



## In vitro analysis of dental ceramics: evaluation of the radiopacity and chemical composition by Raman spectroscopy

Análise in vitro de cerâmicas odontológicas: avaliação da radiopacidade e composição química por espectroscopia Raman

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### ABSTRACT

**Objective:** This study compared the radiopacity of different ceramic systems by means of digital radiographs and evaluate the chemical composition of the samples by Raman spectroscopy. The hypothesis tested was that there was a difference in radiopacity among the tested materials. **Material and Methods:** Specimens were prepared for each ceramic tested: FLD - VM7 (VITA Zahnfabrik), LD - IPS Empress e.max Press (IPS Empress), AL - In Ceram Alumina (VITA Zahnfabrik), ALYZ - In Ceram Zirconia (VITA Zahnfabrik), YZ - Lava All Ceram (3M/ESPE), and MYZ - Zircons Zahn (Talladium Brasil). The specimens were radiographed and submitted to radiographic density readings using a histogram tool. The spectrometer coupled to a petrographic microscope was used for Raman spectroscopy measurements. Analysis of variance (ANOVA) and a Tukey post-hoc test were used to compare radiopacity of the different materials. **Results:** For all tested materials, the radiopacity showed statistically significant differences, except YZ and MYZ. Lava All Ceram and Zircons Zahn had high radiopacity values and VM7 and IPS Empress e.max Press showed lower radiopacity than human dental structures. **Conclusion:** It was possible to conclude that radiopacity is closely linked to ceramic chemical composition.

### KEYWORDS

Dental ceramics; Radiopacity; Raman spectroscopy test; Restorative dentistry.

### RESUMO

**Objetivo:** Este estudo comparou a radiopacidade de diferentes sistemas cerâmicos por meio de radiografias digitais e avaliou a composição química das amostras por espectroscopia Raman. A hipótese testada foi que haveria diferença na radiopacidade entre os materiais testados. **Material e Métodos:** Os espécimes foram preparados para cada cerâmica testada: FLD - VM7 (VITA Zahnfabrik), LD - Impressora IPS Empress e.max (IPS Empress), AL - Em Ceram Alumina (VITA Zahnfabrik), ALYZ - In Ceram Zirconia (VITA Zahnfabrik), YZ - Lava All Ceram (3M / ESPE) e MYZ - Zircons Zahn (Talladium Brasil). Os espécimes foram radiografados e submetidos a leituras de densidade radiográfica utilizando uma ferramenta de histograma. O espectrômetro acoplado a um microscópio petrográfico foi utilizado para medidas de espectroscopia Raman. Análise de variância (ANOVA) e teste post-hoc de Tukey foram usados para comparar a radiopacidade dos diferentes materiais. **Resultados:** Para todos os materiais testados, a radiopacidade apresentou diferenças estatisticamente significantes, exceto YZ e MYZ. Lava All Ceram e Zircons Zahn apresentaram altos valores de radiopacidade e o VM7 e o IPS Empress e.max Press apresentaram menor radiopacidade do que as estruturas dentais humanas. **Conclusão:** Foi possível concluir que a radiopacidade está intimamente ligada à composição química da cerâmica.

### PALAVRAS-CHAVE

Cerâmica dentária; Radiopacidade; Teste de espectroscopia Raman; Odontologia Restauradora.

## INTRODUCTION

Dental ceramics are the materials of choice for various esthetic restorations due to their characteristics, such as high compressive strength and abrasion resistance, chemical stability, favorable esthetic features, translucency, biocompatibility, fluorescence and thermal expansion coefficient similar to that of the dental structure [1,2].

One of the most desirable characteristics of any restorative material is a radiopacity that allows it to be distinguished from dental structures, helps with detection of secondary carious lesions, marginal defects, restoration contours, adaptation of restorations to cavity walls, contact points between adjacent teeth, cement excess, and interfacial gaps [3,4].

Clinically, adaptations to cervical and proximal margins are difficult to evaluate, especially when finishing line under gingival level is performed. In this case, radiopacity may help in the radiographic follow-up evaluation [5,6].

In order to improve radiographic images, digital imaging has been proven to be an easy and fast resource [7]. Features such as immediate image capture, patients being exposed to low levels of radiation, easy manipulation, low cost, acquisition of accurate radiodensity evaluation, and no need for fill processing, as in the case of traditional images, are highlighted [8]. Another advantage is that in the evaluation of digital images, the radiographic density is easily observable, since the software can determine image pixels and have grayscale values [7,8].

