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Campus de São José dos Campos

ORIGINAL ARTICLE
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EDS analysis of gutta-percha cones disinfected by 1% and 2.5% sodium hypochlorite solutions

Análises de EDS dos cones de guta percha desinfetados por soluções de hipoclorito de sódio 1% e 2,5%

Alessandra Sverberi CARVALHO[1], Flávia Martins LEAL[1], Rafaela Andrade VASCONCELOS[1], Rafael Binato JUNQUEIRA[1], Simone Helena Ferreira GONÇALVES[2]

1 – Department of Restorative Dentistry – School of Dentistry – Institute of Science and Technology – UNESP – Univ Estadual Paulista – São José dos Campos – SP – Brazil.

2 – Department of Bioscience and Oral Diagnosis – School of Dentistry – Institute of Science and Technology – UNESP – Univ Estadual Paulista – São José dos Campos – SP – Brazil.

ABSTRACT

Objective: To evaluate the effects of 1% and 2.5% sodium hypochlorite (NaOCl) solutions at different periods on the composition of gutta-percha cones using Energy Dispersive Spectroscopy (EDS). **Material and Methods:** 110 master gutta-percha points were divided into 3 control groups and 8 experimental groups. Cones were immersed in 1% and 2.5% NaOCl for 30 min, 6, 12, 24 h. Then, composition of the samples was analyzed by EDS. Statistical analysis was performed using the ANOVA and Tukey tests ($p < 0.05$). **Results:** There was an increase of oxygen in experimental groups, and decrease of zinc when NaOCl was used, concentration and time-dependent (Critical comparison value: Oxygen = 9.21; Zinc = 15.19). **Conclusion:** The disinfection with NaOCl solution causes alterations in the composition of gutta-percha, which were concentration and time-dependent.

KEYWORDS

Disinfection; Endodontics; Gutta-percha; Sodium hypochlorite.

RESUMO

Objetivo: Avaliar o efeito das soluções de hipoclorito de sódio (NaOCl) 1% e 2,5% em diferentes períodos de tempos na composição dos cones de guta percha usando estereomiscroscopio de energia dispersiva (EDS). **Material and Metodos:** Cento e dez cones principais de guta percha foram divididos em 3 grupos controles e 8 grupos experimentais. Os cones foram imersos em solução de NaOCl 1% e 2,5% por 30 min, 6 h, 12 h e 24 h. Depois do tempo de imersão, a composição química das amostras foram analisadas por EDS. A análise estatística foi realizada utilizando ANOVA, e os testes de Tukey e Dunnett ($p < 0,05$). **Resultados:** Um aumento de peso (%) do oxigênio e diminuição do peso (%) de zinco foram observados nos grupos experimentais dependendo da concentração e tempo de NaOCl utilizados (Valor crítico de comparação: Oxigênio = 9,21; Zinco = 15,19). **Conclusão:** A desinfecção com soluções de NaOCl causam alterações na composição dos cones de guta percha, sendo tais alterações concentração e tempo dependentes.

PALAVRAS-CHAVE

Desinfecção; Endodontia; Guta percha; Hipoclorito de sódio.

INTRODUCTION

One of the major objectives of endodontic treatment is elimination of microorganisms found in the root canal system [1,2]. Root canal filling is an important procedure for preventing a root canal reinfection [3]. Gutta-percha cones are the most commonly used material for obturation of the root canal system [2,4-6].

Gutta-percha is composed of organic (gutta-percha polymer and wax/resins) and inorganic components (zinc oxide, barium sulfate) [5-9]. Colouring agents and antioxidants can also be present in small percentages.

Although gutta-percha cones are produced under aseptic conditions, they can be contaminated by handling, aerossols and during storage [2,4,6,10,11]. Because of their thermoplastic characteristics, they cannot be sterilized by the conventional processes using moist or dry heat, which may cause alterations in their structure, representing a great potential for failure of the endodontic obturation [2,3,5,10,12,13]. Obturating with a contaminated material could reintroduce microorganisms to the root canal system causing persistent infection by delaying or inhibiting healing [14,15]. Therefore, a rapid chair-side chemical disinfection is needed [1,5].

Several chemical solutions have been used for such disinfection [6,10]. Sodium hypochlorite (NaOCl) is one of the most widely used endodontic solution, being used both as an irrigant in the root canal, and for cone disinfection [5,6].

