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Factors Affecting Shear Bond Strength of Composite Resin to Fluorosed Human Enamel

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Clinical Relevance

Age affects shear bond strength of composite resin to fluorosed teeth; when fluorosis is severe, bond failure is most likely to be cohesive in enamel.

SUMMARY

The aim of this work was to determine the effects of age, severity of fluorosis, and etching time on the shear bond strength of direct composite resin to human enamel. A total of 117 teeth, freshly extracted from patients in areas of Saudi Arabia endemic for dental fluorosis, were classified according to age (<40 years and 40+ years) and severity of fluorosis, using the Thylstrup and Fejerskov index, TFI: TFI = 0, TFI = 1-3, and TFI = 4-6. Cylindrical composite resin specimens 5 mm in diameter and 3 mm high were bonded to the flattened midlabial enamel surfaces etched for 60 or 120 seconds and shear bond strength measured, using the Instron Universal Testing Machine at a crosshead speed of 0.5 mm/ minute.

Shear bond strength of the resin varied between 11.2±3.6 and 21.6±4.1 MPa. Three-way analysis of variance and Sheffé's multiple range test showed that the severity of fluorosis had no statistically significant effect on shear bond strength ($p > 0.05$). However, the bond was significantly stronger in teeth from patients <40 years old than from those 40+ years old. Furthermore, at age <40 years, shear bond strength was significantly higher in teeth etched for 120 seconds than those etched for 60 seconds ($p<0.05$), but this was not the case in teeth from the older patients. In teeth with TFI = 1-3, the mode of bond failure was predominantly mixed, but at TFI = 4-6, the bond failure was mostly cohesive in enamel at all ages and etching times. It is, therefore, concluded that both age and etching time affect the shear bond strength of composite resin to fluorosed human enamel.

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INTRODUCTION

Enamel fluorosis is characterized by surface hypermineralization and porosity of the subsurface layer (Thylstrup & Fejerskov, 1978). The hypermineralized surface enamel is difficult to acid etch (Opinya & Pameijer, 1986), while the porous subsurface layer may attract extrinsic stains posteruptively, resulting in tooth discoloration (Fejerskov, Manji & Baelum, 1990). In severe cases of dental fluorosis, the subsurface porosity is so extensive that occlusal trauma may give rise to the detachment and pitting of surface enamel (Thylsrup & Fejerskov, 1978). The discolored and pitted enamel may be esthetically objectionable, sometimes resulting in psychological ill health.

Dental fluorosis is endemic in several parts of the world (Dean, Arnold & Evolve, 1942; Leverett, 1986; Warnakulasuriya & others, 1992; Akpata, Fakiha & Khan, 1997), and the use of fluoride in preventive dentistry over the past few decades has resulted in increased prevalence of the condition in many countries (Leverett, 1986). One of the treatment modalities for moderately or severely fluorosed teeth is the provision of the composite resin veneer (Akpata, 1997). Bonding of the veneer involves etching the acid-resistant fluorosed enamel, and this may necessitate prolonging the etching time.

It was hypothesized that the bond strength of composite resin to fluorosed enamel would be influenced by the severity of fluorosis, etching time, and age of the patient. The purpose of this study, therefore, was to investigate the effects of age, severity of fluorosis, and etching time on the shear bond strength of composite resin to human enamel.

METHODS AND MATERIALS

Diagnosis of Dental Fluorosis

The diagnosis of fluorosis was practiced on 46 clinical slides of fluorosed teeth classified according to the modified Thylstrup and Fejerskov index, TFI (Fejerskov, Manji & Baelum, 1988) by one of the investigators (NA). The diagnosis was repeated after two weeks; the test of intraexaminer reproducibility gave a Cohen's kappa statistic (Cohen, 1960) of 0.974. In addition, both investigators carried out the diagnoses independently, and a test of interexaminer reproducibility resulted in a Cohen's kappa statistic (Cohen, 1960) of 0.972.

