Mechanical Behavior of Bioactive Glass and Hydroxyapatite Reinforced Compomers

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Abstract
The changes in the mechanical behavior of compomers reinforced with hydroxyapatite (HA) and bioactive glass (BAG) nanoparticles were investigated. The compomers reinforced with 4% HA showed the highest compressive strength values. 4% BAG and 1% HA addition to the compomers significantly decreased the elastic modulus of the material. The addition of 3% HA and 3% and 4% BAG particles enhanced the fracture toughness of the compomer restorative material. SEM images showed more voids on the fracture surface of the Bioactive glass groups than the hydroxyapatite groups. The increased proportion of BAG resulted in a decrease in compressive strength, flexural strength and elastic modulus of the compomer while, an increase in the fracture toughness of the material. Based on the results, higher hydroxyapatite content showed an increasing trend of mechanical behavior of compomer restorative materials.

Key words: bioactive glass; hydroxyapatite; compomer; mechanical properties.

1. Introduction

Resin-based restorative materials used in restorative treatments are preferred more by patients and physicians because of some reasons like biocompatibility and aesthetic properties. Compomers are among these materials that combine the fluoride release of glass ionomer cement with the aesthetics and mechanical benefits of composites (Manhart et al., 2000, Manhart et al., 2001).

It is known that compomer restorative materials, which is quite widely used in pediatric dentistry have several benefits and some negative features, as well. The most important problems of resin materials are low abrasion resistance and polymerization shrinkage. It's still undergoing continuous development and modification, in order to find the ideal dental restorative material. The total filler amount and particle form and size of the filler may change the mechanical behavior of the resin materials (Manhart et al., 2000, Jung et al., 2003).

One of the main problems of the light-curing materials is polymerization shrinkage which happens as a result of the aggregation of molecules during monomer to polymer conversion. Polymerization shrinkage causes stress between restoration and tooth interface as the elastic modulus of the composite increases while curing. This stress turns out a bond failure, cuspal flexure, enamel microcracking and secondary dental caries due to marginal leakage, moreover can cause restoration failure (Yap et al., 2001, Maia et al., 2015).

In the literature, until now, many studies have reported the results of hydroxyapatite and bio-active glass addition to glass ionomer cement and composites. In the current study, the results of hydroxyapatite and bio-active glass addition to compomers were carried out. Changes in mechanical behavior of the experimental compomers were evaluated.

2. Materials and methods

2.1. Mechanical Tests

The HA (Sigma-Aldrich, St Louis, MO, USA) and BAG (SCHOTT AG, Landshut, Germany) particles (1%, 2%, 3% and 4% w/w) were mixed mechanically as described previously (Kasraei and Azarsina, 2012) with the compomer restorative materials (Dyract®eXtra, Dentsply DeTrey, Konstanz, Germany)

using a plastic spatula for 30 min. The specimens were assigned to nine experimental groups and prepared for compressive strength (CS), flexural strength (FS) ,elastic modulus (EM) and fracture toughness (FT). For compressive strength 10 cylindrical specimens per group were prepared and inserted into stainless steel molds (SSM) consistent with the ISO 9917 (height 6 mm, diameter 4 mm) (ISO 9917-1, 2007). 10 specimens for each group were prepared consistent with International Organization for Standardization, Dentistry -Polimer-based restorative materials standard (ISO 4049, 2009) in SSM with internal dimensions 25x2x2 mm. American Society of Testing and Materials Standard test method E-399-90 (ASTM E399-90, 1997) was used to determine FT. Ten specimens for each experimental groups (25 mm long×2.5 mm wide×5 mm in height) were prepared with a 0.5 mm notch width and 2.5 mm depth.
The surface of the experimental compomers was lined by a Mylar matrix strip and pressed between 2 glass slides. All specimens were light-cured through the glass slides on top and bottom surfaces following manufacturers' directions with a light curing device (Elipar S10 LED, 3M ESPE, St. Paul. MN, USA). The samples were stored in deionized water and incubated at 37°C±2°C for 24 h.
CS, FS, EM and FT were measured automatically using a universal testing machine (Hounsfield H10KS, Hounsfield Ltd, UK) connected to the PC. The FS and the EM were calculated automatically by Qmat 3.63 computer program. FT, was calculated as described previously from the presented equation (Lucas et al., 2003, Eick et al., 2007):

$$K_Q: (P_Q S/BW^{3/2}).\ f(a/w)$$
$$f(a/W)=3(a/W)^{½}[1.99-(a/W)(1-a/W)*(2.15-3.93a/W+2.7a^2/W^2)]/2(1+2a/W)(1-a/W^3)\ ^{3/2}$$

2.2. SEM Evaluation
The fractured surfaces of the samples in the FT test were observed under a scanning electron microscope (SEM) (Quanta FEG 450, FEI, USA). The samples were gold coated by a sputter coater before SEM observations.

