Published online: 2019-10-04

Original Article

Hardness and modulus of elasticity of primary and permanent teeth after wear against different dental materials

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ABSTRACT

Objectives: The purpose of this study was to determine the Young's modulus and the hardness of deciduous and permanent teeth following wear challenges using different dental materials. **Materials and Methods:** Wear challenges were performed against four dental materials: A resin-based fissure sealant (Fluoroshield®), a glass ionomer based fissure sealant (Vitremer®), and two microhybrid composite resins (Filtek Z250 and P90®). Using the pin-on-plate design, a deciduous or a permanent tooth was made into a pin (4 mm × 4 mm × 2 mm) working at a 3 N vertical load, 1 Hz frequency, and 900 cycles (15 min) with Fusayama artificial saliva as a lubricant. Before and after the tribological tests, the hardness and elasticity modulus of the tooth samples were measured by creating a nanoindentation at load forces up to 50 mN and 150 mN. All of the results were statistically analyzed using ANOVA and *post-hoc* Duncan's tests ($P < 0.05$). **Results:** No difference in hardness was encountered between deciduous and permanent teeth ($P < 0.05$) or modulus of elasticity ($P < 0.05$) before or after the wear challenges for all of the dental materials tested. **Conclusions:** Wear challenges against the studied dental materials did not alter the properties of permanent or deciduous teeth after the application of a 3 N load.

Key words: Composite resin, glass ionomer sealant, nanoindentation, resin fissure sealant, two-body wear

INTRODUCTION

Tooth enamel is the hardest and most mineralized biological substance in the human body, and it presents heterogeneous and anisotropic properties.[1-3] In addition, the enamel has a compact and complex matrix made of phosphate and calcium salts in the shape of large hexagonal hydroxyapatite crystals.[2]

Tooth enamel cannot be effectively substituted by any restorative material because of its specific substrate. One of its main characteristics is resistance to wear despite the wide range of working conditions it faces, which include varied loads, alternate movements, shocks and impacts, temperature oscillations, and possible acidic challenges.[4] The enamel is subjected to different situations within the oral cavity during mastication, and if it becomes ruptured as a result of a fracture or considerable wear, the subjacent dentin becomes exposed. The enamel is thicker at the cusps (2–3 mm) and thinner at the cement-enamel junction.[5] Enamel loss substance through mastication at a rate of 10–40 μm/year, while the mean wear for dental restorative materials under varied clinical situations ranges from 8 to 9 μm/month.[6] The difference in wear is the principal reason several

How to cite this article: Galo R, Contente MM, Galafassi D, Borsatto MC. Hardness and modulus of elasticity of primary and permanent teeth after wear against different dental materials. Eur J Dent 2015;9:587-93.

DOI: 10.4103/1305-7456.172635

researchers seek to improve our understanding of dental enamel wear.

Investigating enamel's wear against restorative materials is a fundamental step toward understanding its properties, measuring stress distribution, and developing biometric restorative materials.[7,8] Clinical trials are undoubtedly the best way to establish tooth wear; however, they present some difficulties, such as high costs and lengthy time requirements. Preliminary tests involving *in vitro* approaches are less costly and are reasonably effective in achieving experimental goals. The main disadvantage is the difficulty of translating *in vitro* findings into clinical practice.[5]

Fundamentally, the tribological response depends upon the mechanical properties of the materials used (i.e., elasticity modulus, resistance, and hardness). Nanoindentation trials have been used to examine tooth tissue.[2] These trials rely on a short symmetrical indenter which penetrates the enamel area with a known load. The reading device continuously registers changes in depth and indentation during loading and unloading cycles. From these readings, elasticity modulus and hardness can be calculated as functions of a visual analysis of the indentation produced.[9,10]

The present study analyzed tooth wear in experimentally controlled conditions that simulate the mastication system. Although tooth wear may involve enamel and dentin, this study focused on the wear features of the enamel. The research was performed using a tooth wear simulation designed to examine enamel wear. In this simulation, mastication cycles consisted of a bidirectional movement test associated with a specific load. The tested null hypothesis was that there was no difference in hardness or modulus of elasticity (Young's modulus) for deciduous and permanent teeth following wear challenges against different dental materials.