Collection and Grouping of Experimental Teeth

A total of 117 fluorosed and nonfluorosed anterior and posterior teeth (12 incisors, 69 premolars, and 36 molars) were used in this study. These were obtained from Hail and Al-Ras, two areas of Saudi Arabia endemic for dental fluorosis. The teeth had no obvious damage as a result of extraction, caries, or abrasion. The extracted teeth were cleaned with an ultrasonic scaler, polished with a brush and nonfluoride flour of pumice, and then stored in distilled water containing 0.2% thymol as a disinfectant, in a refrigerator. The teeth were kept in two separate jars according to the age of the patient from whom they were extracted: < 40 years and 40+ years of age.

Prior to use, the experimental teeth in each age group were classified according to the severity of fluorosis, using the TFI: TFI = 0, TFI = 1-3, and TFI = 4-6. The distribution of teeth in each group is shown in Figure 1.

Preparation of Enamel Surface

To standardize the enamel reduction, depth orientation grooves 0.5 mm deep were prepared on the midlabial surfaces of each of the experimental teeth. The teeth were then mounted in autopolymerizing acrylic resin in standardized PVC molds 20 mm high, with an internal diameter of 26 mm. The grooved midlabial surfaces of the experimental teeth were then flattened with 240- and 600-grit silicon carbide paper.

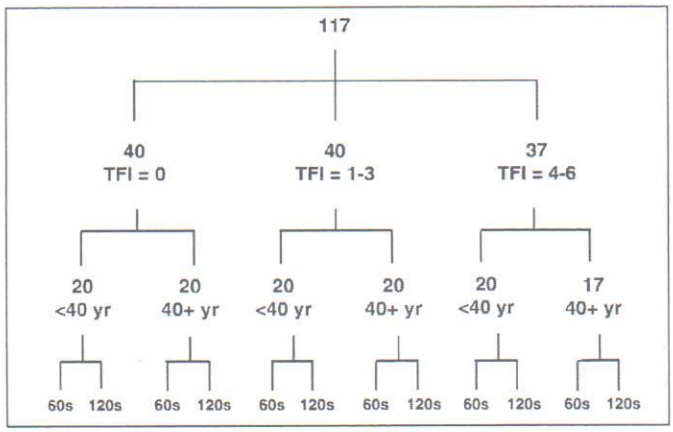

Figure 1. *Grouping of teeth.*

The flattened midlabial surface of each tooth specimen was polished with a rubber cup and nonfluoride flour of pumice for 20 seconds, washed with distilled water for 20 seconds, and dried with an air syringe for an additional 20 seconds. Air from the syringe was filtered with a single filter (AF100; Yoshida Works, Osaka, Japan).

Etching

Coltène Etchant Gel (Brilliant Esthetic System, Batch No EJ435; Coltène/Whaledent Inc, Mahwah, NJ 07430) containing 35% phosphoric acid was applied with a brush to cover the flattened enamel surface of each specimen. Half of the specimens in each group were etched with the phosphoric acid for 60 seconds and the other half for 120 seconds. However, in the group of teeth with TFI = 4-6 from patients aged 40+ years, nine teeth were etched for 60 seconds and the remaining eight teeth for 120 seconds (Figure 1). The etched surfaces were then washed with a gentle stream of distilled water for 20 seconds and dried with filtered air from the syringe for 20 seconds.

Application of Bonding Resin and Composite Resin

Coltène Margin Bond (Brilliant Esthetic System, Batch No EJ435), an enamel bonding agent, was applied with a brush to the etched enamel surface of each specimen. The bonding agent was lightly air sprayed to produce a thin film and cured for 20 seconds with a light-curing unit (Coltolux 4; Coltène/ Whaledent). The power output of the curing unit was checked periodically to ensure that it was not less than 300 mW/cm^2 (Barghi, Berry & Hatton, 1994).

Coltène Brilliant Enamel hybrid composite resin, shade A2 (Brilliant Esthetic System, Batch No RJ435) was applied to the etched midlabial surface of each specimen in a cylindrical split Teflon mold 3 mm high, with an internal diameter of 5 mm. The mold was seated firmly at the center of the flattened tooth surface. After curing the composite resin for 60 seconds, the mold was removed. The specimen was then stored at 37°C in distilled water for 24 hours before shear bond testing.

Shear Bond Strength Measurement

Bond strength between the composite resin and enamel was measured in shear mode with an Instron Universal Testing Machine (Model 1197; Instron Corp, Buckinghamshire, England). The specimen was mounted in a fixture and a knife-edge loading device applied to the composite resin specimen as close to its junction with enamel as possible at a crosshead speed of 0.5 mm per minute. Fracture loads were recorded in newtons on a strip chart. The recorded loads were converted to MPa by dividing the loads by the cross-sectional area of the composite resin cylinder.