The radiopacity of a material is primarily defined by the chemical elements present in its composition, with zinc, strontium, zirconium, barium, ytterbium, and lanthanum being radiopacifying elements with a high atomic number, present in the constitution of various dental materials, such as contemporary ceramic

systems [3]. Raman spectroscopy allows the analysis and characterization of the vibration spectra, not only of the minerals of which the sample is composed. In this technique, more complex systems, such as dental materials, may also be evaluated by analysis of light diffusion caused by monochromatic laser excitation. They show the different chemical and behavioral patterns of each analyzed ceramic system. This spectroscopy presents several advantages, such as simplicity of sample preparation, facility of analyzing bands, and linear response about mineral and chemical element concentrations [9].

Information on the radiopacity of different ceramics used in restorative dentistry is limited. Consequently, the aim of this study was to compare the radiopacity of different ceramic systems, aluminum step wedge and molar slice (enamel and dentin) by digital radiography and to evaluate the chemical composition of samples using Raman spectroscopy. The study tested the hypothesis that there was a difference in radiopacity between the different tested materials.

## MATERIALS AND METHODS

This study was approved by Human Research Ethics Committee of the Federal University of Juiz de Fora (protocol number: 177/2010).

### *Sample selection and preparation*

Eight ceramic specimens were made for each material: FLD - VM7 (VITA Zahnfabrik, Bad Säckingen, Germany), LD - IPS Empress e.max Press (IPS Empress, Ivoclar Vivadent, Germany), AL - In Ceram Alumina (VITA Zahnfabrik), ALYZ - In Ceram Zirconia (VITA Zahnfabrik), YZ - Lava All Ceram (3M/ESPE, St. Paul, USA), and MYZ - Zirkonzahn (Talladium do Brazil, Curitiba, Brazil) (Table 1).

**Table 1** - Manufacturer name and chemical composition of ceramic system according to groups

Groups	Manufacturer name	Composition
FLD	VM7 (VITA Zahnfabrik, Bad Säckingen, Germany)	Feldspar veneering ceramic SiO <sub>2</sub> (66.8%); Al <sub>2</sub> O <sub>3</sub> (15.6%); K <sub>2</sub> O (10.5%); Na <sub>2</sub> O (3.2%); CaO (2.7%); ZrO <sub>2</sub> (0.8%)
LD	IPS Empress e.max Press (IPS Empress, Ivoclar Vivadent, Germany)	Pressed lithium disilicate SiO <sub>2</sub> (70%); Al <sub>2</sub> O <sub>3</sub> (1.4%); K <sub>2</sub> O (7.9%); ZrO <sub>2</sub> (1.1%); ZnO (12.3%); P <sub>2</sub> O <sub>5</sub> (5.2%); La <sub>2</sub> O <sub>3</sub> (0.6%)
AL	In Ceram Alumina (VITA Zahnfabrik, Bad Säckingen, Germany)	Vitreous ceramic infiltrated by alumina SiO <sub>2</sub> (0.04%); Al <sub>2</sub> O <sub>3</sub> (99.8%)
ALYZ	In Ceram Zirconia (VITA Zahnfabrik, Bad Säckingen, Germany)	Alumina infiltrated vitreous ceramic and zirconia stabilizing particles Al <sub>2</sub> O <sub>3</sub> (64.8%); ZrO <sub>2</sub> (35%)
YZ	Lava All Ceram (3M/ESPE, St Paul, MN, USA)	Milled Y-TZP ceramic ZrO <sub>2</sub> (95%); Y <sub>2</sub> O <sub>3</sub> (3%); HfO <sub>2</sub> (2%)
MYZ	ZirkonZahn (Talladium do Brasil, Curitiba, Brazil)	Stabilized zirconia ceramic ZrO <sub>2</sub> (95%); Y <sub>2</sub> O <sub>3</sub> (3%); HfO <sub>2</sub> (2%)

Ceramic specimens with the same measurements were obtained for all groups (1 x 3 x 3 mm). An acrylic resin pattern was used to produce VM7, In Ceram Alumina and In Ceram Zirconia specimens. These materials were manipulated according to the manufacturers' instructions.