MATERIALS AND METHODS

For the present study that was approved by the Ethics Committee of University of São Paulo (2011.1.1123.58.3). Freshly, third molar teeth and primary molar human were taken from different subjects of varying age and different dietary habits. All the teeth were cavity free and did not have any visible surface cracks and they were cleaned with water/pumice slurry in rotating bristle brushes to remove calculus and root-adhered debris, and were examined under a ×20 magnifier to discard those with structural defects. Teeth were stored in saline (0, 9%) with sodium azide (0, 4%) at 4°C.

The crowns were established on plates. The pieces of the teeth were removed using a double-faced diamond disk (KG Sorensen, 7015, Barueri, SP, Brazil) mounted on a low-speed handpiece to expose the testing surface, under tap water irrigation. Twelve fragments with standard dimensions (2 mm) in thickness were created. Then, the samples were fixed with wax in a cylindrical Plexiglass® using a parallelometer to ensure that the enamel surface was maintained perpendicular to the horizontal plane. The final fragments had dimensions of 4 mm height × 4 mm width × 2 mm thick. To ensure that the surfaces of the exposed teeth were free from deformations and risks, the samples were polished before indenting. To reach these results, the surface material has been removed through successively thinner abrasive particle size. The process of grinding and polishing is summarized in Table 1 and was based on the work of Mahoney *et al*.[11]

Then, the fragments were embedded in resin (Epofix Kit-Struers, A/S, Ballerup, Denmark) using polyvinyl chloride base using a paralleling machine. After resin polymerization, the specimens were kept in distilled water and removed 24 h before the tribological tests were started.

The tribological experiments were performed *in vitro* using a pin-on-plate design and alternating sliding movements against the surface of the restorative dental materials (0.646 cm^2), following a 4 mm path (stroke) with the pin made from a human tooth (4 mm^2) as the antagonist. The readings were registered using a tribometer (Tribometer TE 67, Plint, Tribology Products, UK). The tribological parameters for the wear challenges were set as 3 N of load and 1 Hz frequency for 900 cycles (15 min). Fusayama's artificial saliva consisting of NaCl (400 mg/L), $CaCl_2 \cdot H_2O$ (795 mg/L), KCl (400 mg/L), $NaS_9 \cdot H_2O$ (5 mg/L), $NaH_2PO_4 \cdot H_2O$ (690 mg/L), and urea (1,000 mg/L; Sigma Chemical Company,

Table 1: Summary of the grinding and polishing steps

Step	1	2	3	4
Abrasive	Silicon-carbide	Silicon-carbide	Diamond particle	Diamond particle
Grit/grain size	500#	1000#	9 mm	1 mm
Lubricant	Water	Water	DP-Green	DP-Red
Rotational speed (rpm)	150	150	150	150
Cleanser time (min)	10	10	10	10

*Mahoney E, *et al*. J Dent 2000;28:589-94.[11] #finer sizes of abrasive particles

St. Louis, MO, USA) with a pH of 5.5 at 24°C was used as a lubricant. This specific test setting has been selected because according to Zheng *et al*.,[12] wear tests and friction on the teeth should be performed in a reciprocal sliding mode, rather than a unidirectional sliding pin-on-plate or other tribological testing mode, to better simulate mastication behavior.

The dental materials used in this study were a resin-based pits-and-fissures sealant (Fluroshield, Dentsply/Caulk, Milford, DE, USA), a resin-modified glass ionomer cement for pit and fissure sealing (Vitremer, 3 M/ESPE, St. Paul, MN, USA) and a microhybrid composite resin containing a silorane-based organic matrix (Filtek P90, 3 M/ESPE, St. Paul, MN, USA).

A cylindrical Teflon® matrix with a dimension of the 5 mm diameter and 2 mm height was fixed onto the tooth surface to hold the material during insertion. This matrix was filled with Fluroshield® for Group 1, Vitremer® glass ionomer cement for Group 2, Filtek Z250® composite for Group 3, and Filtek P90® composite for Group 4 following the manufacturer's instructions and taking care to avoid air bubbles. The Group 1 specimens were light-cured for 20 s using a halogen light (400–470 mW/cm^2). For Group 2, the cement was mixed at 1:3 for fluidity, followed by 20 s light-curing and glaze (finish gloss). The Groups 3 and 4 specimens were built following the incremental method and were light-cured.[13]

The test samples were stored in distilled water at 37°C for 48 h prior to thermal cycling in alternating baths of 5°C and 55°C. The immersion time was set at 30 s, and the interval between baths was 30 s, for a total of 500 cycles. The samples were then rinsed and stored in distilled water in an incubator at 37°C for 24 h prior to air drying and tribological testing.