Mode of Bond Failure

The mode of failure of the bond between the composite resin and enamel was determined using the light microscope at X10 magnification and classified into adhesive, cohesive, and mixed types of failure (Nakajima & others, 1995). Adhesive failure was recorded when there were no signs of enamel fracture or remnants of resin on the tooth, cohesive fracture when there was complete fracture of enamel or resin, and mixed failure when the samples showed both adhesive and cohesive failures. In addition, 40 representative specimens from the 12 sample subgroups that included those with adhesive, cohesive, and mixed modes of failure were selected for scanning electron microscopic study. The specimens were thoroughly washed with distilled water, dried, and secured onto metal stubs with colloidal carbon adhesive. They were then sputter coated with gold and examined at x1000-2000 magnification under the scanning electron microscope (JEOL SMT 330; JEOL Ltd, Tokyo, Japan) at 15 kv. Photomicrographs were taken using Kodak Verichrome Pan 120mm black-and-white negative films, VP 120.

Statistical Analysis

Statistical significance among shear bond strength, TFI, age, and etching time was determined at a 5% probability level using the three-way analysis of variance (ANOVA) with interaction, as well as Scheffé's multiple range test.

Table 1: *Mean shear bond strength in MPa (± standard deviation) of composite resin to enamel with varying severity of fluorosis after etching for 60 or 120 seconds (shear bond strength values with different letters in superscript indicate statistically significant differences).*

	TFI	Etching 60 s.	Time 120 s.
Age <40 yr.	0	18.2 (7.1)^{a}	20.8 (2.9)^{c}
	1-3	19.3 (5.3)^{a}	21.6 (3.0)^{c}
	4-6	18.8 (5.2)^{a}	21.6 (4.1)^{c}
Age 40+ yr.	0	10.7 (2.1)^{b}	12.4 (3.0)^{b}
	1-3	19.0 (6.1)^{d}	12.9 (6.1)^{b}
	4-6	14.2 (7.3)^{bd}	11.2 (3.6)^{b}

RESULTS

Teeth

Each sample subgroup of 10 teeth (Figure 1) comprised one incisor, six premolars, and three molars, except the two groups with TFI = 4-6 from patients aged 40+ years that consisted of five and four premolars, respectively.

Shear Bond Strength

The three-way ANOVA and Sheffé's multiple range test showed that the severity of fluorosis had no significant effect on shear bond strength of composite resin to enamel ($p>0.05$). The only exception were the teeth with TFI = 1-3, which had significantly stronger bond than those with TFI = 4-6 at age 40+ years ($p<0.05$). However, the bond strength was significantly higher in teeth extracted from patients aged <40 years than in those aged 40+ years ($p<0.05$) (Table 1).

At age <40 years, shear bond strength was significantly higher in teeth etched for 120 seconds than in those etched for 60 seconds ($p<0.05$), but this was not the case in teeth extracted from patients aged 40+ years ($p>0.05$) (Table 1).

The coefficient of variation of the shear bond strength measurement ranged from 14-51%, and the power of the test was approximately 70%.

Mode of Bond Failure

In nonfluorosed teeth etched for 60 seconds, adhesive mode of failure was most predominant in teeth from patients aged <40 years. However, when etching time increased from 60 to 120 seconds, more cases of mixed failure became evident (Tables 2-3). In teeth with TFI = 1-3, the mode of failure was predominantly mixed at all ages and etching times. At TFI = 4-6, the mode of failure was mostly cohesive in enamel when etched for 60 or 120 seconds at all ages. There were no cases of cohesive failure in composite resin (Tables 2-3).