The specimens of VM7 was started by making a refractory model, duplicated from the acrylic resin pattern model. The liquid was mixed to the powder, forming a paste. This was deposited on the surface of the refractory model until it reached the desired thickness. The whole was taken to the oven for burning.

To produce the specimens of In Ceram Alumina and In Ceram Zirconia, it was started by making a refractory model, duplicated from the acrylic resin pattern model. This material is composed of an aqueous suspension of aluminum oxide and/or zirconia particles and a stabilizing agent. A modelable mass was deposited on the surface of this refractory model until it reached the desired thickness. The assembly was baked for initial firing, resulting in a solid phase structure of slightly porous metal oxide particles. On this porous infrastructure was applied the lanthanum glass, which, carried to the second firing, impregnated in the open pores during the first firing.

The ceramic specimens were placed on a refractory base in a Vacumat oven (VITA Zahnfabrik) following firing cycles as recommended by the respective manufacturers.

The specimens were finished with a double face diamond disc (KG Sorensen) and a rubber disc with Supermax diamond paste (Edenta AG Dental, Hauptstrasse, Switzerland) [11]

IPS Empress e.max Press specimens were fabricated using a metal matrix measuring 1 x 3 x 3 mm. The metal matrix (previously lubricated with vaseline petroleum jelly) was placed on a 10 mm-thick glass plate, also lubricated, in order to facilitate removal of the wax models afterwards. The GEO-Classic wax (Renfert GmbH, Hilzingen, Germany) was liquified and poured onto the glass plate until the entire steel matrix cavity surface was covered. After the wax hardened, a straight sharp instrument was used to flatten it and remove excess wax. In the next step, a refractory impression was made by inverting the wax pattern in refractory material. To do this, a wax pattern was connected parallel to a sprue. This connection was made at the edge of the pattern to enable the glass-ceramic material to penetrate into the pattern. The refractory cylinder was put into an electric ring oven EDG 3000 (EDG, São Paulo, Brazil) pre-heated to 700 °C in accordance with the manufacturer's instructions. The next step was the injection process. A glass ceramic ingot of IPS Empress e.max Press was introduced into the central canal of the refractory mold, and then an aluminum cursor (also pre-heated) was inserted into the refractory mold. After this, the refractory mold was transferred to the hot injection chamber. At the end of this process,

the refractory cylinder was removed from the oven and left on the laboratory bench to cool. When it was cold, the body-sprue was divested. The specimens were finished with a double face diamond disc (KG Sorensen) and a rubber disc with Supermax diamond paste (Edenta AG Dental, Hauptstrasse, Switzerland) [11].

To make the specimens of Lava All Ceram and Zirkozahn, pre-sintered blocks of zirconia were used, which were sliced with a diamond disc (KG Sorensen). The blocks underwent an additional sintering process in the oven recommended by the manufacturers at 1500 °C for 4h [12]. Shrinkage occurred in these blocks, after which they measured 1 x 3 x 3 mm. The specimens were finished with a double face diamond disc (KG Sorensen) and a rubber disc with Supermax diamond paste (Edenta AG Dental, Hauptstrasse, Switzerland) [11].

The final specimen thickness was measured with a digital caliper (Digimatic Caliper, Mitutoyo, Aurora, USA) confirming the final thickness of 1 mm.

The radiopacity of the ceramics was compared with that of dental structures (dentin and enamel). For this, a recently extracted inferior first molar of a 25-year-old male was sliced using Labcut 1010 (Excet Corp, Enfield, USA) with a diamond disc. Longitudinal slices 1 mm thick in the more central area of tooth were used.

#### **Radiopacity analysis**

To take the radiographs, a periapical X-ray appliance Gendex Expert DC® (Gendex, Des Plaines, USA) was used, operating at 7 mA, 65 kVp, and exposure time of 0.1 s. The object-sensor distance was kept the same, with the use of a standardized device that provided incidence of the radiation beam perpendicular to the plane in which the sensor and radiographed objects were placed. Radiographic images were obtained using a direct digital radiography apparatus Visualix eHD (Gendex). The following items were put on the sensor: a molar slice, an aluminum step wedge ranging from 1 mm to 11 mm in thickness in steps of 1 mm each, and one specimen of each tested ceramic. Three images were obtained of each radiographed set. They were obtained at a resolution of 1200 dpi, in

TIFF format. No changes in brightness and/or contrast were made.

