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Original
Effect of sterilization treatment on the corrosion behavior of NiTi endodontic instruments / Rosalbino, F., Casella, G.. - ELETTRONICO. - (2023), pp. 1-9. [10.5281/zenodo.8164686]

Availability:
This version is available at: 11583/2980533 since: 2023-11-30T20:38:06Z

Publisher:

Published
DOI:10.5281/zenodo.8164686

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19 November 2026

Effect of sterilization treatment on the corrosion behavior of NiTi endodontic instruments

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Abstract

This study investigates the influence of the sterilization treatment conducted at 50 °C in 5% sodium hypochlorite solution (NaClO) on the corrosion behavior of endodontic instruments made of NiTi alloy (GT Rotary and K3) and stainless steel (K File) by measuring potentiodynamic polarization curves in the same environment at 37°C. For the sake of comparison, the electrochemical characterization was also carried out on endodontic instruments that were not sterilized. The characterization of the sample surface after the electrochemical tests were carriedout by means of scanning electron microscopy (SEM) coupled with X-ray energy dispersive spectroscopy (EDX). There was no significant influence of the sterilization treatment on the corrosion behavior of K File and GT Rotary endodontic instruments. On the contrary, a negative influence of the sterilization treatment on the corrosion resistance of K3 endodontic instruments was observed, and the effect appears to be more dramatic for longer treatment periods.

Keywords: NiTi endodontic files; Corrosion behaviour; Sodium hypochlorite; Potentiodynamic polarization curves

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1. Introduction

Rotating endodontic instruments composed of nickel–titanium alloy risk fracture during the shaping of root canals. Generally, the fracture of NiTi endodontic instruments occurs through two mechanisms: torsion or flexion. Fracture through torsion occurs when the point of the file or a part of the instrument remains in the root canal while the handle continues to rotate [1, 2, 3]. When this happens, the elastic limitation of the alloy is reached; the metal is deformed and then cracks. The other type of crack occurs though flexion, and is caused by stress and fatigue in the metal (cyclic fatigue) [4, 5]. Following Novoa et al., 2007 [6], we investigate the phenomenon of corrosion in endodontic instruments, which presents as pitting corrosion followed by a weakening of the structure of these instruments. Several studies [7, 8] have shown that corrosion of the endodontic instruments can degrade the mechanical properties and suddenly cause undesirable cracks during root canals. Topuz et al., 2008 [9] have investigated the corrosive effects on NiTi instruments using electron microscopy and have determined that this corrosion is inevitable. Many studies have tested the corrosion resistance of NiTi alloys in different solutions [6, 8, 9, 10], but no studies have been published using potentiodynamic polarization curves to understand corrosion. It is important to identify the mechanisms of corrosion in order to understand the biocompatibility of endodontic files in practical use and to provide a guide for clinical applications of NiTi alloys.

The aim of the present work is to investigate the influence of sterilization treatment in sodium hypochlorite on the corrosion behavior of endodontic files composed of NiTi and stainless steel (AISI 316). This was done by recording potentiodynamic polarization curves in 5% sodium hypochlorite solution. For the sake of comparison, the electrochemical characterization was also performed on instruments that were not sterilized. The characterization of the surface of samples after electrochemical testing was carried out by means of scanning electron microscopy equipped with EDX microanalysis.

2. Experimental

Two different types of endodontic instruments were tested, one Ni-Ti, GT Rotary and K3 instrument (diameter in point .25, taper .06), and one stainless steel K File instrument (diameter .25, taper .2). All instruments were submitted to sterilization treatment by immersion in 5% sodium hypochlorite (NaClO) solution for 5 and 10 min in special glass containers. Only the working part of each instrument was subjected to the electrochemical characterization.

The corrosion behavior was assessed by recording potentiodynamic polarization curves in 5% NaClO aerated solution at 37°C with a scan rate of dE/dt = 1 mV/s, using a saturated calomel electrode (SCE) as reference. For the sake of comparison, the same characterization was carried out on non-sterilized base materials.

The surface morphology of the samples after electrochemical testing was characterized using scanning electron microscopy (SEM) coupled with energy dispersion X-ray spectroscopy (EDX) microanalysis.

