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# SODIUM HYPOCHLORITE'S EFFECT ON NICKEL-TITANIUM ROTARY INSTRUMENTS AND ITS EFFECT ON RESISTANCE TO FRACTURE

A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Dentistry at Virginia Commonwealth University.

by

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## Abstract

### SODIUM HYPOCHLORITE'S EFFECT ON NICKEL-TITANIUM ROTARY INSTRUMENTS AND ITS EFFECT ON RESISTANCE TO FRACTURE

By Michael Shane Smith, D.D.S.

A Thesis submitted in partial fulfillment of the requirements for the degree of Master of  
Science in Dentistry at Virginia Commonwealth University.

Virginia Commonwealth University, 2007

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The purpose of this study was to examine the effect of partial and total immersion in sodium hypochlorite on nickel-titanium rotary files and to determine whether resistance to fracture was influenced by the immersion time. One hundred K3™ and 100 ProFile® rotary files were either partially or totally immersed in 5.25% sodium hypochlorite for zero, one, five, thirty, or sixty minutes. After immersion, files were subjected to cyclic fatigue

testing. Time to fracture was recorded and analyzed by a two-way ANOVA. Tukey's honest significant difference was used to identify any differences in immersion times. Within all ProFile groups and partial immersion K3 groups, there was no significant decrease in time to fracture with increased immersion time in sodium hypochlorite. Only the K3 total immersion groups revealed a significant decrease in time to fracture with increased immersion time in sodium hypochlorite.

## CHAPTER 1 Introduction

Over the years the corrosive effects of sodium hypochlorite (NaOCl) on endodontic files have been investigated by several authors (1-10). In 1978, Oliet and Sorin (5) reported that traditional carbon steel endodontic instruments were subject to severe corrosion when immersed in sodium hypochlorite. Many practitioners considered root canal files unreliable because they were easily weakened through corrosion. Since then, stainless steel instruments have replaced carbon steel instruments in endodontics (11).

Oliet and Sorin (5), in agreement with Darabara et al. (9), have shown significant corrosion resistance of stainless steel instruments. Alternatively, Oztan et al. (12) found 0.2% chlorhexidine and 5.25% NaOCl caused severe surface corrosion of stainless steel files.

In 1988, Walia et al. (13) described endodontic instruments manufactured from nickel-titanium (NiTi) alloy. NiTi or Nitinol was discovered by Buehler et al. (14) in 1963 at the Naval Ordnance Laboratory. It has since been used in the medical field for stents (15), prosthetics (16) and fixation devices (17). The alloy has also been used in dentistry as endodontic instruments (13) and orthodontic arch wire (18). Nickel-titanium is a unique alloy with the properties of superelasticity, shape memory, high corrosion resistance, and biocompatibility (19, 20). In contrast to stainless steel, it exhibits remarkable flexibility, a high resistance to torsional fracture (13, 19) and a low modulus of elasticity (4, 13). In 1999 Stokes et al. (2) found NiTi and stainless steel files to have similar resistance to

corrosion. As a result of these findings, nickel-titanium rotary instruments have become very popular (21).

Mechanical preparation and irrigation are requisites for successful root canal treatment (22, 23). Mechanical instrumentation serves these basic purposes; removal of infected pulp tissue and dentin, removal of microorganisms, and enlargement and shaping of the canal before obturation (24). Endodontic irrigants are used to provide flushing of debris, tissue dissolution, antibacterial action, and lubrication (25). As mechanical instrumentation may not remove all the tissue remnants of the pulp chamber and canals (26, 27), one must rely on irrigants (25).

Sodium hypochlorite is widely used as an irrigating solution during root canal preparation (6, 28, 29). Although, sodium hypochlorite has the ability to dissolve organic matter (30) and is bactericidal and virucidal (31, 32, 33), its corrosive effect on endodontic files may negatively alter their performance (2).