Table 2: *Frequency of teeth with varying severity of fluorosis at age >40 years according to the mode of failure of bonded composite resin.*

Mode of Failure	TF=0		TFI=1-3		TFI=4-6		Total
	60 sec.	120 sec.	60 sec.	120 sec.	60 sec.	120 sec.	
Cohesive in enamel	1	1	4	2	6	4	18
Cohesive in composite	0	0	0	0	0	0	0
Adhesive	5	2	2	0	4	0	13
Mixed	4	7	4	8	0	6	29
Total	10	10	10	10	10	10	60

Table 3: *Frequency of teeth with varying severity of fluorosis at age 40+ years according to the mode of failure of bonded composite resin.*

Mode of Failure	TF=0		TFI=1-3		TFI=4-6		Total
	60 sec.	120 sec.	60 sec.	120 sec.	60 sec.	120 sec.	
Cohesive in enamel	3	0	0	0	5	6	14
Cohesive in composite	0	0	0	0	0	0	0
Adhesive	3	2	2	2	0	0	9
Mixed	4	8	8	8	4	2	34
Total	10	10	10	10	9	8	57

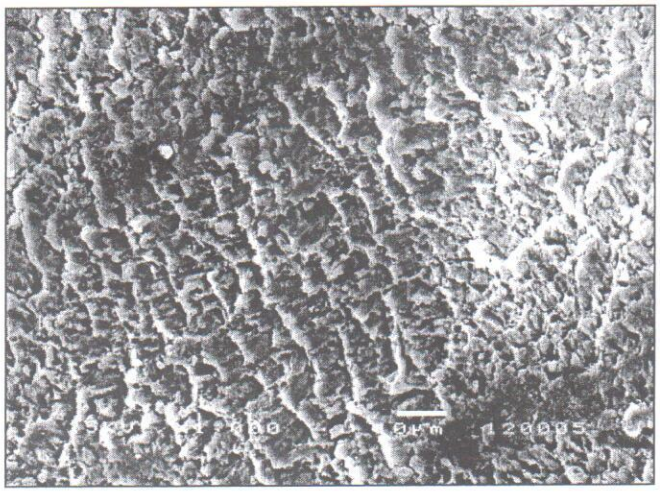

Figure 2. *Scanning electron micrograph showing enamel surface after adhesive failure on a tooth with TFI=0 at age <40 years and etching time of 60 seconds (original magnification x1000).*

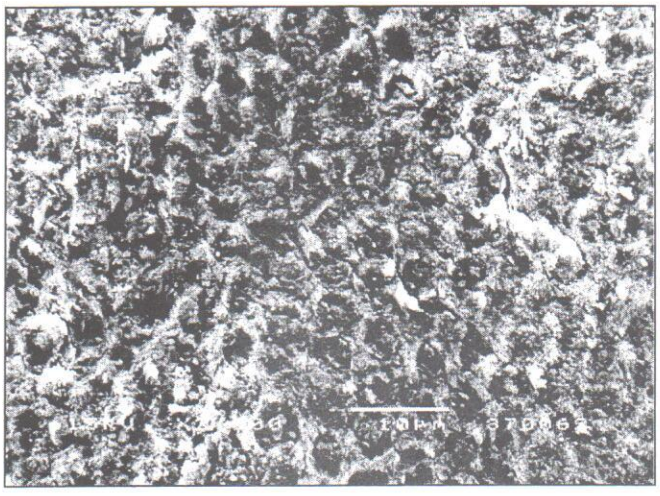

Figure 3. *Scanning electron micrograph showing enamel surface after adhesive failure on a tooth with TFI=1-3 at age 40+ years and etching time of 120 seconds (original magnification x2000).*

In cases with adhesive mode of failure, SEM examination revealed typical etching patterns (Figures 2 and 3), especially in the nonfluorosed teeth. In teeth with TFI = 4-6, exposed dentinal tubules could be seen in some cases with cohesive or mixed failure (Figure 4), suggesting detachment of enamel at the dentinoenamel junction. The small sample size did not permit statistical analysis for the significance of the differences between the different modes of failure.