3. Results and discussion

Figure 1 shows the potentiodynamic polarization curves recorded in 5% NaClO solution for the stainless steel K File instruments. The sterilization treatment causes a shift of the corrosion potential toward more negative values (base material: ≈ – 110 mV/SCE; 5 min sterilized material: ≈ – 200 mV/SCE; 10 min sterilized material: ≈ – 300 mV/SCE). Nevertheless, comparing the anodic branches of the polarization curves indicates no significant change between the base and sterilized materials. In fact, the sterilized materials exhibit current density values comparable to, if not lower than, those of the base material. A similar behavior is observed for the NiTi GT Rotary instruments, as shown in Figure 2. By comparing the anodic branches of the polarization curves, no significant change between the base material and the sterilized samples is observed. In fact, the sterilized materials exhibit corrosion potential and current density values similar to those of the base material.

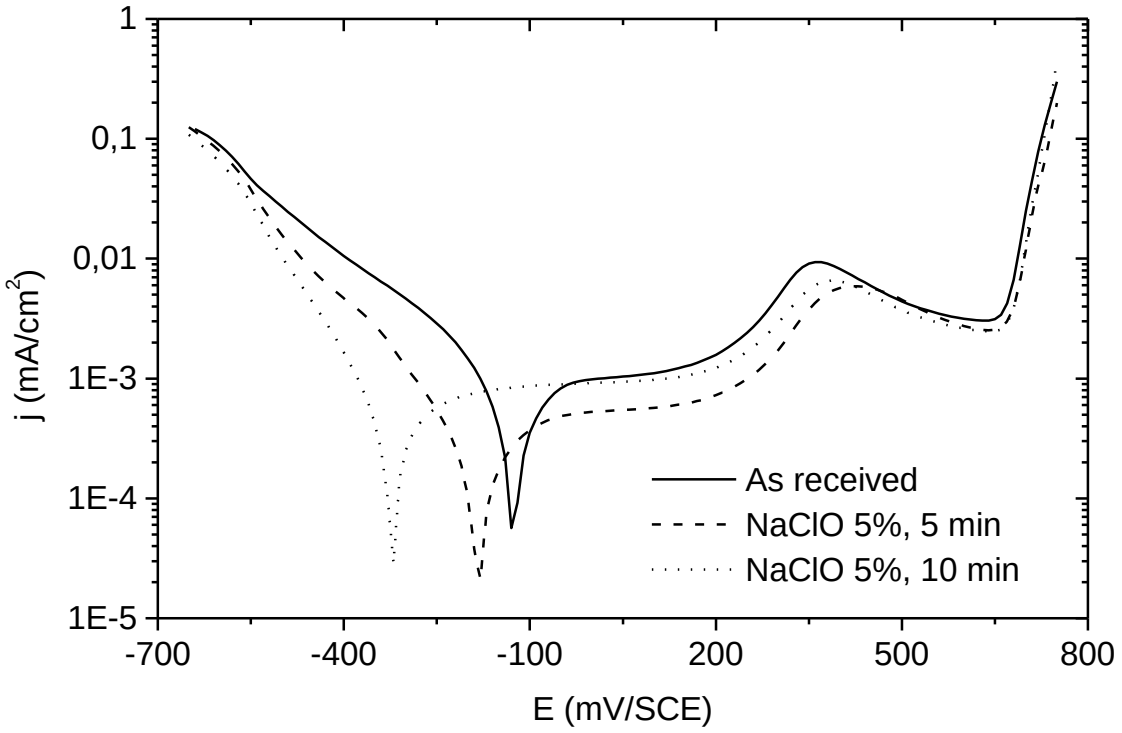


Figure 1 – Potentiodynamic polarization curves recorded at 37°C in 5% NaClO solution on stainless steel K File endodontic instruments

Figure 3 reports the potentiodynamic polarization curves recorded in 5% NaClO solution on K3 NiTi endodontic instruments. In this case, the corrosion behavior of the sterilized instruments is worse than that of the base material. The sterilized samples exhibit current density values higher than those of the base material. By analyzing the anodic branches of the polarization curves, the increase in current density is particularly accentuated for the 10 min sterilized samples, as also evidenced in Table 1, which reports the current density values at a potential of 1000 mV/SCE.