During chemo-mechanical debridement, endodontic files are exposed to sodium hypochlorite (28) and these files may be susceptible to the highly corrosive effects of the popular irrigant (1, 5, 8, 10, 12). Corrosion may also occur during chemical sterilization with NaOCl (9, 34). Nickel-titanium's corrosion pattern usually involves pitting and porosity that potentially weakens the structure of the instruments (35, 36). After NaOCl exposure, it is possible that pitting corrosion (36) might occur, thus altering the fracture mechanism of the files from conventional fatigue failure to corrosive fatigue failure (37). Corrosive fatigue failure may lead to premature instrument separation which might decrease the prognosis of an endodontically treated tooth (38, 39).

An investigation by Berutti et al. (10) showed a decrease in cyclic fatigue resistance when NiTi files were completely immersed in 5% NaOCl for five minutes. On the other hand, O'Hoy et al. (1) found no significant reduction in the number of rotations to flexural fatigue or torque at fracture when NiTi instruments were subjected to 1% NaOCl for 15 minutes during a standardized cleaning procedure. In addition, Haikel et al. (3) did not find any significant detriments of 2.5% NaOCl exposure on the mechanical properties of NiTi files. Furthermore, Yared et al. (40) reported that the clinical use of NiTi files with 2.5% NaOCl in four molars did not significantly influence cyclic fatigue in comparison with new files.

Previous studies have tested partial and total immersion of endodontic files in bleach solutions (2, 5, 12, 10). During partial immersion only the instrument's working end is exposed to NaOCl. With total file immersion a galvanic coupling is created between the instrument's shank and the nickel-titanium working end when it is placed in an electrolytic solution (NaOCl). This electro-chemical reaction creates an environment which may lead to corrosion (8).

Corrosion is an electrochemical reaction that involves the transfer of electrons, and thus the flow of electricity, from one area of a metal to another, through a solution that is capable of conducting electricity. The anode is the portion of the metal from which electrons leave the metal (oxidation) to enter the solution, and thus where corrosion occurs. The cathode is the portion of the metal from which the current leaves the solution (reduction) and enters the metal. When these two reactions are in equilibrium, there is no net electron flow. It is the potential of the metal that keeps the anodic and cathodic

reactions in balance. The equilibrium potential of the metal in the absence of an electrical connection to the metal is called the open circuit potential, or  $E_{oc}$ . If the voltage is positive with respect to  $E_{oc}$  (anodic), corrosion takes place. Conversely, if the voltage is negative with respect to  $E_{oc}$  (cathodic), the metal will be protected from corrosion.

In the clinical practice of endodontics, there are times in which a new file may be needed for use prior to being subjected to autoclaving. Roth et al. (34) have recently shown that 12.7% of 150 new, unused files were contaminated with viable microorganisms. Moreover, 46.7% of the 15 types of files that were tested contained at least one contaminated file. In addition, Linsuwanont et al. (7) reported that 5% of the 40 files tested in their study were biologically contaminated, and yet another investigation saw what appeared to be epithelial cells between the flutes of new, unused files (41).

Since there is no guarantee of file sterility, a quick sterilization process may be of tremendous benefit. Roth et al. (34) have proposed a simple chairside sterilization technique which was shown to sterilize endodontic files by placing them in 5.25% NaOCl for at least five minutes. Therefore, the purpose of this study was to examine the effect of total immersion vs. partial immersion in sodium hypochlorite on nickel titanium rotary files of two different manufacturers and to determine whether resistance to fracture was influenced by the immersion time.

## CHAPTER 2 Materials and Methods

### Instrument types

A total of 200 ISO size 25 0.04 taper nickel-titanium (NiTi) endodontic rotary files of 25mm length were used in this experiment. One hundred of the instruments were ProFile<sup>®</sup> rotary files (Dentsply Tulsa Dental, Tulsa, OK, USA) and 100 of the files were K3<sup>™</sup> NiTi rotary files (Sybron Endo, Glendora, CA, USA). Files were opened from their original packaging and used immediately in this study. The files were randomly placed into one of the four groups. The study was performed in two experimental situations, differing only in type of immersion performed.