2.3. Statistical Analysis
Statistical analysis of the study results was performed with IBM SPSS Statistics-22 program (IBM SPSS, Turkey) by One-way ANOVA, Tukey HDS, paired sample t-tests.

3 Results and discussions
The results of CS, FS, EM and FT tests were presented in Tables 1a-3a, respectively. A statistically significant difference was found within the experimental groups in all tests (One way ANOVA, $p < 0.05$).

Table 1a: Compressive Strength of experimental groups. The data represent mean ± standard deviation(SD). One way ANOVA represents a significant difference between groups.*($p < 0.05$).

	Compressive Strength (Mpa)
	Mean± standard deviation (SD)
Control	261.9± 9.6
1% HA	223.0±17.7
2% HA	216.4± 9.7
3% HA	234.2±10.6
4% HA	249.4±10.6
1% BAG	215.9±12.2
2% BAG	216.3±10.5
3% BAG	200.4±6.3
4% BAG	186.7±5.1
p	0.001*

Oneway ANOVA Test,

Table 2a: Flexural strength and elastic modulus of experimental groups. The data represent mean ± standard deviation(SD). One way ANOVA represents a significant difference between groups.*($p < 0.05$).

	Flexural Strength (MPa)	Elastic Modulus (GPa)
Control	116.9±13.7	10.5±1.3
1% HA	78.7±7.0	8.6±1.2
2% HA	77.8±4.8	10.3±0.4
3% HA	87.9±2.3	9.4±0.6
4% HA	106.9±5.1	10.7±0.9
1% BAG	90.3±9.6	11.0±0.7
2% BAG	85.0±12.6	10.0±0.8
3% BAG	98.9±4.9	10.8±0.9
4% BAG	99.7±6.4	8.3±0.4
p	0.001*	0.001*

Oneway ANOVA Test,

Table 3a: Fracture toughness of experimental groups. The data represent mean ± standard deviation(SD). One way ANOVA represents a significant difference between groups.*($p < 0.05$).

	Fracture Toughness (MPa $m^{1/2}$)
Control	1.5±0.1
%1 HA	1.5±0.1
%2 HA	1.5±0.1
%3 HA	1.7±0.1
%4 HA	1.5±0.0
%1 BAG	1.6±0.1
%2 BAG	1.6±0.1
%3 BAG	1.7±0.1
%4 BAG	1.7±0.1
p	0.001*

Oneway ANOVA Test* $p<0.05$

The comparison between the groups was performed by Tukey HSD test (Table 1b-3b). The CS of the control and 4% HA groups were similar and significantly increased compared to the other groups ($p=0.001$). The compomers reinforced with 4% HA showed the highest CS values. 3% and 4% BAG groups presented decreased CS values in comparison to the other groups ($p < 0.05$), (Table 1b).

Table 1-b: Multiple comparison of compressive strength of experimental groups using post-hoc tests. Tukey HSD Test, represents a significant difference among groups. * p<0.05, ** p<0.01