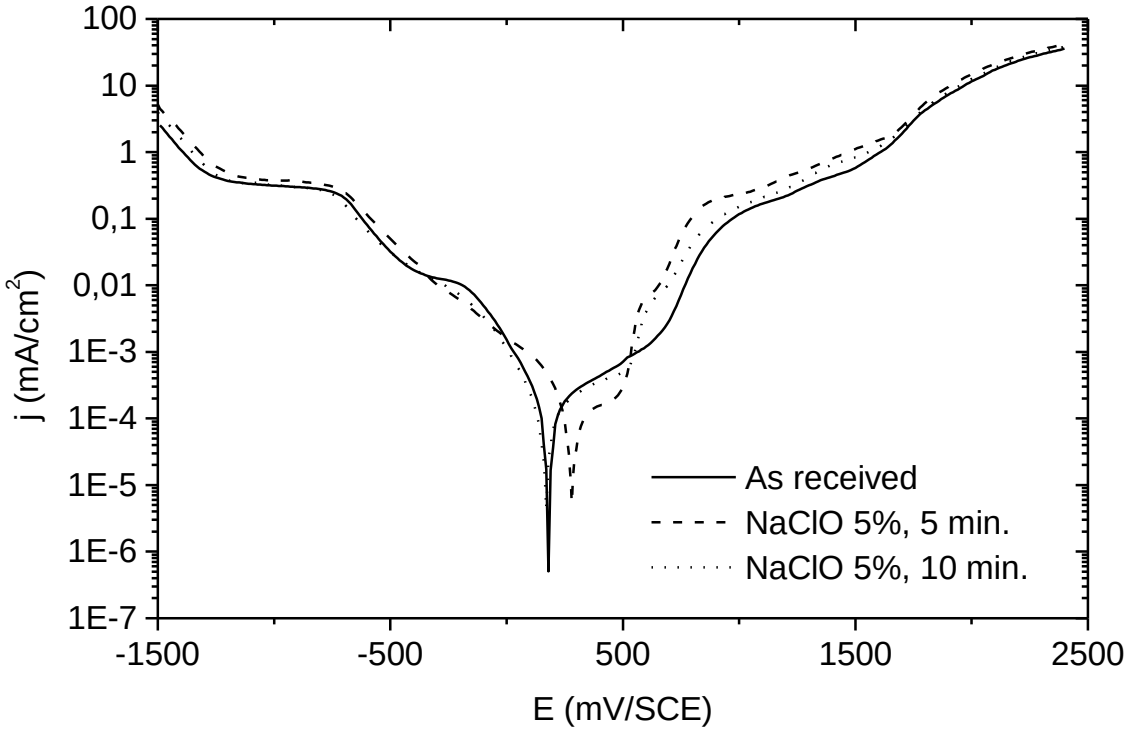


Figure 2 – Potentiodynamic polarization curves recorded at 37°C in 5% NaClO solution on NiTi GT Rotary endodontic instruments

Sample	$\mathbf{j_{(E = 1000\ mV/SCE)}}$ **(mA cm^{-2})**
As received	$5.11 \cdot 10^{-3}$
NaClO sterilized: 5 min	$1.62 \cdot 10^{-1}$
NaClO sterilized: 10 min	1.04

Table 1 – Anodic current density values at the potential of 1000 mV/SCE, deduced by the analysis of polarization curves reported in Figure 3.