### Partial Immersion Groups

Five groups of ten files were made for each file type producing a total of ten groups. Within each file type, ten files were exposed to 5.25% NaOCl for one, five, thirty, or sixty minutes, and ten files for each type were left unexposed to NaOCl to serve as negative controls. Disposable flint glass culture tubes (6mm x 50mm) (Fisher Scientific, Hampton, NH, USA) were filled with 750 $\mu$ l of 5.25% NaOCl and their tops were sealed with Parafilm-M<sup>®</sup> (Pechiney Plastic Packaging, Menasha, WI, USA). The working ends of the files were placed through the Parafilm-M barrier until the measuring stop touched the sealing material. At this point, approximately 17mm of the NiTi portion of the file was immersed in NaOCl and the shank of the instrument was left unexposed.



After removal from the NaOCl, files were ultrasonicated for one minute in an L&R Model Q140T ultrasonic bath (L&R Manufacturing, Keary, NJ, USA) filled with distilled water. The instruments were then wiped with cotton gauze and allowed to air-dry overnight.

### **Total Immersion Groups**

Five groups of ten files were made for each file type producing a total of ten groups. To mimic a clinical situation, high density polyethylene painting cups (to mimic NaOCl's original storage container) were used for total file immersion (shank and working end). These cups were filled with 150ml of 5.25% NaOCl. Within each file type, ten files were exposed to 5.25% NaOCl for one, five, thirty, or sixty minutes, and ten files for each type were left unexposed to NaOCl to serve as negative controls. Ten files were placed randomly in each cup to mimic a clinical situation.

After removal from NaOCl, files were ultrasonicated for one minute in an L&R Model Q140T ultrasonic bath filled with distilled water. The instruments were then wiped with cotton gauze and allowed to air-dry overnight.

### **Fracture Testing**

In the second half of the experiment, all files were subjected to fracture testing. The method used to test time to fracture was similar to that used by Kitchens et al. (42). Files were attached to an electric endodontic handpiece (Aseptico Endo ITR™, Dentsply Tulsa Dental, Tulsa, OK, USA) held in a universal testing machine (Instron Corporation, Canton, MA, USA) and lowered 14mm against a polished, grooved metal ramp set at 86°. The files were rotated at 300 rpm at maximum torque until fracture occurred. Time to

fracture was recorded with the experimenter blinded to the immersion time, although file types were obvious.

### **Statistical Analysis**

The experiment was designed to determine the time to fracture as a function of two factors; file type (ProFile vs. K3) and immersion time (zero, one, five, thirty, or sixty minutes). A two-way ANOVA was used to analyze the time to fracture in each immersion category. Tukey's honest significant difference (HSD) was used to identify any differences in immersion times. Level of statistical significance was set at  $p=0.05$ .

## CHAPTER 3 Results

### Part I: Partial Immersion Groups

The average minutes to failure, along with the range of failure times for the partial immersion groups can be seen in Table 1. The table also shows the 95% confidence intervals on the mean time to failure in each immersion category.

The two-way ANOVA indicated that there was no significant decrease in time to failure associated with partial immersion time ( $p=0.5305$ ). Figure 1 is a graphic illustration of the failure times for each file type.

### Part II: Total Immersion Groups

The average minutes to failure, along with the range of failure times for the total immersion groups can be seen in Table 2. The ANOVA procedure performed yielded the 95% confidence intervals.

The two-way ANOVA indicated a significantly different relationship between immersion time and time to failure for the K3 and ProFile groups ( $p<0.0001$ ). Figure 2 illustrates the different trends.

As shown graphically, the ProFile groups kept a constant time to failure across all immersion times ( $p=0.1787$ ). At the 95% confidence level, a file in the ProFile groups would fail between 1.66 and 1.96 minutes when all immersion times were considered. On

the other hand, the K3 groups had a significant decrease as immersion times increased ( $p < 0.0001$ ).