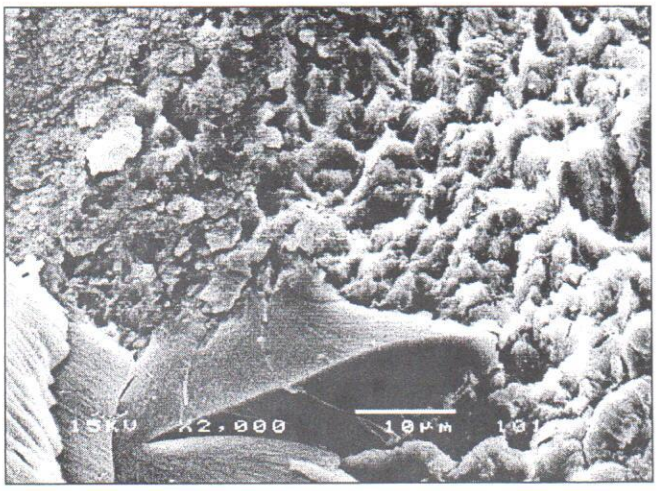

Figure 4. *Scanning electron micrograph showing enamel surface after mixed failure on a tooth with TFI=4-6 at age 40+ years and etching time of 120 seconds. Some open ends of dentinal tubules are visible on the top left corner (original magnification x2000).*

DISCUSSION

The classification of fluorosis in this study was according to the modified Thylstrup and Fejerskov index (Fejerskov & others, 1988), which is based on the clinical changes in fluorosed teeth. The main advantage of this classification is that it is consistent with the histopathological changes in fluorosed enamel. Moreover, the index is highly reproducible, as evidenced by the high kappa stastistics (Cohen, 1960) of 0.972-0.974 obtained from inter- and intraexaminer reproducibility tests in the present study.

Teeth with TF I = 1-3 were grouped together because Al-Sugair and Akpata (1999) have shown that the pattern and depth of etch were similar within this group. Furthermore, Opinya and Pameijer (1986) reported that tensile bond strength of resin composite to teeth with TFI = 1-3 was similar.

As the number of teeth available for the study was limited, we decided to divide the samples into only two age groups: <40 years and 40+ years. This decision was based on the fact that most teeth tend to be lost because of periodontal disease after age 40 (Reich & Hiller, 1993; Murray, Locker & Kay, 1996). Thus the chances of finding caries-free teeth for the study was increased. The effect that different age groups might have on the results may be elucidated by future research.

Knoll, Gwinnett, and Wolff (1986) reported statistically significant differences in the shear strength of orthodontic brackets bonded to anterior and posterior teeth. They attributed the lower values in posterior teeth to adaptation and nonuniform resin thickness, rather than differences in etching pattern. To minimize the effect of tooth types in the present study, each sample subgroup comprised approximately equal proportions of incisors, premolars, and molars. Furthermore, bonding was to the flattened midlabial surfaces of the teeth.

As far as we know, there are no reports of previous studies on the shear bond strength of composite resin to fluorosed enamel. Consequently, the predetermination of the sample size was not easy. Each of our sample subgroups comprised approximately 10 teeth, as in several previous studies utilizing nonfluorosed teeth (Gilpatrick, Ross & Simonsen, 1991; Hadavi & others, 1993), and each of the fluorosis sample groups consisted of approximately 40 teeth (Figure 1). Moreover, the coefficient of variation obtained (14-51%) in the present study is within the range reported in the literature (Barkmeier, Shaffer & Gwinnett, 1986; Opinya & Pameijer, 1986; Guba, Cochran & Swartz, 1993; Holtan & others, 1995). As the power of the test was approximately 70%, the sample size, in an investigation of this nature, may be regarded as reasonably adequate. The use of the same tooth types in future studies may reduce the variability in the shear bond strength and also increase the power of the test.

Opinya and Pameijer (1986), in their study of tensile bond strength of composite resin to fluorosed teeth, etched enamel for 120 seconds. However, Al-Sugair and Akpata (1999), in their investigation of the etching pattern of fluorosed teeth, recommended an etching time of at least 30 seconds for fluorosed teeth with TFI = 4 and at least 90 seconds for more severely fluorosed teeth with pitting (TFI = 5+). Therefore in the present study, etching times of 60 (mean of 30 and 90) and 120 seconds were used.

Richards, Fejerskov, and Baelum (1989) showed that the highest concentration of fluoride is in the outer 200 μm of fluorosed enamel. This outer hypermineralized layer is highly resistant to acid etching (Al-Sugair & Akpata, 1999). Opinya and Pameijer (1986) observed that grinding away this outer hypermineralized layer before etching resulted in higher tensile bond strength. In the present study, the outer 0.5 mm of enamel was ground away to flatten the enamel surface for shear bond strength measurements. This is consistent with clinical practice when the outer 0.5 mm of labial enamel is removed during tooth preparation for composite resin or porcelain laminate veneer. On the other hand, bond strength values might have been lower if the composite resin were bonded to the hypermineralized enamel surfaces of the fluorosed teeth, without grinding. There is a need to develop a technique for measuring shear bond strength of composite resin to fluorosed enamel without grinding to flatten the hypermineralized surface layer. The result from such an investigation would be useful to clinicians who place laminate veneers without tooth preparation.

