more superficial, non-oriented scratches.

In contrast, the surface produced by coarse abrasive paper (Figure 2E) was the most uneven, apparently having the roughest

Table 2: *Means Comparison*

Dentin Preparation/ C-factor	Bond Strength Means (MPa)				
	60-grit	Diamond Bur	600-grit	Carbide Bur	60-grit
Fine/Flat		34.76 A		32.62 A	
Fine/Cavity		28.82 B		27.20 B	
	25.47		33.26		25.47
Coarse/Flat		31.86 C		25.96 D	
Coarse/Cavity		28.90 E		20.21 **F**	

Between diamond and carbide burs, the same letters in the same line indicate similar bond strength values (p>0.05); between abrasive papers and burs, the arrows indicate no significant difference (p>0.05).

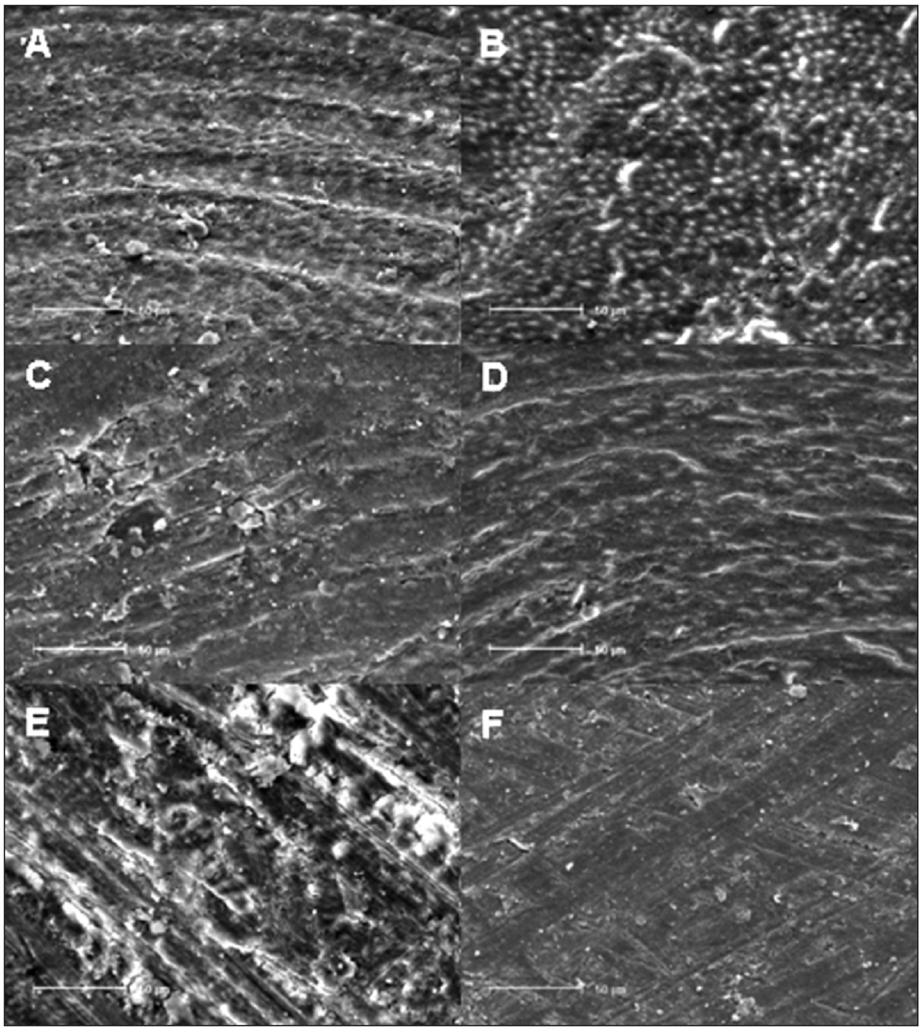

Figure 2. *Surface aspects of the smear layer produced by different dentin preparation methods.* ***A****: coarse carbide bur;* ***B****: fine carbide bur;* ***C****: coarse diamond bur;* ***D****: fine diamond bur;* ***E****: 60-grit SiC abrasive paper and* ***F****: 600-grit SiC abrasive paper (original magnification: 1000x).*

aspect and covered by the smear layer with different densities, as suggested by the areas of thick deposits of granular particles irregularly distributed, which seemed to be attached to the underlying dentin at varied degrees, probably depending on the pressure suffered during the surface preparation.

Comparing surfaces produced by carbide burs (A and B), the surfaces appeared to be lightly covered by the smear layer, especially for the fine carbide bur (2B) that presented evidence of the underlying dentinal tubules, as denoted by the regularly distributed electron-dense spots compatible to dentin tubules in both size and distribution, which was clearly observed only for this experimental condition. The coarse carbide bur presented surfaces with clearly oriented scratches, which were regularly spaced, forming an undulating surface not observed for the fine carbide bur surfaces.

Considering the coarse preparation methods, a tendency to form thick deposits of the smear layer was observed, (A, C and E), which decreased the apparent differences between surfaces prepared by the coarse diamond bur and 60-grit SiC paper. Nevertheless, for the fine preparation methods (B, D and F) that appeared to be more favorable for a mild self-etch adhesive, because of the uniformity of their resulting surfaces, a crescent denseness of the smear layer can be noted from the fine carbide bur to the 600-grit SiC paper with an intermediary aspect for the diamond bur, as the dentin tubules are progressively less apparent in this order.