The radiographic density of the digital images was evaluated using the histogram tool of the Adobe Photoshop® 8.0 software (Adobe, New York, USA). With this software, mean gray values of all steps of the aluminum scale, of the studied specimens, and of the enamel-dentin of the sliced tooth were obtained. The radiopacity of the tested ceramic as well as dental structures were expressed in aluminum-equivalent millimeters (mm Al), allowing comparison among them. The comparison of the radiopacity of the different materials was done by analysis of variance (ANOVA) and a Tukey post-hoc test, with a level of significance of 5%, using BioStat software (Version 5.0, AnalystSoft, Vancouver, Canada).

#### **Raman spectroscopy analysis**

The Raman spectra were obtained in a Horiba Jobin-Yvon LabRam HR spectrometer, coupled with a full petrographic microscope using 10x, 50x, or 100x magnification objectives, and a Peltier-cooled (-70°C) CCD detector. A 10 mW HeNe laser with 632.8 nm wave-length was used, and neutral density filters were selected to adjust the laser power in order to avoid damage and/or transformation of the samples. Laser power over the samples was used at less than 1 mW, to avoid any thermal damage, and each spectrum was obtained at least twice to guarantee wavenumber precision and intensity reproducibility.

## **RESULTS AND DISCUSSION**

In order to compare the radiopacity of ceramic systems with that of human dental structure, the radiopacity of the tested materials, enamel, and dentin were presented in aluminum-equivalent thickness (mm Al). The radiopacity found for 1 mm of aluminum corresponded to the radiopacity of dentin in the same thickness, and for 2 mm corresponded to the radiopacity the enamel. The radiopacity of all the tested materials presented statistically significant differences ( $p < 0.01$ ) except between YZ (Lava) and MYZ (Zirkozahn) (Table 2).

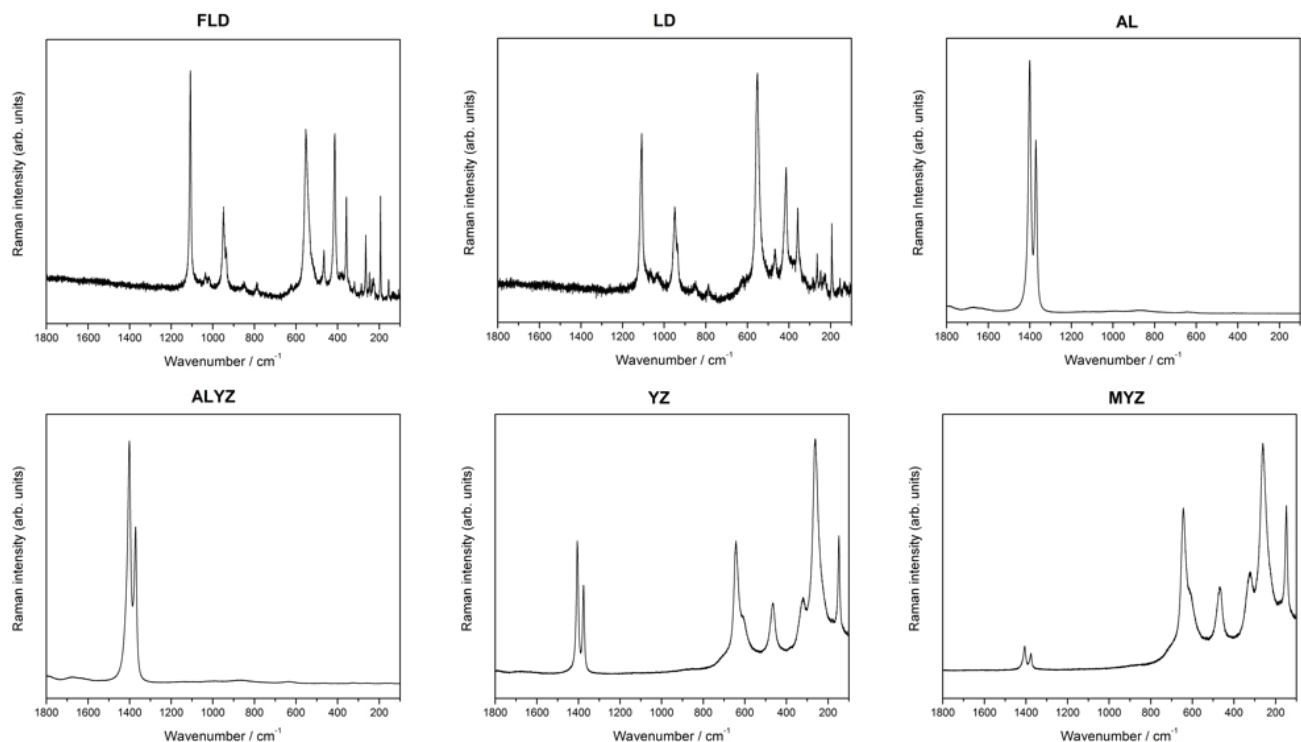
**Table 2** - Mean of radiopacity and thickness equivalent in aluminum for tested ceramic