Tukey's HSD was used to find where the significant differences occurred in the K3, total immersion groups. Zero minutes of immersion proved to be significantly different than all other immersion time periods. The one and five minute groups were not significantly different, and the five, thirty, and sixty minute time periods were not significantly different.

Unexpectedly, it was observed that eleven of the files in the K3 total immersion groups were shortened by two to four millimeters after exposure to the NaOCl solution. There were two shortened files in the thirty minute group and eight in the sixty minute group.

#### **Corrosion Products:**

During the partial immersion of files, only four of the files in the ProFile groups showed black particulate matter on the NiTi end. Two were in the thirty minute group and two were in the sixty minute group. No corrosion products were seen on the K3 file groups in the partial immersion testing.

In the total immersion groups, black particulate matter began forming within five minutes of immersion in NaOCl, and this worsened over time for both file types. Visually, the K3 files produced more corrosion products than the ProFiles. By the sixty minute time mark, the K3 files could hardly be visualized through the darkened NaOCl solution.

## CHAPTER 4 Discussion

This study evaluated the effect of total immersion and partial immersion in sodium hypochlorite on nickel titanium rotary files from two different manufacturers, and determined whether resistance to fracture was influenced by the immersion time. The results showed no significant effect on endodontic rotary files if the instruments were partially immersed in 5.25% NaOCl. Only the K3 files, in the total immersion groups, showed a significant decrease in time to failure with cyclic fatigue testing. Within the K3 total immersion groups, there was a significant difference in fatigue failure after only one minute of immersion.

Initially, the investigators in this study intended to compare the file types from the two different manufacturers in relation to time to failure. As the experiment was underway it was noticed that the K3 and ProFile instruments were bent to different degrees under the same experimental conditions. Using Schneider's technique (43), the angle measurements were determined for each type of instrument. The angle formed by the K3 files was 33.5°, and the angle formed by the ProFile instruments was 25°. There was, therefore, a noteworthy difference in flexibility between file types. Within our experimental parameters, the ProFile instruments were placed at an 8.5° more acute angle than the K3 files. This would not occur in a clinical situation where the files would progress through the same curvature in a root and ultimately be bent at the same angle. Therefore, with the

limitations of the testing apparatus, it was not appropriate to compare the two different file types in relation to time to failure measurements.

Within this study, effervescence was noted on the NiTi working ends of multiple files after immersing them into NaOCl, and black particles were seen in the cutting flutes of certain files as immersion time increased. This is in agreement with Stokes et al. (2) who reported visualizing corrosion on the cutting flutes of two NiTi files. Berutti et al. (10) also noted “dark particle” formation with effervescence after NiTi file immersion into NaOCl. More corrosion products were noted in the total immersion half of this experiment where two dissimilar metals on the file were immersed in NaOCl. In fact, the corrosive action was so severe in the K3 total immersion groups that eleven files were shortened in length approximately two to four millimeters after NaOCl exposure. Although corrosion products were noted in this experiment, it was not the intent of this study to measure corrosion. The intent was to measure the effects of the corrosive process. Therefore, there was no quantitative measurement of file corrosion in this study.

The eleven files which were corroded so severely as to shorten their length were in the thirty and sixty minute total immersion groups. Two were in the thirty minute group, and eight were in the sixty minute group. For the purpose of statistical analysis, the shortened files were labeled as fracturing at zero minutes in the fatigue failure testing part of the experiment.

Since several authors have shown biological contamination of new root canal files (7, 34, 41), the endodontist must consider a mode of sterilization for new files. Autoclave and dry heat sterilization appear to be viable options for file sterilization (40, 44, 45).

Yared et al. (40) reported no significant effect on cyclic fatigue of files after four autoclave cycles. Hilt et al. (44) studied the effects of autoclave sterilization on the properties of torque, hardness, and microstructure on NiTi and stainless steel files. Their study revealed no significant effects from the type of autoclave used or the number of cycles of sterilization. In 1985, Iverson et al. (45) found a slight increase in torque strength when stainless steel files were subjected to dry heat sterilization.