	Compressive Strength (Mpa)
	P
Control / 1% HA	0.001**
Control / 2% HA	0.001**
Control / 3% HA	0.001**
Control / 4% HA	0.205
Control / 1% BAG	0.001**
Control / 2% BAG	0.001**
Control / 3% BAG	0.001**
Control / 4% BAG	0.001**
1% HA / 2% HA	0.911
1% HA / 3% HA	0.341
1% HA / 4% HA	0.001**
1% HA / 1% BAG	0.864
1% HA / 2% BAG	0.901
1% HA / 3% BAG	0.001**
1% HA / 4% BAG	0.001**
2% HA / 3% HA	0.012*
2% HA / 4% HA	0.001**
2% HA / 1% BAG	1.000
2% HA / 2% BAG	1.000
2% HA / 3% BAG	0.035*
2% HA / 4% BAG	0.001**
3% HA / 4% HA	0.058
3% HA / 1% BAG	0.008**
3% HA / 2% BAG	0.011*
3% HA / 3% BAG	0.001**
3% HA / 4% BAG	0.001**
4% HA / 1% BAG	0.001**
4% HA / 2% BAG	0.001**
4% HA / 3% BAG	0.001**
4% HA / 4% BAG	0.001**
1% BAG / 2% BAG	1.000
1% BAG / 3% BAG	0.048*
1% BAG/ 4% BAG	0.001**
2% BAG / 3% BAG	0.037*
2% BAG / 4% BAG	0.001**
3% BAG / 4% BAG	0.125

The FS of the control and 4% HA groups were similar and significantly increased compared to the other groups (p=0.001). The addition of 4% BAG and 1% HA to compomers significantly decreased the elastic modulus of the material (p=0.001), (Table 2b).

Table 2b : Multiple comparison of flexural strength and elastic modulus of experimental groups using post-hoc tests.Tukey HSD Test, represents a significant difference among groups. * p<0.05, ** p<0.01

	Flexural Strength (MPa)	Elastic Modulus (GPa)
	p	**p**
Control / 1% HA	0.001**	0.001**
Control / 2% HA	0.001**	1.000
Control / 3% HA	0.001**	0.114
Control / 4% HA	0.163	0.999
Control / 1% BAG	0.001**	0.916
Control / 2% BAG	0.001**	0.965
Control / 3% BAG	0.001**	0.996
Control / 4% BAG	0.001**	0.001**
1% HA / 2% HA	1.000	0.001**
1% HA / 3% HA	0.240	0.442
1% HA / 4% HA	0.001**	0.001**
1% HA / 1% BAG	0.049*	0.001**
1% HA / 2% BAG	0.733	0.006**
1% HA / 3% BAG	0.001**	0.001**
1% HA / 4% BAG	0.001**	1.000
2% HA / 3% HA	0.143	0.320
2% HA / 4% HA	0.001**	0.952
2% HA / 1% BAG	0.026*	0.655
2% HA / 2% BAG	0.572	0.999
2% HA / 3% BAG	0.001**	0.919
2% HA / 4% BAG	0.001**	0.001**
3% HA / 4% HA	0.001**	0.017*
3% HA / 1% BAG	0.999	0.002**
3% HA / 2% BAG	0.997	0.737
3% HA / 3% BAG	0.086	0.011*
3% HA / 4% BAG	0.047*	0.152
4% HA / 1% BAG	0.001**	0.999
4% HA / 2% BAG	0.001**	0.645
4% HA / 3% BAG	0.418	1.000
4% HA / 4% BAG	0.572	0.001**
1% BAG / 2% BAG	0.872	0.253
1% BAG / 3% BAG	0.342	1.000
1% BAG / 4% BAG	0.223	0.001**
2% BAG / 3% BAG	0.009**	0.561
2% BAG / 4% BAG	0.004**	0.001**
3% BAG / 4% BAG	1.000	0.001**

The addition of 3% HA and 3% and 4% BAG particles enhanced the fracture toughness of the compomer restorative material (p= 0.006, p=0.001), (Table 3b).

Table 3b: Multiple comparison of fracture toughness of experimental groups using post-hoc tests. Tukey HSD Test, represents a significant difference among groups. * p<0.05, ** p<0.01

	Fracture Toughness (MPa m1/2)
	p
Control / 1% HA	0.859
Control / 2% HA	1.000
Control / 3% HA	0.006**
Control / 4% HA	0.879
Control / 1% BAG	0.612
Control / 2% BAG	0.278
Control / 3% BAG	0.001**
Control / 4% BAG	0.001**
1% HA / 2% HA	0.975
1% HA / 3% HA	0.291
1% HA / 4% HA	1.000
1% HA / 1% BAG	1.000
1% HA / 2% BAG	0.989
1% HA / 3% BAG	0.079
1% HA / 4% BAG	0.013*
2% HA / 3% HA	0.021*
2% HA / 4% HA	0.981
2% HA / 1% BAG	0.850
2% HA / 2% BAG	0.521
2% HA / 3% BAG	0.003**
2% HA / 4% BAG	0.001**
3% HA / 4% HA	0.267
3% HA / 1% BAG	0.552
3% HA / 2% BAG	0.870
3% HA / 3% BAG	1.000
3% HA / 4% BAG	0.943
4% HA / 1% BAG	1.000
4% HA / 2% BAG	0.985
4% HA / 3% BAG	0.070
4% HA / 4% BAG	0.011*
1% BAG / 2% BAG	1.000
1% BAG / 3% BAG	0.207
1% BAG / 4% BAG	0.043*
2% BAG / 3% BAG	0.509
2% BAG / 4% BAG	0.161
3% BAG / 4% BAG	0.999