Before and after the tribological tests, the hardness and elasticity modulus of the tooth samples were measured using nanoindentation. In each tooth, 20 indentations were made on the teeth enamel. Half of the 20 indentations in the enamel were conducted at a load force up to 50 mN, and the other half had a loading force of up to 150 mN. The distance between the nanoindetations was 50 µm in both the x- and y-axis. The software lowered the indentor until a contact force of 0.2 mN was encountered. This was taken as the baseline datum point from which the load was gradually increased in 25 increments at a rate of one incremental increase per 0.1 s up to the maximum load (either 50 or 150 mN) where there was a delay of 30 s followed by the same incremental unload process. This protocol has been used in other studies.[10] Testing each tooth took approximately 6 h (contact force – 0.2 mN; dwell at load/max/unload –0.1/30/0.1 s; maximum indentation force – 50 or 150 mN; array row size –3; array column size – 17–35; delay between locations –30 s; testing conditions – 23 ± 1°C and 50 ± 10% RF). There was some concern regarding the possible effects of drying over this period; however, earlier work showed that teeth could be dry for up to some days (2 days) without influencing the modulus of elasticity and hardness of either enamel or dentine.[11]

Compositional analysis was conducted using energy-dispersive spectroscopy (EDS), and the microstructure was assessed using the scanning electron microscope (SEM). All of the results were statistically analyzed using ANOVA and *post-hoc* Duncan's tests ($P < 0.05$).

RESULTS

Tables 2 and 3 show the enamel hardness and elasticity modulus results for primary and permanent teeth before and after tribological tests. A total of 10 nanoindentations were created in the enamel of each tooth under each load to examine the relationship between elasticity modulus and hardness for enamel after/before tribological tests under loads of 50 and 150 mN. No statistically significant relationship between the elasticity modulus and hardness of the enamel under either load (50 and 150 mN).

The mean hardness of the primary tooth enamel [Table 2] before the tribological tests was 3.81 ± 0.73 GPa at 50 mN and 4.11 ± 0.94 GPa at 150 mN, and the mean hardness after the tribological tests was 3.47 ± 1.08 GPa at 50 mN and 3.76 ± 0.92 GPa at 150 mN. The mean elasticity modulus of this enamel after the tribological tests were 94.13 ± 7.99 at 50 mN and 85.04 ± 16.31 at 150 mN; before the tribological tests, the mean elasticity modulus was 96.84 ± 7.68 at 50 mN and 82.10 ± 12.47 at 150 mN.

The mean hardness of the permanent tooth enamel [Table 3] before the tribological tests was 4.70 ± 0.60 GPa at 50 mN and 4.39 ± 0.39 GPa at 150 mN; after the tribological tests, the mean hardness was 4.55 ± 0.84 GPa at 50 mN and 4.49 ± 0.65 GPa at 150 mN. The mean elasticity modulus of the permanent tooth enamel before the tribological testing

Table 2: Means average and SD (GPa) of primary enamel nanohardness and elastic modulus immediately and after wear

Load applied	Properties	Experimental groups (means±SD)							
		Dental material before wear				Dental material after wear			
		Fluroshield	Vitremer	Z350	P90	Fluroshield	Vitremer	Z350	P90
50 mN	Hardness	3.95±1.03	3.75±0.34	3.71±0.80	3.95±0.67	3.91±0.51	3.25±1.31	3.25±1.32	3.18±1.26
	Modulus of elasticity	95.42±9.33	99.49±3.74	95.62±9.55	100.43±4.20	94.12±6.65	96.19±10.12	92.10±8.14	98.43±9.02
150 mN	Hardness	4.50±1.44	3.85±0.24	3.97±0.83	4.12±0.77	3.74±1.00	3.85±1.01	3.70±0.96	3.95±0.45
	Modulus of elasticity	81.92±14.69	83.30±10.70	81.08±14.53	83.20±16.68	95.94±8.26	97.57±8.04	92.32±5.42	77.02±19.66