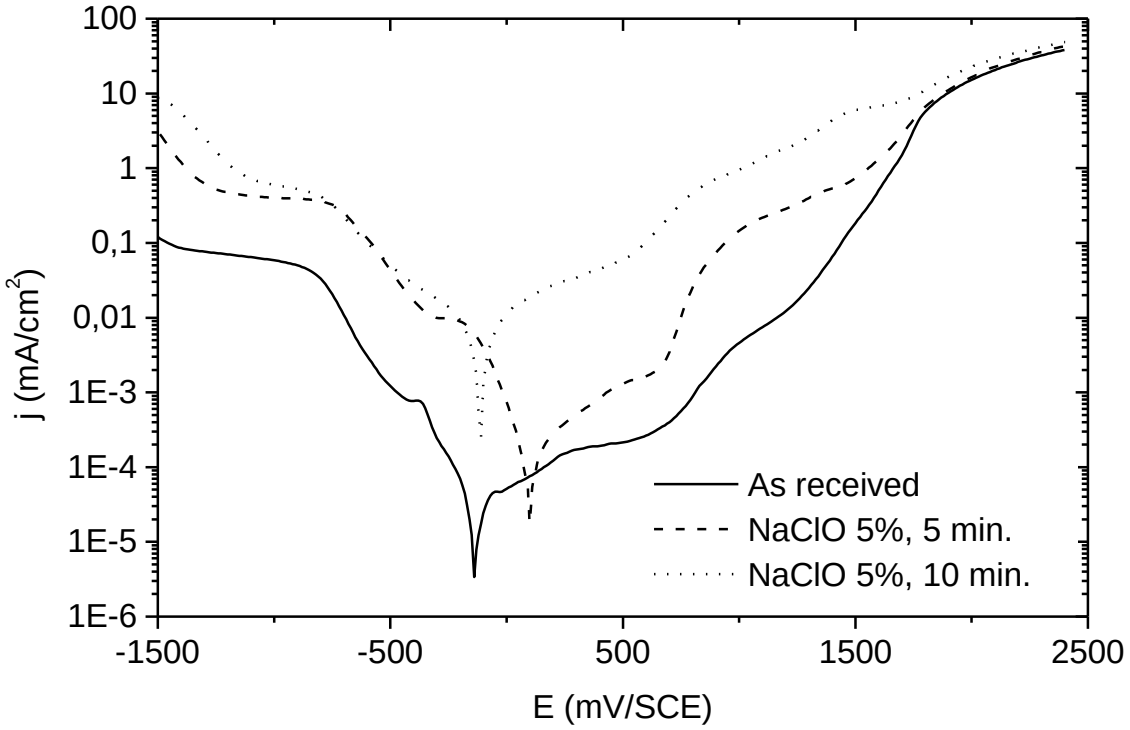


Fig. 3 – Potentiodynamic polarization curves recorded at 37°C in 5% NaClO solution on NiTi K3 endodontic instruments

SEM characterization of the sample surface after the electrochemical tests revealed the presence of cracks as a result of corrosion attack (Fig. 4).

The microstructure appears to play an important role in determining the corrosion behavior of the endodontic instruments investigated. The possible presence of secondary phases may explain the decrease in the corrosion resistance of the K3 sterilized samples due to galvanic coupling phenomena between these phases and the NiTi matrix. Such galvanic interactions may negatively affect the stability of the naturally formed passive TiO_2 film present at the sample surface, thereby leading to localized corrosion phenomena during the sterilization treatment, which is enhanced when the sterilization period is increased. As a consequence, a marked worsening of the corrosion behavior is observed with respect to the non-sterilized base material.