Similar to the rapid sterilization of gutta percha cones recommended by Senia et al. in 1975 (46), Roth et al. (34) proposed a chairside sterilization technique for endodontic files. Their study demonstrated sterilization of endodontic files after five minutes of immersion into a 5.25% NaOCl solution. Under the conditions of our study, this technique could be recommended with ProFile NiTi rotary files. On the other hand, the technique would appear to have significant detrimental effects on K3 NiTi rotary files if they are totally immersed in 5.25% NaOCl.

Clinically, total immersion of files in NaOCl would seem to be the likely option. Since one of the two types of files tested was significantly affected by the total immersion in NaOCl at only one minute, caution should be used in this type of sterilization. Considering the hustle and bustle of modern dental offices, it would be easy for files to be left in NaOCl for more than five minutes. In our study it only took thirty minutes for some files to lose two to four millimeters of length at their working end. With corrosion occurring so rapidly, endodontists should be aware of the possible changes in physical properties which may occur with NaOCl immersion.

One should keep in mind that this study only studied two of the several file systems available on the market today. Only size 25, 0.04 taper files were used in this study. Therefore, to draw a more definitive conclusion on this matter, further research is warranted with multiple brands of files in multiple sizes and in differing concentrations of bleach.



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Literature Cited

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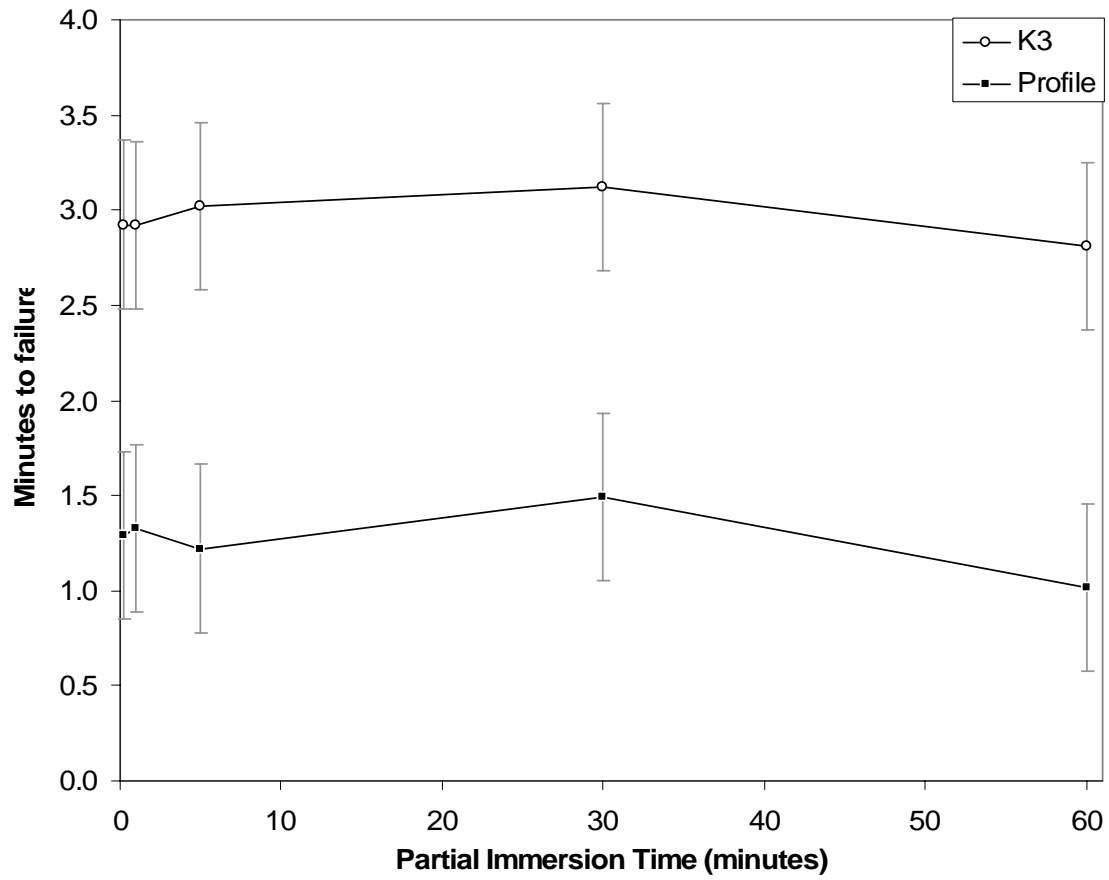
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## APPENDIX A