SEM images showed more voids on the fracture surface of the experimental compomers than the control group (Figures 1 and 2). Bio-active glass groups showed more voids in comparison with hydroxyapatite groups.

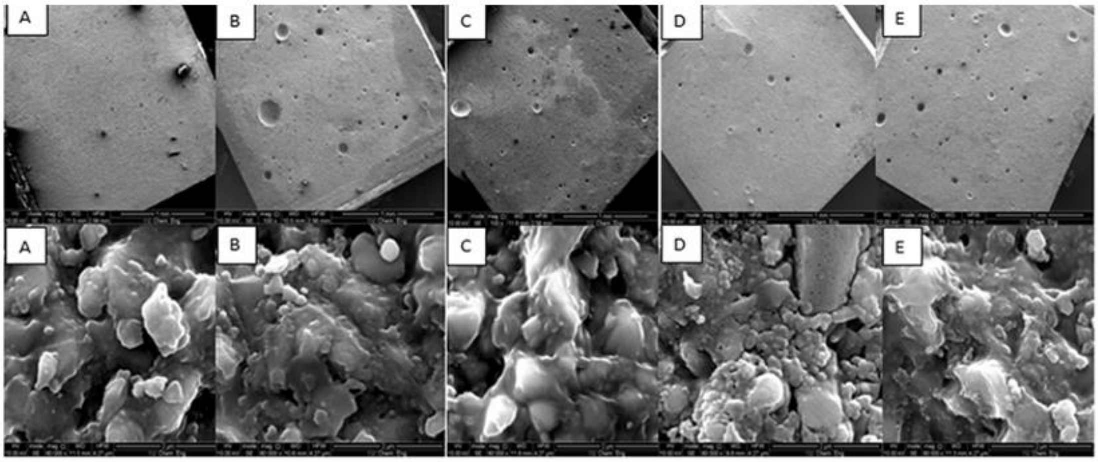

Figure 1: The fractured surfaces of HA modified compomer restorative materials, SEM images. A: Control, X1.000, A: Control, X60.000, B: %1 HA, X1.000, B: %1 HA, X60.000, C: %2 HA, X1.000, C: %2 HA, X60.000, D: %3 HA, X1.000, D: %3 HA, X60.000, E: %4 HA, X1.000, E: %4 HA, X60.000.