Groups	Ceramic Systems	Mean of radiopacity	Thickness equivalent in aluminum
FLD	VM7	13.51 <sup>A</sup>	<1 mm Al
LD	IPS Empress e.max Press	22.59 <sup>B</sup>	<1 mm Al
AL	In Ceram Allumina	58.66 <sup>C</sup>	2 mm Al
ALYZ	In Ceram Zirconia	155.03 <sup>D</sup>	9 mm Al
YZ	Lava All Ceram	247.22 <sup>E</sup>	>11 mm Al
MYZ	ZirkonZhan	248.81 <sup>F</sup>	>11 mm Al
-	enamel	65.71 <sup>C</sup>	2 mm Al
-	dentin	44.99 <sup>G</sup>	1 mm Al

\*Followed values for distinct letters present statistical differences

Ceramic systems should have a radiopacity similar to or higher than the aluminum-equivalent thickness in order to enable detection of marginal infiltrations, and cementation failures [3,9]. Aluminum is a comparison reference material because it has a radiopacity similar to that of dentin

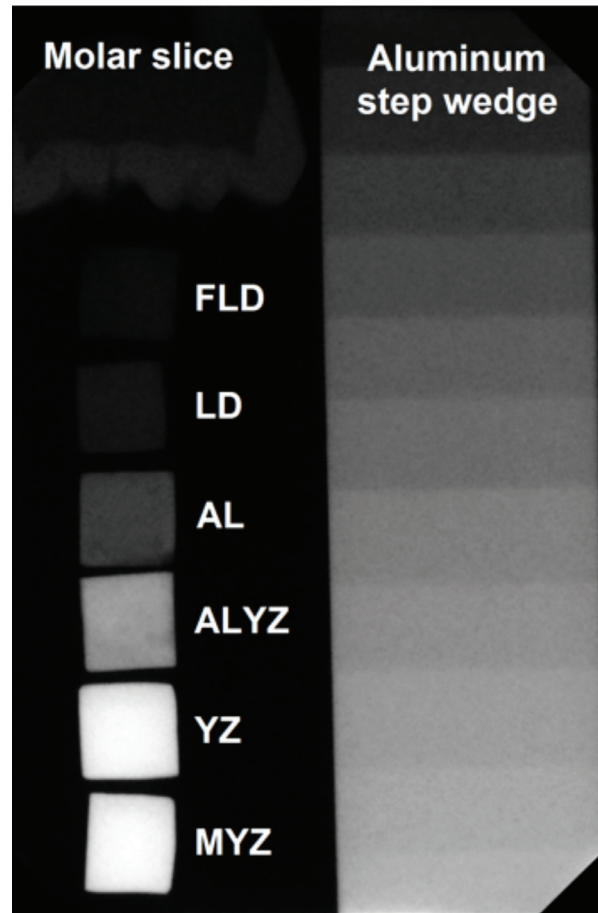
[4], corroborating the results found in this study (Figure 1). The International Standards Organization (ISO 4049) established that the radiopacity of restorative materials should be equal to or greater than that of 1,100 aluminum alloy with the same thickness [13].



**Figure 1** - Radiographic image including specimens of each tested material (FLD, LD, AL, ALYZ, YZ and MYZ), molar slice (enamel and dentin), and aluminum step wedge.