Sodium hypochlorite solution is a strong oxidizing agent that can cause structural deformations to a wide range of materials [1,6,16]. Therefore, potential adverse changes to gutta-percha cone structure after NaOCl solution exposure at any concentration needs to be studied [9].

Commonly, clinicians keep the gutta-percha points more time than necessary for disinfection in NaOCl solution. That way, the aim of this study was to evaluate the effects of 1% and 2.5% sodium hypochlorite solutions at different periods on the composition of gutta-percha cones using Energy Dispersive Spectroscopy (EDS).

The null hypothesis of this study was the overtime immersion of gutta-percha in NaOCl solution disinfection with different concentrations and at different times does not influence at gutta-percha composition structure.

MATERIAL & METHODS

A hundred and ten master gutta-percha points taper 30 (Dentsply Maillefer, Petrópolis, RJ, Brazil) were divided into 3 control groups (GC1, GC2 and GC3) and 8 experimental groups, according to the sodium hypochlorite concentrations used and the time of immersion in selected solutions (Table 1).

Table 1 - Division of experimental groups according to the concentration of NaOCl and the time of gutta-percha points disinfection (G1A, G2A, G3A, G4A, G1B, G2B, G3B, G4B)

Concentration / Disinfection time	A- NaOCl 1%	B- NaOCl 2.5%
G1	30 min	30 min
G2	6 h	6 h
G3	12 h	12 h
G4	24 h	24 h

Control groups were divided as follows: control group 1 (GC1) - Without disinfection of cones, control group 2 (CG2) - Immersion point in sodium hypochlorite 1% for 20 min, and control group 3 (CG3) - Immersion point in sodium hypochlorite 2.5% for 10 min.

For each group, 10 gutta-percha points (n = 10) were used, and accommodated in disposable Petri dishes. The 4 mm from the cervical portion of the cones were sectioned, to prevent an interference of the gutta-percha marker dye in the reading of the chemical components. With the exception of one control group (CG1) that suffered no disinfection, all other gutta-percha specimens remained immersed in 10 ml of the basic substance sodium hypochlorite by varying the concentration and time of disinfection.

After each period of the groups, the gutta-percha points were removed from solutions, rinsed with distilled water, placed in other dry Petri and lined with filter paper. The plates were closed and taken for analysis the next treatment day.

Energy Dispersive Spectroscopy (EDS) was selected to perform gutta-percha points chemical analysis. Scanning microscope (JSM 840[a] Jeol, Tokyo, Japan) was positioned to read at a distance of approximately 25 mm from specimens to be analyzed and an area of 0.5 mm2 was irradiated with a voltage of 20 kV for 100 s, providing an electron beam penetration to a depth of 1 μm. EDS quantified chemical elements concentration found in most of the specimens tested, as follows: carbon (C), oxygen (O), aluminum (Al), sulfur (S), phosphorus (P), zinc (Zn), barium (Ba), silicon (Si), sodium (Na) and chlorine (Cl). Through readings performed, gains or losses gutta-percha points structural suffered after disinfection processes were observed.

The results obtained were statistically analyzed by using ANOVA, Tukey test to compare only the control groups and Dunnett test to compare all experiments groups with the control group (GC1), with a significance level of 5% ($p < 0.05$).

RESULTS

Analyzing the components of gutta-percha cones in control groups, only carbon, oxygen and zinc revealed considerable change in concentration.

The oxygen concentration has increased when the cones were immersed in 2.5% sodium hypochlorite for 10 min (GC3), differing significantly from the group where the 1% solution was used for 20 min (GC2). When comparing to GC1, no significant difference was observed. Zinc concentration significantly increased in groups GC2 and GC3 compared to the group not immersed in the hypochlorite solution. For carbon, there was a significant decrease in the levels of this element after its immersion in 1% hypochlorite for 10 min (GC2). However, when was used disinfection with hypochlorite at 2.5% for 20 min (GC3), the carbon concentration did not differ significantly in relation to the other control groups (Table 2).

Table 2 - Comparison of chemical components weight (%) between control groups

Mean (±SD)			
Control Groups	Carbon (%)	Zinc (%)	Oxygen (%)
GC1	31.07 (2.32) A	43.22 (5.32) B	25.31 (3.64) AB
GC2	28.12 (1.86) AB	50.21 (3.41)A	21.12 (2.60) B
GC3	28.98 (1.97) B	43.40 (7.73) B	27.00 (5.84) A

Different letters (A, B) mean significant difference at 5% level by Tukey test.