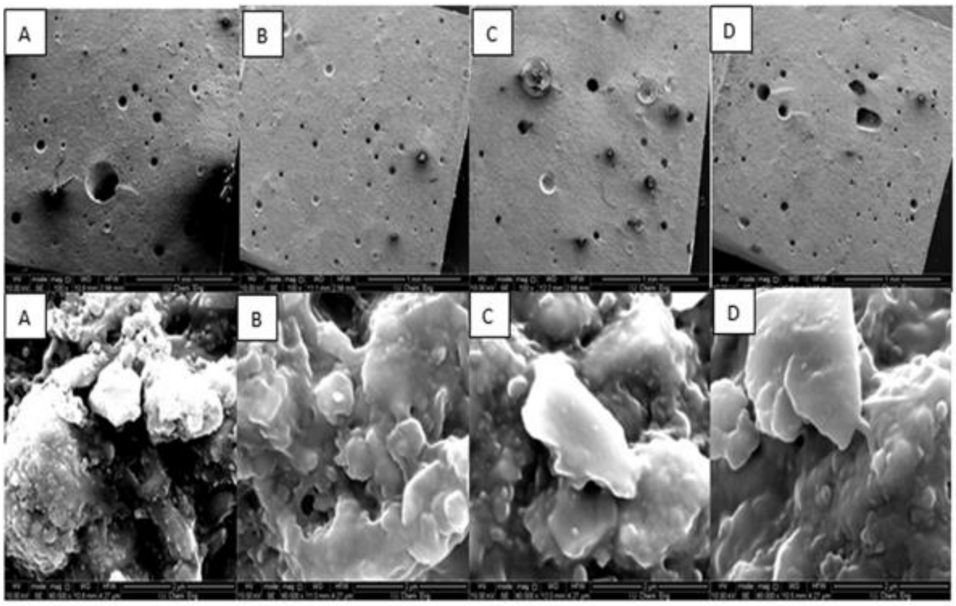

Figure 2: The fractured surfaces of BAG modified compomer restorative materials, SEM images. A: %1 BAG, X1.000, A: %1 BAG, X60.000, B: %2 BAG, X1.000, B: %2 BAG, X60.000, C: %3 BAG, X1.000, C: %3 BAG, X60.000, D: %4 BAG, X1.000, D: %4 BAG, X60.000.

Bioactive glasses are synthetic materials generally made of calcium, phosphorus, silicon, and sodium oxides and release ions needed for remineralization of tooth tissue (Khvostenkoa et al., 2013, Osorio et al., 2016).

The incorporation of reinforcing nano particles in dental restorative materials enhance aesthetics, antimicrobial activity and physical properties (Yap et al., 2002, Mitra et al., 2003, Mu et al., 2007, Zakir et al., 2013).

There are several reports which indicate a strong reaction between cement matrix and nanoparticles. Moshaverinia et al. (2008) added 5% nano-hydroxyapatite and fluorapatite particles to the GIC and reported a positive impact on the mechanical properties of the cement.

The release of calcium ions from the surface of nanoparticles and higher occurrence of crystallization reactions are strongly related with the enhance in mechanical properties of the cements (Suchanek et al., 1996, Moshaverinia et al., 2008, Lee et al., 2010).

The addition of nano-hydroxyapatite at different weight percentages was evaluated by several studies.

Gu et al. (2005) reported higher compressive and tensile strengths compared to the original cement by addition of 4 and 12% hydroxyapatite to conventional glass ionomer powder.

Moshaverinia et al. (2008) added 4-5% w/w hydroxyapatite to glass ionomer cement and reported an increase in compressive strength.

Mu et al. (2007) reported the increase in flexural and compressive strength of 8% nano- hydroxyapatite -added conventional glass ionomer.

Mohammadi Basir et al. (2013) reported that 5% nano-hydroxyapatite addition to a resin-modified glass-ionomer cement (RMGI) enhanced the CS of the material.

The incorporation of nano-hydroxyapatite up to 10wt% showed an increase in the wear resistance of a resin-modified GIC and the highest increase was reported in 2% and 5% wt nano-hydroxyapatite modified materials (Poorzandpoush et al., 2017).

Sharafeddin et al. (2017) reported that the hardness of RMGI and Zirconomer increased by 5 and 15 wt% of microhydroxyapatite addition, but the increased amount of HA resulted a decrease in the hardness of the materials.

Based on previous studies (Hammouda, 2009, Garoushi et al., 2011, Khaghani et al., 2013, Khoroushi et al., 2013), in this study, the HA and BAG nanoparticles were added at 1-4 % w/w to compomers in order to prevent polymerization problems that can negatively affects the material’s unique properties.

The restorative materials reinforced with bio-active glasses showed a decrease in compressive strength (Matsuya et al., 1999, Ana et al., 2003, Yli-Urpo et al., 2005). Matsuya et al., (1999) reported that the decrease in compressive strength of the materials was possibly related with the pH of the polymeric acid and the reactivity of the glass powder, without a relation to the structure or the amound of additives.

Ana et al., (2003) reported the possible mechanism of lower compressive strength with reduced amount of aluminum cations during partial replacement of cement powder with bio-active glass.

In the present study, we added hydroxyapatite and bio-active glass to compomers (polyacid modified composite resins) instead of the conventional glass powder or RMGI.