It is undesirable for the radiopacity of restorative materials to be lower than that of the replaced hard dental tissues. Therefore, it is important that dental ceramics, which are used to replace enamel tissue, are more radiopaque than human dental enamel [1,7]. Based on the aluminum scale, an aluminum equivalent of 2 mm corresponds to enamel radiographic density, and 1 mm to dentin radiographic density. The feldspathic ceramic VM7 and IPS e-max Press presented lower density than dentin. The In Ceram Allumina showed the same radiopacity than enamel and all other tested ceramics presented higher density than enamel.

The results support the hypothesis that the radiopacity measurements of ceramic materials differ from one another. Ceramic material radiopacity is directly linked to its chemical composition. This finding was noted in this study, as there was statistical difference in radiopacity between ceramics with different chemical compositions. Only YZ (Lava All Ceram) and MYZ (Zirkonzhan) presented no statistical difference between them, confirmed by Raman spectra vibration bands that did show similar intensity and wave number values. The Raman spectra are represented in Figure 2. They show the different chemical and behavioral patterns of each analyzed ceramic system. This fact confirms that the chemical composition of the two ceramics is very similar. The superior radiopacity of Y-TZP ceramics is probably a result of the high atomic number and molecular weight of yttrium (Y, atomic number 39), zirconium (Zr, atomic number 40), and hafnium (Hf, atomic number 72) [14].



**Figure 2** - Raman Spectra of evaluated ceramic.

Also in relation to Raman spectra, some vibrational modes characteristic of each system were found. For example, it was possible to observe a band at  $1100\text{ cm}^{-1}$  in FLD and LD, which refers to the symmetrical stretching mode for the Si-O bond, while a band at  $580\text{ cm}^{-1}$  corresponds to the symmetrical stretching mode for of Si-O group coupled to the deformation mode of the Si-O-Si structure<sup>15</sup>. For samples AL and ALYZ, the appearance of vibrational bands characteristic of ceramic materials was found to be inhibited by the emission of fluorescence resulting from the high concentration of  $\text{Al}_2\text{O}_3$ , as previously described by GOUADEC, et al. [16]. Bands at  $148\text{ cm}^{-1}$  and  $266\text{ cm}^{-1}$  were found in groups YZ and MYZ, which are vibrations characteristic of the tetragonal phase of  $\text{ZrO}_2$  [17].

The International Standards Organization (ISO 6872) [17] recommends that a spectroscopy test should be used to study the screening of specimen adulteration. This procedure was performed by Raman spectroscopy, which did not detect the presence of any impurity in the chemical composition of each tested ceramic. Furthermore, this test is an excellent tool for the characterization of the various chemical systems present in the composition of each sample, in addition to being a nondestructive analytical technique.

Although manufacturers do not show the quantity of each radiopacifying agent, it was possible to observe statistically significant differences in radiopacity values between materials and their relationships with the atomic number of components. If the atomic number is high, the material is more radiopaque [3,18]. Corroborating the findings of OZKURT, et al. [18] and PEKKAN, et al. [19], this study showed that zirconia has the highest radiopacity level. These higher levels are useful to monitor marginal adaptation by radiographic examination [6,20]. But these materials, when used in the clinical practice, may mask cement dissolutions and caries, which could occur under the crown [20].

The difference found in the radiopacity values is of clinical significance in radiographic evaluation. Zirconia-based materials are more radiopaque and are easier to detect in a radiographic examination. On the other hand, less radiopaque materials, such as silica-based vitreous ceramics, may be mistaken for dental structures, carious lesions, restoration failures and marginal defects [3]. The maximum radiopacity value has not yet been established. However, extremely radiopaque materials may be difficult to identify at the tooth/restoration interface. Moderate radiopacity may be more favorable and could facilitate the detection of tooth/restoration interface [3,4,21].

The adequate radiopacity must be accepted as a factor when evaluating the clinical success of ceramic restorations. Furthermore, studies are needed to compare the radiopacity of the adhesive cements used with the different ceramics systems.

## CONCLUSION

Radiopacity is closely linked to the chemical composition of each ceramic system. The groups containing zirconia demonstrated more radiopacity. The feldspathic ceramic VM7 and the vitroc ceramic of disilicate of lithium IPS Empress e.max Press presented lower radiopacity than that of human dental structures.

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