

## *Evaluation of resin composite staining by beverages with acid pH*

## *Avaliação do manchamento de resinas compostas por bebidas de pH ácido*

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### ABSTRACT

**Introduction:** Composite resins are an extensively used esthetic material that has excellent adaptation, reproduces the characteristics of the tooth structure and is suitable for the conditions of the oral environment. However, this material is also very susceptible to staining due to pigmentation coming from the diet and habits of the patient. **Objectives:** To evaluate the staining of two brands of composite resin, one nanoparticle and a nanohybrid type (Filtek Z350XT® and IPS Empress Direct®), in direct contact with the acid pH of beverages. **Methods:** In total 40 specimens were made, 20 of each type of resin studied. After being inserted into a Teflon matrix and light activated, the samples were removed, stored in containers with 2 ml of artificial saliva and kept in the refrigerator. Every day 20 of these samples were placed in direct contact with 2 ml Gatorade, and the other 20 samples, in 2 ml of wine, each set for 30 minutes. After 30 minutes, the acid pH substances were removed, and 2 ml of artificial saliva was again placed in the container. This process was repeated every day for 10 days. **Results:** The results showed that the nanoparticle resin (Filtek Z350XT) was more susceptible to staining than the nanohybrid type (Empress Direct®) when in contact with the wine. When wine and Gatorade were compared, the solution with higher staining capacity was shown to be wine. **Conclusion:** The two resins tested were susceptible to staining in a 10-day period. Mottling and more staining was visibly detected after samples were immersed in the solution with the higher pH, and nanoparticulate resin was more susceptible to staining.

**Indexing terms:** Coloring agents. Composite resins. Esthetics, dental.

### RESUMO

**Introdução:** As resinas compostas são altamente utilizadas por serem um material estético, com excelente adaptação, reproduz as características da estrutura dental e se adequa ao meio bucal. Porém, este material é muito susceptível à pigmentação por corantes

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vindos da dieta e de hábitos do paciente. **Objetivos:** Avaliar o manchamento de duas marcas comerciais de resina composta: Nanoparticulada e Nanohíbrida, em contato direto com bebidas de pH ácido (Filtek Z350XT® e IPS Empress Direct®). **Métodos:** Foram confeccionados 40 corpos de prova, 20 de cada tipo de resina estudada, depois de inseridas em matriz de teflon e fotoativadas, os corpos de prova foram removidos, armazenados em recipientes com 2ml de saliva artificial e mantidos na geladeira. Diariamente 20 dos mesmos eram colocados em contato direto com 20mL Gatorade e os outros 20mL de vinho durante 30 minutos cada. Passados os 30 minutos, as substâncias de pH ácido eram retiradas e novamente, 2 mL de saliva artificial eram colocadas no recipiente. Este processo foi repetido todos os dias durante 10 dias. **Resultado:** Os resultados mostraram que a resina nanoparticulada (Filtek Z350XT®) é mais susceptível ao manchamento que a nanohíbrida (Empress Direct®) quando em contato com o vinho. Quando comparados vinho e Gatorade, a solução com maior teor de manchamento é o vinho. **Conclusão:** As duas resinas testadas foram sensíveis ao manchamento num período de 10 dias, esse manchamento foi visivelmente detectado, a solução com o pH mais elevado manchou mais e a resina nanoparticulada é mais susceptível ao manchamento.

**Termos de indexação:** Corantes. Resinas compostas. Estética dentária.

## INTRODUCTION

Resin materials appeared in the 1970s, and up to now have undergone constant reformulations in an endeavor to enhance their physical, chemical and mechanical properties [1]. As the years went by, these resins have undergone a constant evolution, and the main change in their development is related to the type and size of the filler particles of which they are composed. These have led to the resins such as the nanohybrid and nanoparticle types being widely used in dental restorative procedures [2].

Composite resins are among the dental restorative materials chosen by many dentists because they are well accepted by patients and because of their capacity for bonding to the dental structures, excellent esthetic properties, favorable mechanical properties, relatively low cost (in comparison with ceramic) and application in both the anterior and posterior regions of the dental arches. However, as these resins age, significant changes in their properties may occur [3,4].