When comparing the experimental groups with the control group without immersion in sodium hypochlorite solution (GC1), oxygen levels have changed significantly in all groups immersed in the tested solutions, irrespective of time and concentration. In relation to zinc, there was a significant reduction in their concentrations when used hypochlorite at 1% for 12 h and 24 h (G3A and G4A) and at 2.5% for 6 h, 12 h and 14 h (G2B, G3B, G4B) In the other groups, the variation of concentration of this element was not significant (Table 3).

Table 3 - Comparison of chemical components weight (%) between GC1 and experimental groups

	OXYGEN (%)	ZINC (%)
Groups	Mean (±SD)	Mean (±SD)
GC1	25.31 (3.64) *	43.22
G1A	14.20 (1.46) *	49.20
G2A	36.64 (15.83) *	36.97
G3A	56.26 (5.62) *	9.37 *
G4A	38.68 (3.41) *	2.62 *
G1B	13.55 (1.68) *	49.54
G2B	42.31 (13.59) *	29.25*
G3B	42.20 (3.74) *	9.65 *
G4B	39.70 (2.96) *	20.03 *

Critical comparison value: Oxygen = 9.21; Zinc = 15.19.
*Significant difference at 5% by Dunnett's test.

DISCUSION

According to the results the null hypothesis was rejected because the time and the concentration of NaOCl solution affected the gutta-percha composition structure

The aseptic chain must be installed and maintained in the different phases of endodontic therapy, an essential factor for success of root canal system treatment. Gutta-percha is the most frequent solid material used for filling of root canals and, due to its constitution it doesn't resist the conventional methods of sterilization by moist or dry heat [3].

Some authors stated that disinfection of gutta-percha before use at the time of filling is unnecessary, once it should be free from microorganisms in the original packaging. Also there is a possible antimicrobial activity of cones attributed to the zinc oxide and the recognized antiseptic action of endodontic sealer [8,17,18].

However, it is known that the cones may be contaminated when sealed in their packages [19], and commercial presentation in boxes with multiple units may further contamination during handling in clinical practice [20,21]. The negligence in maintaining aseptic chain may be related to persistence of infection, preventing the repair of periapical tissues. Thus, the absence of microorganisms on the surface of the cones is important, since they remain in the apical third near to the periradicular tissues [13].

Several chemical agents are described in the literature with the aim to promote cones disinfection [12,14,15]. In the present study the sodium hypochlorite was used in different concentrations because it is considered a standard solution. Nevertheless, previous studies have related that it causes structural and physical changes [1,3,6,9,11,16].

NaOCl has been used for this purpose because of its important properties of antimicrobial effect [2,3,4]. However, NaOCl led to changes in the composition of the cones.

Zinc and oxygen are usually present in large amounts on gutta-percha, since zinc oxide is the main component [7]. In this study, the oxygen amount increased when NaOCl was used in the disinfection, proving the great oxidative potential of the solution. EDS also revealed a significant reduction of zinc when NaOCl was employed in cone disinfection.

The deterioration of gutta-percha cone can occur after NaOCl disinfection, this happens because of loss of gutta-percha cone components by the oxidizing agent, NaOCl [16]. Some changes can be noticed as brittleness, stiffness, tensile strength, radiopacity, flow, plasticity, elongation, inherent tension force and thermal behavior [22,23]. Moreover, cones become more rigid with high concentrations of inorganic components and low percentage of gutta-percha.

The alterations of gutta-percha cones can increase the risk of leakage once create large interfacial gaps between the gutta-percha cone and the root canal wall [24]. NaOCl produces a large quantity of chloride crystals on the cone surface increasing depth of surface irregularities and loss of elasticity, which could make it difficult to achieve appropriate sealing [16].

In conclusion, the disinfection with NaOCl solution causes alterations in the composition of gutta-percha cones, which were concentration and time-dependent.

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Simone Helena Ferreira Gonçalves
(Corresponding address)
School of Dentistry – Institute of Science and Technology – UNESP
Av. Eng. Francisco José Longo, nº 777
Jardim São Dimas
12245-000 - São José dos Campos, SP
Email: simone@fosjc.unesp.br

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