**Table 1: Time to Failure in Partial Immersion Groups**

| Type    | Soak | Total Minutes to Failure |       |       |       |        |       |
|---------|------|--------------------------|-------|-------|-------|--------|-------|
|         |      | Mean                     | SD    | Range |       | 95% CI |       |
| K3      | 0    | 2.924                    | 0.520 | 2.149 | 3.565 | 2.484  | 3.364 |
|         | 1    | 2.918                    | 0.540 | 2.339 | 3.718 | 2.478  | 3.358 |
|         | 5    | 3.019                    | 0.397 | 2.479 | 3.723 | 2.579  | 3.459 |
|         | 30   | 3.123                    | 0.236 | 2.811 | 3.635 | 2.682  | 3.563 |
|         | 60   | 2.807                    | 0.537 | 2.208 | 3.825 | 2.366  | 3.247 |
| Profile | 0    | 1.289                    | 1.070 | 0.405 | 3.068 | 0.848  | 1.729 |
|         | 1    | 1.329                    | 0.796 | 0.312 | 2.453 | 0.888  | 1.769 |
|         | 5    | 1.221                    | 0.719 | 0.472 | 2.556 | 0.781  | 1.662 |
|         | 30   | 1.491                    | 0.896 | 0.441 | 2.662 | 1.050  | 1.931 |
|         | 60   | 1.016                    | 0.866 | 0.075 | 2.183 | 0.576  | 1.457 |