The discoloration of these restorative materials after extensive exposure to the oral environment, may be one of the most significant changes that could occur. This is a great disadvantage considering that one of the most important clinical references for the success of an esthetic restoration is with respect to color stability [1].

Discoloration of restorations may be due to extrinsic (exogenous) or intrinsic (endogenous) causes. Intrinsic factors that involve discoloration may occur due to change in the resin matrix and at the interface of the resin matrix and interface of the filler particle load. Extrinsic factors may also be responsible for resin composite staining, particularly a diet rich in acids that promote wear of the composite. Thus the occurrence of pigment penetration occurs, causing staining and interfering in the esthetics of the treatment. The acids that cause these stains are present in soft drinks and citric fruit juices [5].

The low pH value affects the resistance of restorative materials in terms of interaction with the filler particles and in terms of decomposition of the matrix. Foods with an acid pH may trigger erosive processes on the tooth surface and on the surfaces of resin composite restorations [6].

Due to adsorption and absorption of liquids by resin composite, with consequent retention of pigments, their color stability had been jeopardized, particularly by beverages with an acid pH. Resins absorb water, ions and molecules of the pigments become adhered to the resin surface causing staining. Among the beverages with higher potential for pigmentation, the most outstanding type is red wine with a pH of 3.5, followed by cola-based soft drinks with pH 3.0 and coffee pH 7.0 [7,8].

The aim of this study was to evaluate the staining potential of two resin composites - a nanohybrid and the other, a nanoparticulate type, after contact with beverages that have an acid pH.

## METHODS

### Experimental design

The factors under study were the nanoparticulate resin composite Filtek Z350XT® (3M ESPE, St Paul, MN, USA) and nanohybrid resin composite IPS Empress Direct (Ivoclar Vivadente®), both in shade A2. The experimental units were 40 cylindrical test specimens and the design of treatment of these units was a random process. The polymerization method was use of a fourth generation LED light appliance Polywave Blue Phase, with polymerization performed for a time of 20 seconds of light activation. The response variable was color instability of the samples before and after having been immersed in beverages with an acid pH (Gatorade and Red Wine).

### Fabrication of test specimens

The composition and characteristics of the resin composites used in this study are described in chart 1.

**Chart 1.** Commercial brand, composition of composites tested and manufacturers.

Material	Composition	Manufacturer
Z350XT	bis-GMA, UDMA, TEGDMA, and bis-EMA. To reduce polymerization shrinkage, PEGDMA was replaced by a portion of TEGDMA in the Filtek Supreme XT resin. The particles are a combination of silica with a size of 20nm non-agglomerated/non-aggregated, zirconia with a size of 4-11nm non-agglomerated/-aggregated, and agglomerated, clusters, aggregated particles of zirconia/silica (combination of silica particles of 20nm and Zirconia of 4-11nm). The colors of Dentin, Enamel and Body (DEB) have a mean particle size of the agglomerate of approximately 0.6-10 microns. The translucent shades (T) have a mean particle size of the agglomerate of 0.6-20 microns. The inorganic filler particles represent approximately 72.5% by weight (55.5% by volume) for Translucent colors and 78.5% by weight (63.3% by volume) for all the other colors.	3M ESPE, St Paul, MN, USA
IPS Empress Direct	The monomer matrix is composed of dimethacrylates (20-21.5 % by weight, opalescent color 17 % by weight). The particles are constituted of barium glass, ytterbium trifluoride, mixed oxides, silicon dioxide and copolymer (77.5-79 % by weight, opalescent color 83 % by weight). Additives, catalyzer, stabilizers and pigments (< 1.0 % by weight) are additional components. The total inorganic filler content is 75-79 % by weight or 52-59 % by volume (opalescent color 60.5% by weight or 45 % by volume). The size of inorganic particles is situated between 40 nm and 3000 nm, with a mean particle size of 550 nm. Its photoinitiator contains Camphorquinone.	Ivoclar, Vivadent

The test specimens were fabricated by a single operator, in accordance with the specifications of ISO 4049, under the same conditions of temperature, illumination and relative humidity of the air. They were obtained by using a cylindrical Teflon matrix 2mm thick with an internal diameter of 5mm. In total 40 test specimens (n=10) were fabricated; that is 10 samples for each resin tested were used as positive control (chart 2).