The shear bond strength obtained in this study would be expected to be similar to initial bond of direct composite resin veneer. This is because bond strength (in MPa) is independent of the thickness of the composite resin used for bond strength testing, provided the loading plane is as near the interface as possible. Besides, there appears to be no method available for the direct measurement of purely shear bond strength of composite resin veneer to enamel. As the restoration ages, however, the bond may deteriorate. The procedures most often used to simulate aging are long-term water storage at a constant temperature or thermocycling. The validity of thermocycling has been questioned by many investigators (Crim & Mattingly, 1981; Brännström, 1984; Eackle, 1986), who were of the opinion that the experimental procedure may change the properties of the restorative material and hardly simulates clinical situations. In the present study, prior to bond testing, we stored the specimens in water at 37° C for 24 hours.

The studies on tensile bond strength of composite resin to fluorosed teeth reported by Opinya and Pameijer (1986) as well as Ng'ang'a and others (1992) were aimed at determining dislodging forces on orthodontic brackets, and these are predominantly tensile. In the present study, we measured shear bond strength because it more closely approximates to the dislodging forces acting on composite resin veneers.

The shear bond strength of 18±7.1 MPa obtained for teeth with TFI = 0 (ie, nonfluorosed teeth) etched for 60 seconds in this study is rather similar to about 20 MPa reported for nonfluorosed teeth by other investigators (Barkmeier & others, 1986; Gilpatrick & others, 1991; Gwinnett, 1992). In fact, for teeth with TFI = 4-6 in the present study, shear bond strength was as high as 21.6±4.1 MPa at age <40 years (Table 1). In general, however, severity of fluorosis per se did not have a statistically significant effect on shear bond strength (Table 1). This is in agreement with the report by Ng'ang'a and others (1992), who observed no significant difference between the mean tensile bond strengths in teeth with TFI = 0, 3, or 4.

Posteruptive trauma weakens the surface enamel in severely fluorosed teeth (TFI = 5+), resulting in its detachment and, therefore, surface pitting (Thylstrup & Fejerskov, 1978). Although there is no pitting at TFI = 4, subsurface porosity is quite extensive, and the surface enamel could still be weakened with advancing age. This weakening of surface enamel may explain the higher prevalence of cohesive mode failures in enamel in teeth with TFI = 4-6. It may also explain why increasing etching time from 60 to 120 seconds did not result in a significant increase in the shear bond of the composite resin to severely fluorosed teeth (TFI = 4-6) of patients aged 40+ years (Table 1). However, the effect of age on the bond to enamel of nonfluorosed teeth needs to be investigated by future research. The exposure of dentinal tubules in some cases with mixed mode of failure or cohesive failure in enamel is consistent with clinical experience: enamel tends to shell off in some severely fluorosed teeth. Further research is needed to elucidate the effect of fluorosis on the dentinoenamel junction.

In clinical practice, therefore, a resin composite veneer is likely to be more retentive when etching time is increased from 60 to 120 seconds in patients aged below 40 years, but not in older patients. Furthermore, composite resin veneers are more likely to be debonded from severely fluorosed teeth (TFI = 4+) due to cohesive bond failure in enamel, especially in those patients aged 40+ years.

CONCLUSIONS

The severity of fluorosis has no significant effect on shear bond strength of composite resin to enamel. However, increasing etching time from 60 to 120 seconds resulted in a significant increase in shear bond strength for teeth of patients aged <40 years. Bond failure in severely fluorosed teeth was most likely to be cohesive in enamel.

Acknowledgment

This work was done in partial fulfilment of the requirements for the Master's degree of the King Saud University College of Dentistry, Riyadh, Saudi Arabia. We are grateful to the College of Dentistry Research Center for providing the facilities for this work.

(Received 29 December 1998)

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