Results of the current study demonstrated that the CS of compomers increased with the increasing amount of hydroxyapatite, but presented a decrease as the amount of bio-active glass increased. It seems that 1-3 wt % of nanohydroxyapatite addition is not effective to enhance the compressive strength of compomers. The 4 wt % of nanohydroxyapatite addition showed similar compressive strength compared to the original cement. The presented compressive strength results of BAG modified compomer restorative materials were similar to previous studies (Matsuya et al., 1999, Ana et al., 2003, Yli-Urpo et al., 2005) which reported the decrease of CS due to increasing amount of bio-active glass particles.

The flexural strength of the material significantly decreased by hydroxyapatite and bio-active glass addition. Results of the flexural strength test revealed higher flexural strength by increasing the weight percentage of hydroxyapatite nanoparticles, but the flexural strength of bio-active glass modified groups didn’t show an increase by the increasing amount. The 4 wt % of nanohydroxyapatite addition showed similar flexural strength compared to the original cement.

In this study, the increased amount of bio-active glass resulted a decrease in elastic modulus of the compomer. Elastic modulus increased as the amount of hydroxyapatite increased.

Flexural strength and elastic modulus results of modified groups were similar to that reported by Arcis et al., (2002), Zhang et al., (2012) and Yang et al., (2013). However, different results were obtained from Lohbauer et al., (2003), Mu et al., (2007) and Hammouda, (2009). These differences may be due to the amount of hydroxyapatite and type of bio-active glass. Further designed studies are necessary to investigate the effects of higher percentages of nanohydroxyapatite on compressive strength, flexural strength and elastic modulus of compomers.

Lucas et al., (2003) added 8 wt% of HA to the glass ionomer cement and reported an increase in toughness and bond strength to dentin.

Lucksanasombool et al., (2002) added 0%, 10%, 20%, 30% w/w glass fiber to glass ionomer and they reported a decrease in fracture toughness with increased porosity.

Lohbauer et al., (2004) and Hammouda, (2009) reported the increase of fracture toughness with increasing fiber concentration in glass ionomer cement.

In this study, the incorporation of the 3-4 wt % of BAG and HA particles increased the fracture toughness of the material.

Filler morphology is very important for reinforcing the efficacy of dental materials.

Zhang at al., (2012) added hydroxyapatite whiskers and nano-scale powder in varied proportions to dental composites and reported a significant decrease in flexural strength with increasing volume fraction of hydroxyapatite powder.

Whiskers had sensible dispersibility and wettability than hydroxyapatite nano-scale powder with a bis-GMA-based polymer (Roeder et al., 2003).

Lezaja et al., (2013) added different form and size of hydroxyapatite to the composites and reported that the modified composites presented increased flexural strength than the control group.

In the present study, 1-4 % w/w nanopowder (<200 nm p.s) of hydroxyapatite and inorganic glass powder (grain size K5) of bio-active glass were added manually to compomers.

The differences between present study and other researchs may be due to the experimental setups like silanization, hand-mixing and non-uniform dispersion of nanoparticles.

The addition of BAG to a polymeric matrix has been shown to alter the degradation rate of the material by changing parameters such as hydrophobicity, water absorption, weight loss, pH and surface morphology (Li and Ghang, 2005, Osorio et al., 2016).

Osorio et al., (2016) investigated the effects of BAG on surface nanoroughness and topography of RMGICs. They reported the changes on the surface morphology after wet and dry storage conditions. The changes depended on the particles size of the RMGICs in dry conditions, but the changes were related with the dissolution of the BAG particles, a silica-rich gel formation and a hydroxyl carbonate apatite precipitation on the surface of the materials in wet conditions (Osorio et al., 2016).

In this study, SEM observations of fractured surfaces showed a lot of voids in the experimental groups than the control group. Bio-active glass groups showed more voids in comparison with hydroxyapatite groups. Hand-mixing of the nano-particles to compomer restorative materials may cause the formation of voids and adversely affect the mechanical properties.

4. Conclusion

The resuts of this study suggests that mixing HA and BAG nanoparticles into compomer produced changes in the mechanical behavior of the restorative material.

These changes depended the amount of additives. The increased amount of BAG resulted a decrease in flexural, compressive strength and elastic modulus of the compomer while, an increase in the fracture toughness of the material. The increased hydroxyapatite amount showed an increasing trend of mechanical behavior of compomer restorative materials. Thus, addition of higher percentages of nanohydroxyapatite to compomers may improve its mechanical properties.

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