SD: Standard deviation

Table 3: Means average and SD (GPa) of permanent enamel nanohardness and elastic modulus immediately and after wear

Load applied	Properties	Experimental groups (means±SD)							
		Dental material before wear				Dental material after wear			
		Fluroshield	Vitremer	Z350	P90	Fluroshield	Vitremer	Z350	P90
50 mN	Hardness	4.87±0.66	4.65±0.63	4.58±0.62	4.45±0.41	4.60±0.85	4.39±1.01	4.65±0.82	4.41±0.91
	Modulus of elasticity	106.04±7.72	100.79±7.57	104.61±8.44	101.58±8.24	104.97±13.44	102.93±12.98	107.12±10.93	106.62±14.15
150 mN	Hardness	4.57±0.33	4.25±0.49	4.37±0.34	4.16±0.41	4.47±0.84	4.36±0.43	4.35±0.76	4.64±0.51
	Modulus of elasticity	95.94±8.26	97.57±8.04	92.32±5.42	94.18±8.56	95.17±11.30	94.52±9.42	94.30±11.02	101.22±4.95

SD: Standard deviation

was 103.81 ± 7.69 at 50 mN and 95.28 ± 7.18 at 150 mN; the mean elasticity modulus after the tribological tests was 105.01 ± 11.71 at 50 mN and 94.66 ± 9.83 at 150 mN.

Representative SEM images reveal the microstructure of the primary and permanent enamel. In Figure 1, SEM analysis shows the cracks on the tooth surface and detached "platelet" particles. In additional, EDS examinations of the tooth surface indicate that the "platelet" particles in the base are composed of elements obtained from the debris as a result of material transfer from the opposite specimens.

DISCUSSION

Tooth wear is a natural and inevitable process that results from the physiological role of the teeth in the mouth. Exceptional wear can lead to the inadequate articulation of the teeth, which in turn can disrupt the masticatory efficiency of the system and can obliterate the masticatory surfaces. Moreover, the anisotropy of human teeth, in terms of mechanical property variations and gradient of mineral concentration between the dentin and the enamel, may also influence the teeth's tribological behavior.[5,7,14]

Another contribution of the enamel prisms is attributed to the prisms' compound nature. As noted by Katz,[15] stresses will be carried by the high stiffness crystals if indentations are made parallel to the rod axis, and higher hardness and elasticity modulus values will be obtained. As opposed, when stress is applied across the crystals, the low stiffness, the deformable organic matrix surrounding them, producing lower elasticity modulus and hardness in this direction carries it. As a consequence, the resulting enamel anisotropy can be greater than that of single apatite crystals.

The null hypothesis was accepted because the hardness and elasticity modulus values did not reach statistical significance for either deciduous or permanent teeth when the values before and after the tribochemical action were compared.

The custom-made teeth-on-composite sliding wear machine used in this study was operated in the attrition mode, that is contact occurred between the antagonist teeth (primary and permanent) and the underlying composite in saliva to simulate attrition wear. Considering that clinical studies present considerable limitations, such as complex methodology and difficulties with measurement and precise analyses,[16] *in vitro* studies can be more readily controlled, thus increasing our understanding of wear mechanisms.[16]

A two-body device was used in this study to simulate direct contact between the teeth and the test samples. The present study was essential for understanding deciduous and permanent tooth behavior when submitted to wear challenges without abrasion. It is hard to predict the effect of wear resistance teeth