A polyester strip (Dentsply, Petrópolis, Rio de Janeiro, Brazil), and a Teflon matrix were placed on a glass slide. Shade A2D was standardized for the two types of resin. The resin composites were inserted into the matrix with the aid of a titanium spatula, in a single increment, and accommodated so that there would be no excess material.

**Chart 2.** Division of study groups.

Group	Description
G1	Z350XT pigmented (red wine)
G2	Z350XT pigmented (isotonic)
G3	IPS Empress pigmented (red wine)
G4	IPS Empress pigmented (isotonic)

Teflon matrices filled with the Filtek Z350XT and IPS Empress Direct resins, respectively, in a single increment, were placed on glass slides, then the polyester strip was placed on top of each matrix that had been filled, for the purpose of obtaining a smooth level surface and a standardized thickness. Light activation was performed for 20 seconds, using the fourth generation LED light appliance - Bluephase 4. After light polymerization, the test specimens were removed from the matrices, and excess resins were removed. This procedure was repeated 40 times until 40 test specimens had been obtained.

**Chart 3.** LED Light Activation Appliance, description, power density and manufacturer.

Material	Description	Power Density	Manufacturer
Bluephase	LED POLIWAVE Light Activation Appliance, cordless, operates with Lithium battery. Does not use conductor tip; Has black light output point 10 mm in diameter. Fourth generation LED	1400 mW / cm <sup>2</sup>	Ivoclar / Vivadent

## Staining of samples and color readout

### Staining of samples

Before the staining procedures, an initial color readout of the samples was made. The staining protocol was based on a pilot test, which was performed as follows: for 10 days, 20 test specimens, 10 of Filtek Z350XT resin and 10 of IPS Empress Direct resin were immersed in 2ml of Grape Flavor Gatorade for 30 min. Right after this time elapsed, the substance with acid pH was removed and 2ml of artificial saliva was put in its place. The composite remained in this medium until the same time on the next day. Of the remaining 20 test specimens, 10 of Filtek Z350XT resin and 10 of IPS Empress Direct resin were immersed in 2ml of red wine, brand Gato Negro, and exactly the same procedure was followed as that performed with the test specimens tested in Gatorade.

### Color readout

Initially, the 40 test specimens were submitted to color assessment by means of a Vita EasyShade spectrophotometer and the data were interpreted by the OnColor Lite software coupled to the appliance. The spectrophotometer was calibrated according to the manufacturers' recommendations.

### Color change analysis ( $\Delta E$ )

The color stability was analyzed by spectrophotometer and calculated by the difference ( $\Delta E$ ) between the coordinates obtained from the samples before and after the staining procedure. The total change in color  $\Delta E$ , is commonly used to represent a difference in color and is calculated by using the formula:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

The classification adopted for the  $\Delta E$  values was that determined by the National Bureau of Standards (NBS), which considers values from 0.0 to 0.5 – extremely slight alteration; 0.5 to 1.5 – slight alteration; 1.5 to 3.0 – perceptible alteration; 3.0 to 6.0 – significant alteration; 6.0 to 12.0 – extremely significant alteration; 12.0 or higher – change to another color [4,6].

## RESULTS

### Statistical analysis

After exploratory analysis of the data, the factorial analysis of variance (ANOVA) was applied to the color stability test ( $\Delta E$ ) and multiple comparisons were made by the Tukey test. In all analysis a significance level of 5% was considered.

### Color Stability

Table 1 presents the means and standard deviations of the color stability values ( $\Delta E$ ), considering the resins tested and acid staining solutions. In this case statistically significant difference was observed between the resins tested and solutions tested ( $p < 0.05$ ). The results showed that for the IPS Empress Direct resin there was no statistically significant difference in relation to the staining solutions ( $p = 0.003$ ). However, for the Z350 XT resin, the red wine solution demonstrated a higher staining power in comparison with the Gatorade solution. Another significant observation was that the Z350 XT resin showed significant staining in comparison with the IPS Empress Direct resin, only when in contact with the wine solution.