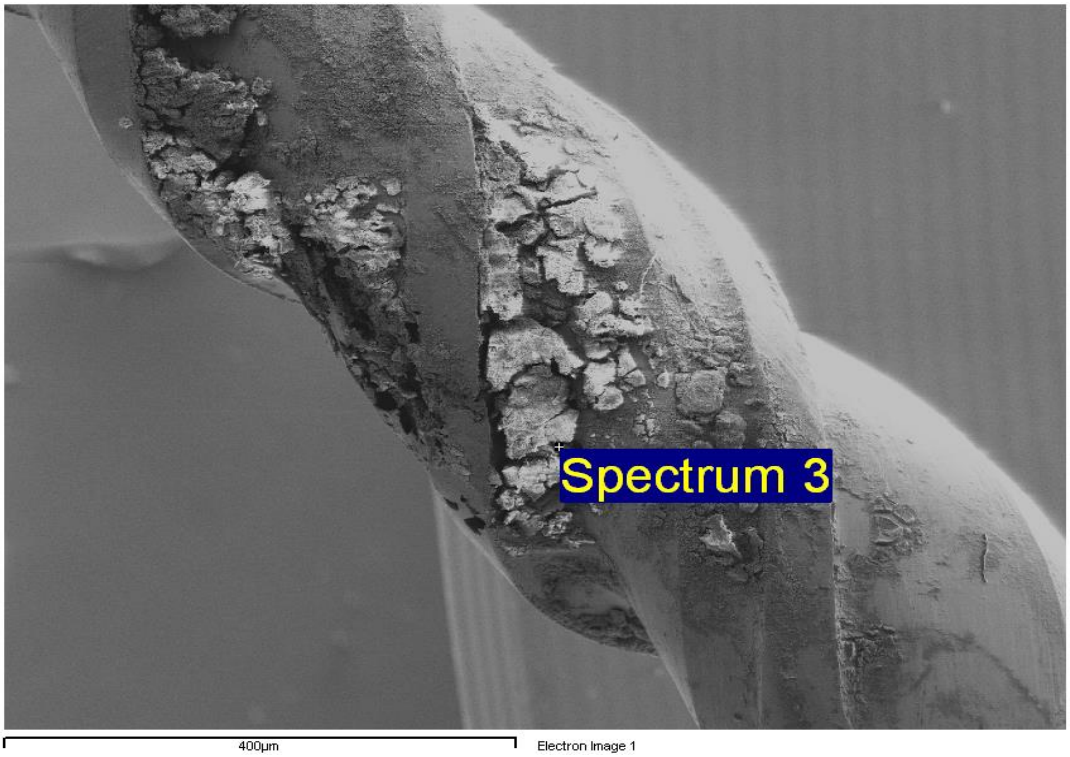


Figure 4 – SEM micrograph of the surface of the NiTi K3 endodontic instrument sterilized 10 min after polarization curve in 5% NaClO solution at 37°C.

EDX analysis carried out on the K3 instruments revealed the presence of significant amounts of Cr in addition to Ni and Ti (Fig. 5), supporting the above hypothesis. Cr is able to form the intermetallic compound $TiCr_2$ [10]. As a consequence, galvanic coupling phenomena between this phase and the NiTi matrix may occur, leading to the destabilization of the TiO_2 surface film.

4. Conclusions

The influence of the sterilization treatment conducted at 50°C in 5% NaClO solution on the corrosion behavior of NiTi (GT Rotary and K3) and stainless steel (K File) endodontic instruments was investigated by measuring potentiodynamic polarization curves recorded in the same environment at 37°C.

The sterilization treatment had no significant influence on the corrosion resistance of K File and GT Rotary instruments. On the contrary, sterilization resulted in a worse corrosion behavior of NiTi K3

instruments compared to the unsterilized sample, and this effect was more pronounced with longer sterilization periods. Observing these results is necessary the development of different instruments using materials that will not corrode in response to sterilization.

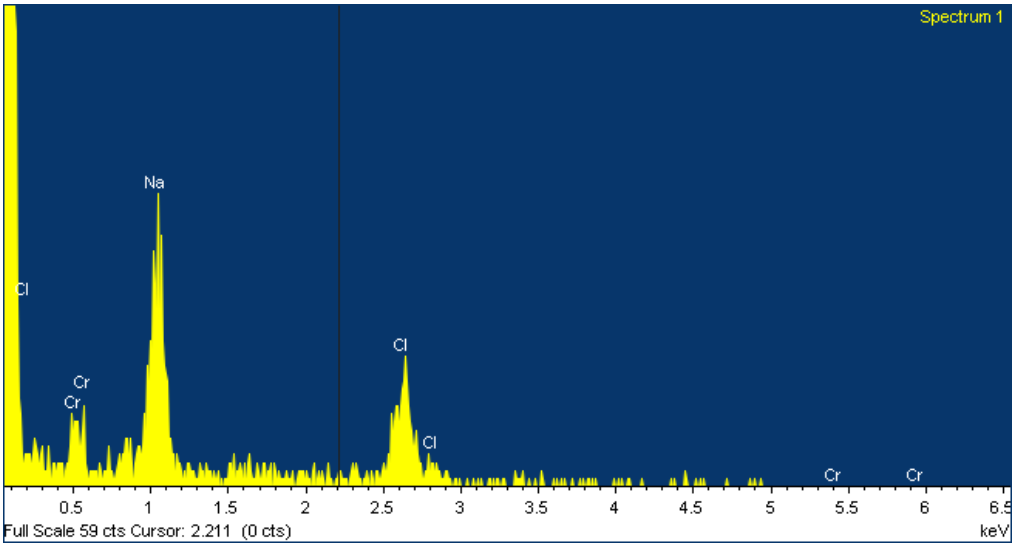


Figure 5 - EDX spectrum recorded at the surface of NiTi K3 endodontic instrument 10 min after polarization curve in 5% NaClO solution at 37°C.

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