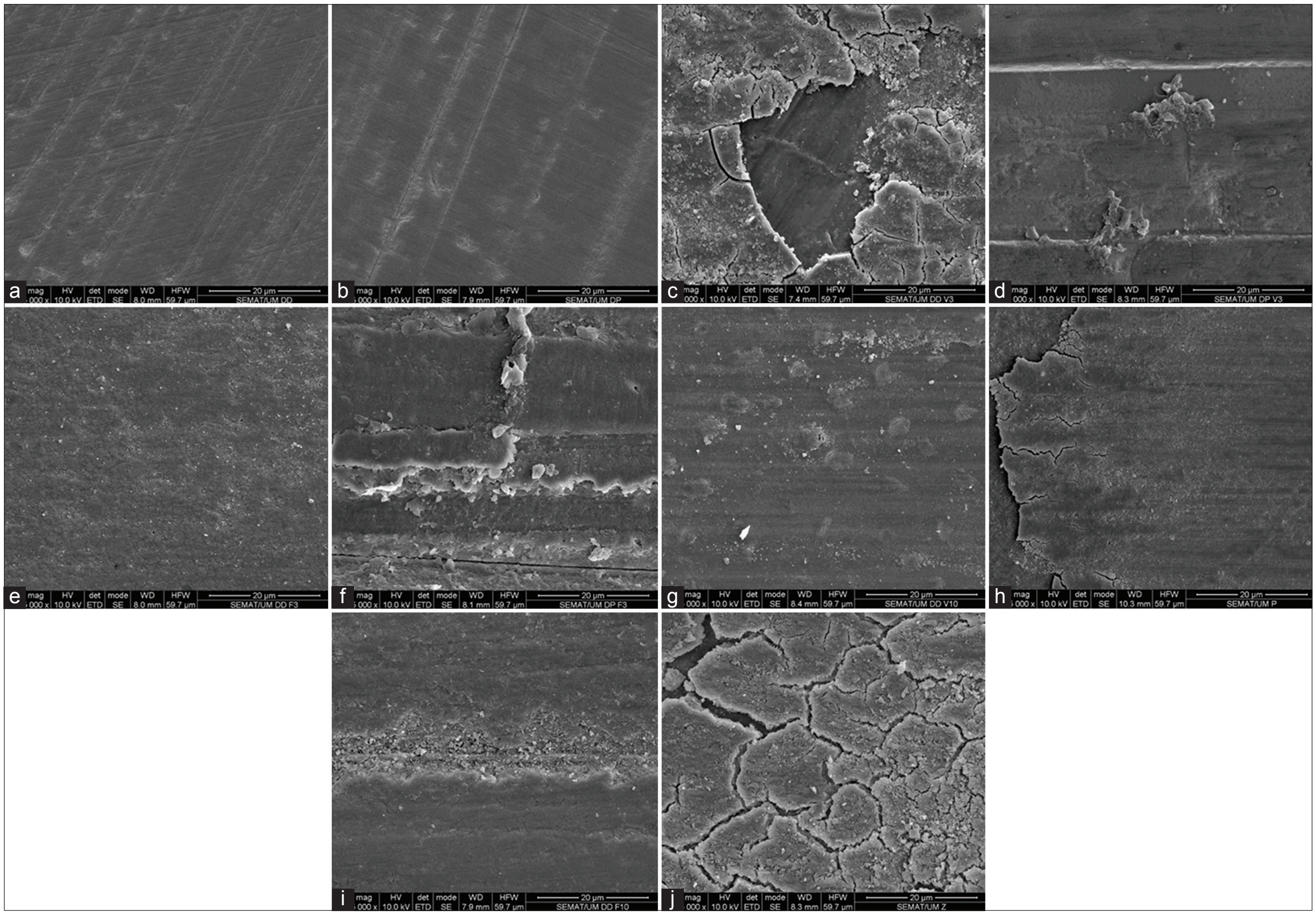

Figure 1: Representative scanning electron micrographs of superficial wear. (a) Primary teeth baseline; (b) permanent teeth baseline; (c) primary teeth after wear with Vitremer; (d) permanent teeth after wear with Vitremer; (e) primary teeth after wear with Fluorshield; (f) permanent teeth after wear with Fluorshield; (g) primary teeth after wear with composite P90; (h) permanent teeth after wear with composite P90; (i) primary teeth after wear with composite Z250; (j) permanent teeth after wear with composite Z250

environment. A few *in vitro* studies have demonstrated conflicting results with progressive wear.[17,18] With a limited layer of enamel, a vertical wear facet most likely invades the enamel sublayer. It can affect total wear resistance. In this study, the orientation of test area was monitored to focus on the enamel sublayer.