**Table 1.**  $\Delta E$  - Mean and standard deviation considering type of composite.

Resin	Staining solution	
	Grape Isotonic beverage	Red Wine
IPS Empress Direct	7.54( 2.00) Aa	10.38( 3.90) Aa
Z350XT	9.20 (1.74) Ab	13.65 (2.08) Bb

Note: Means followed by equal letters, (capitals in the horizontal and lower case in the vertical) do not differ among them ( $p \leq 0.05$ ).

## DISCUSSION

The fast restorative treatment together with low cost and capacity for obtaining esthetic results have made composite resin restorations the first restorative option and a reality in the Brazil of the 21st Century, where the population places increasing value on esthetics. Even the contemporary resin composites show change in color, particularly when the patient has poor oral hygiene practices, and by the impregnation of dyes contained in foods [9].

The chemical evolution has brought about improvements in polymerization shrinkage, mechanical strength and chemical stability, and therefore, resin composite has been the esthetic material most frequently used. It has been affirmed that the biphasic composition of resin composites, type of pigmentation agent, and time of immersion determined staining [10].

With regard to extrinsic factors responsible for staining resin composites, the primordial role of diet has been pointed out to be the most common source of acids that promote superficial degradation of restorative materials. Thereby, the penetration of pigments and subsequent staining occur, leading to esthetic failure of the treatment [11].

Nanoparticle resins have been developed by nanotechnology. This technology has allowed the production of materials and structures with dimensions ranging between 0.1 and 100 nanometers; that is to say, approximately ten times smaller size than the particles used in microparticulate resin composites [1,5]. There is also a difference with reference to the percentage concentration of filler particles. These resins may have a mechanical strength similar to that of microhybrid resins, added to the esthetic advantages of microparticulate resins. Nanoparticulate resins may show a higher level of degradation in the oral environment than the hybrid types [12]. Nanohybrid resins contain glass particles

with a mean size smaller than those of microhybrid resins. This guarantees excellent polishing of these resins and good mechanical properties [13].

In this study, the nanoparticle resin composite Filtek Z350XT and the nanohybrid IPS Empress Direct, had some differences in their compositions, which were determinant for the result since statistical difference in the degree of staining of the resins was observed.

The color of our test specimens was verified by using the spectrophotometer, which eliminated subjectivity and possible errors of individual interpretation. This appliance is capable of determining color in numerical terms. In the present research the VITA Easyshade® spectrophotometer was used.

In this study, the value of  $\Delta E$  revealed that the differences between initial and final colors were clinically visible. The samples, particularly those pigmented by red wine, had a color that tended towards being brown.

In this research, we verified that the nanoparticle resins tended to stain to a larger extent than the nanohybrid type. Moreover, between the beverages used, wine stained to a greater extent than Gatorade, possibly due to its coloring and effect of fermentation that results in the production of acids capable of changing the strength of the resins, thereby increasing the possibility of pigment penetration. The test specimens were prepared and did not receive any type of polishing or finishing, clinical procedures that notoriously favor superficial smoothness and consequently, reduce staining [10,12,13]

The test specimens were evaluated in a period of 10 days and obtained a clinically visible result by using only the red wine Gato Negro, grape flavored isotonic beverage and artificial saliva, in which the samples remained stored throughout the process.

## CONCLUSION

In view of the results of this study, it could be concluded that the Z350XT resin stained to a larger extent than the IPS Empress Direct resin, when in contact with red wine. Irrespective of the resins tested, both types were stained to a larger extent by the red wine than by the grape flavored isotonic beverage. Therefore, in this study, the nanoparticulate resin was more susceptible to staining than the nanohybrid type.

## Collaborators

RA Vieira, research, methodology, written. IHP Vieira and LC Prata, research, methodology. WDA Vieira, research, written. DA Miranda, research, methodology, written, supervision.

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