CI-Confidence Interval, SD-Standard Deviation

**Figure 1: Average Time to Failure for Partial Immersion Groups**

Note: The means and 95% confidence intervals are shown

## APPENDIX B

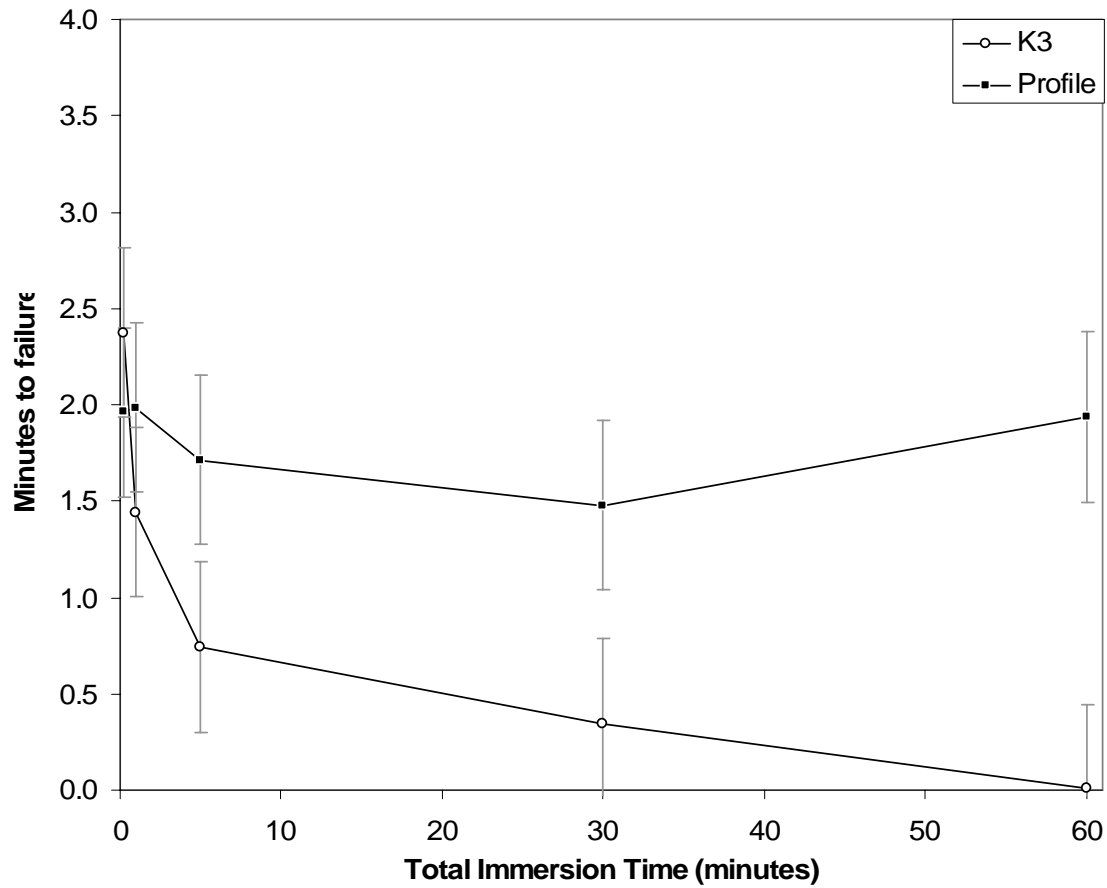
**Table 2: Time to Failure in Total Immersion Groups**

| Type    | Soak | Total Minutes to Failure |       |       |       |        |       |
|---------|------|--------------------------|-------|-------|-------|--------|-------|
|         |      | Mean                     | SD    | Range |       | 95% CI |       |
| K3      | 0    | 2.374                    | 0.177 | 2.175 | 2.676 | 2.032  | 2.716 |
|         | 1    | 1.443                    | 0.471 | 0.773 | 2.114 | 1.101  | 1.784 |
|         | 5    | 0.741                    | 0.722 | 0.217 | 2.319 | 0.400  | 1.083 |
|         | 30   | 0.344                    | 0.985 | 0.000 | 3.146 | 0.003  | 0.686 |
|         | 60   | 0.006                    | 0.012 | 0.000 | 0.032 | 0.000  | 0.347 |
| Profile | 0    | 1.962                    | 0.319 | 1.606 | 2.677 | 1.620  | 2.303 |
|         | 1    | 1.986                    | 0.407 | 1.233 | 2.647 | 1.645  | 2.328 |
|         | 5    | 1.712                    | 0.405 | 1.017 | 2.275 | 1.371  | 2.054 |
|         | 30   | 1.477                    | 0.669 | 0.065 | 2.734 | 1.135  | 1.818 |
|         | 60   | 1.936                    | 0.576 | 1.238 | 2.977 | 1.594  | 2.277 |

CI-Confidence Interval, SD-Standard Deviation

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**Figure 2: Average Time to Failure for Total Immersion Groups**

Note: The means and 95% confidence intervals are shown

## VITA

Michael Shane Smith was born on December 16, 1973 in Dayton, Tennessee. He is a citizen of the United States of America. He graduated from the University of Tennessee at Chattanooga in 1996 with a Bachelor of Arts in Chemistry. He immediately began dental training at the University of Tennessee's Health Science Center in Memphis, Tennessee. In 2000, he obtained the Doctor of Dental Surgery Degree from the University of Tennessee's College of Dentistry. For the next five years he practiced dentistry in a private practice setting in Northeastern Tennessee. From June of 2005 till present, Dr. Smith has attended the Virginia Commonwealth University's graduate endodontic residency program.