In this study, around 10% of the enamel test indentations have been discarded, which is resembling the proportion reported in other studies.[11,19] The results from the indentations were excluded if they provoked surface cracking because surface damage can affect nanoindentation. In enamel, the current values for the elasticity modulus and the hardness did not differ significantly in comparison to the load applied. Moreover, the loads applied in the current study were relatively low and affected mainly the enamel, and it was noted that the load of 50 mN was bit less satisfactory, as there was further evidence of significant cracking in the enamel.

The mechanical, chemical, and microstructural properties of the enamel have also been shown to vary with tooth region. Zheng *et al*.[12] demonstrated that enamel prism orientation plays an important role during attrition and that the enamel at the surface has a higher wear resistance than the enamel closer to the dentin.[19] In addition, the mechanical properties of the proximal surface of a human molar have higher variability than our limited sample could represent. There is clearly room for further investigation. These results represent the majority of the data, detailing the analysis of variations in hardness and elasticity modulus in each tooth during wear challenges.

The enamel hardness of deciduous teeth has been studied before; however, it is very hard to establish proportions because other studies have performed different tests of hardness like the Knoop hardness test.[20] However, Mahoney *et al*.,[11] reported deciduous enamel hardness values of 4.88 ± 0.41 GPa at 50 mN and

4.87 ± 0.29 GPa at 150 mN. These figures were similar to those found in this study, in which the hardness at 50 mN was 3.81 ± 0.73 GPa, and the hardness at 150 mN was 4.11 ± 0.94 GPa.

For permanent teeth, the results were 4.70 ± 0.60 GPa at 50 mN and 4.39 ± 0.39 GPa at 150 mN. These figures are slightly higher than those reported in another study,[21] in which the values were 3.62 ± 0.2 GPa and 3.37 ± 0.15 GPa, respectively. In our study, the permanent teeth had slightly harder enamel (4.60–4.39 GPa) compared with the primary enamel (3.81–4.11 GPa). The results demonstrated that the permanent teeth remained slightly harder (4.55–4.49 GPa) than the deciduous teeth (3.47–3.76 GPa).

The differences in the hardness and elasticity modulus values before and after tribological testing were not statistically significant, although the values were slightly lower after testing, possibly because of dental material residue that was incorporated into the tooth surface, as observed by SEM [Figure 1]. However, the materials used in this study did not influence the hardness and elasticity values in either the deciduous or permanent teeth, although the literature has reported that the mean filler size and volume had quite an effect on the tribological properties of dental materials.[22]

Throughout this paper, the mean enamel hardness values have been quoted. It is obvious, however, that there is a variation in individual teeth, both permanent (4.55–4.49 GPa) and deciduous (3.47–3.76 GPa). These differences may be explained in various ways. First, differences may arise from the fact that the teeth came from several individuals who were exposed to different environmental factors throughout the development and mineralization of the teeth. It is widely accepted that fluoride can influence enamel hardness, during tooth formation and after a tooth's eruption in the mouth. It would have been very hard to address these variables from this study. Curiously, there are also variations in hardness within the same tooth. These variations can occur as a result of variations in crown mineralization.[11]

The elasticity modulus of the enamel is affected by the orientation of the indentations. Xu *et al*.,[21] mentioned that the Young's modulus of the permanent enamel at the occlusal aspect is significantly different from that of the axial region (94.5 GPa vs. 80.4 GPa). In the current study, the elasticity modulus of the permanent tooth at the axial region was 103.81 GPa at 50 mN and 95.28 GPa at 150 mN. However, the values we obtained for the deciduous teeth were 96.84 at 50 mN and 82.10 at 150 mN before the wear challenge. These values are little higher than those reported by Mahoney *et al*.[11] After the wear challenge, the mean values remained similar, demonstrating that there were no property changes in terms of the elasticity modulus.

The present study used a nanoindentation test that provides basic data on the mechanical properties of deciduous and permanent teeth following mechanical wear. Such data may help to predict the behavior pattern of loaded dentition and the interaction between tooth and restoration. Conventional methods for measuring hardness and elasticity modulus have limitations. This research shows that the mechanical properties of human teeth on a microscopic level are highly dependent on the microstructural characteristics of the teeth.

Acknowledgments

The State of São Paulo Research Foundation (FAPESP – no. 2010/05834-9) for financial support of this research.

Financial support and sponsorship

The State of São Paulo Research Foundation.

Conflicts of interest

There are no conflicts of interest.

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