DISCUSSION

When polymerization shrinkage stress is higher than bond strength, there is a rupture of the interface. The likelihood of this occurring depends on the cavity C-Factor (ratio between the bonded and unbonded surface area), for example, below 1 in a flat surface, the interface would be preserved and, for values above 2, which is severe in Class I or Class V cavities, failure at the adhesive interface would be more likely to occur.[15] This occurs, because the higher the confinement level, the less shrinkage stress there is that can be relieved by flow of the free surfaces.

Accordingly, in the current study, bond strength was significantly higher in flat surfaces (C-Factor=0) than in cavities (C-Factor=3). The same observation was made previously with a C-factor of 5 using Clearfil Linerbond 2V, but 600-grit SiC prepared flat dentin surfaces were compared to diamond bur prepared cavities, which could have influenced the results.[25] Therefore, the first null hypothesis was rejected and, despite the excellent mechanical properties and high bond strength observed in several independent studies for Clearfil SE Bond,[20,26-28] this mild self-etch adhesive was unfavorably affected by the high C-Factor tested.

Independent of the adhesive technique (etch&rinse or self-etch), bond strength to dentin is directly related to hybrid layer formation.[29] Regarding mild self-etch adhesives, their interface presents two distinct parts: one part incorporates the smear layer and the other part corresponds to the true hybrid layer formed by the demineralization and resin monomer infiltration of intact subjacent dentin.[2-3] To promote hybridization, acidic resin monomers have to dissolve the smear layer and remain acidic enough to etch the intact subjacent dentin.[2-3,30] Therefore, it is reasonable to suppose that mild self-etch adhesives could present a decrease in bond strength applied to thick smear layers.[7-9,12,31]

Smear layer characteristics depend on the way they were produced: a significant correlation (R=0.802) between the average grain size of the abrasive and the smear layer thickness (0.9 to 2.6 µm) was observed. Small grain sizes lead to a thin smear layer.[32] The roughness also varied between 267.7 and 821.2 nm for abrasive papers (600-grit and 240-grit) and between 425.9 and >1000 nm for carbide burs and coarse diamond burs, respectively.

These aspects might be considered in accordance with the findings of the current study, which clearly presented more uniform surfaces for the fine-grit preparation methods than for the coarse ones that tended to form irregularly distributed thick deposits on their surfaces. This allows for rejection of the third null hypothesis.

It is important to consider that the more heat generation and shear frictional forces, the more the smear layer be will be attached to the underlying dentin and the more acid resistant it will be.[33-34] Thus, it was observed that, for similar smear layer thicknesses, the layer produced by rotary instruments was more resistant to removal by acid solution than the layer formed by abrasive papers, which was attributed to differences in smear layer denseness.[12] Apparently, this effect could be avoided by more acidic self-etch adhesives that, among the different acidity self-etch adhesives, produced the most similar conditioning pattern, resin tag formation and hybrid layer to etch&rinse adhesives;[4,35] though lower bond strength was observed compared to mild self-etch adhesives.[4,36]

In the current study, an assessment was made as to whether the smear layer production method could affect the bond strength of a mild self-etch adhesive. No statistically significant differences were observed in the bond strength of diamond and carbide burs compared to abrasive papers, which supports the widespread use of abrasive papers in *in vitro* studies.[7,28] Therefore, the second null hypothesis was not rejected.

Supporting these findings, no difference was observed previously between different surface preparation methods using the same adhesive.[37] However, in one study, fine diamond bur dentin preparations led to significantly higher bond strength than regular-grit diamond burs for Clearfil SE Bond[38] and, in another study, the carbide bur led to the lowest bond strength compared to 600-grit SiC paper or a diamond bur.[28] This might be explained by differences in turbine speed, pressure applied and use of a device that firmly holds a dental handpiece during preparation, as applied in the current study or in manual preparation.

It was intriguing to observe that the standard deviations were much smaller when carbide burs were used, which could mean that bond strength was more uniform for this condition. In fact, considering the SEM micrographs, the fine carbide bur (Figure 2B) produced surfaces that were apparently lightly covered by the smear layer, with evidence of the underlying dentinal tubules. This suggests that the smear layer would be less attached to the underlying dentin or less compact than the other methods, which might seem to be a more favorable dentin surface compared to using a mild self-etch adhesive. However, this needs further investigation.

Nevertheless, good immediate performance does not guarantee adhesive effectiveness in the long-term, and some adhesive systems present a significant decrease in bond strength over time.[25,39] On the other hand, it is important to achieve a trustworthy initial bond, so that the aging effects can be evaluated *in vitro*;[40] as in *in vivo* studies, it is very difficult to establish the causes of failure, because of the interaction of the multiple factors involved in aging of the restoration.[25,39]

CONCLUSIONS

Under the experimental conditions tested, it was concluded that different dentin preparation methods could not prevent the adverse effect in bond strength of a high C-factor. Moreover, bond strength was not affected by the type of cut (fine or course) when diamond burs were used, contrary to what happened to carbide burs that were adversely affected by the coarse type of cut. All things considered, the most favorable and reliable selection for dentin preparation when a mild self-etch adhesive is used is fine cut carbide burs that achieve high bond strength with low variability, which is compatible with its uniform superficial aspect.

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