The effect of cleaning and sterilization on the torsional properties of protaper rotary Nickel Titanium endodontic instrument

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ABSTRACT
Background: The torsional properties of rotary nickel titanium endodontic instrument may be affected by cleaning and sterilization. This study was conducted to evaluate the effect of the number of cleaning procedures involving immersion in NaOCl with two different concentrations (1% and 5.25%) and sterilization procedures (autoclave and dry heat) on the torsional properties of protaper rotary nickel titanium instrument when subjected to clockwise torsional load. The types of defects for the fractured instruments also were studied.

Material and methods: A total of ninety rotary protaper finisher F3 instruments were divided into 3 groups. Group 1 comprises of ten instruments were subjected to neither cleaning nor sterilization cycles and assigned as the control group. Group 2 and group 3 were subjected to five and ten cleaning and sterilization cycles respectively. Each group comprises of forty instruments, twenty of them soaked within 1% NaOCl and the other twenty were soaked within 5.25% NaOCl, ten of each these twenty instruments, were sterilized with autoclave while, the other ten instruments were sterilized with dry heat oven. Then, all instruments were subjected to a clockwise torsional load until fracture, the torsional moment and angular deflection at fracture were calculated. Then the fractured instruments examined to detect the signs of distortion.

Results: The results show a statistically significant reduction in the torsional strength after cleaning and sterilization cycles regardless the concentration of NaOCl, type and number of sterilization cycles. For the angular deflection, there was a highly significant reduction, where, the number of cycles was the most influencing factor for reduction of the angular deflection whereas, the concentration of NaOCl and the type of sterilization were show no significant influence on the angular deflection. The examination of the fractured instruments show a signs of plastic deformation of the spirals, unwinding, reverse winding, reverse winding with tightening of the flutes of the instruments or a combination of all these signs.

Key words: Torsional strength, angular deflection, NaOCl, nickel titanium.

INTRODUCTION
Rotary NiTi endodontic instruments are gaining popularity based on their superior preparation of canals relative to hand instruments. (1) Because of cost, the instruments are frequently reused, making it essential that cross infection control procedures should be strictly followed. Cross infection between patients could involve bacteria and viruses. To remove the organic matter and debris completely from the files, effective cleaning procedures must be carried out before proceeding to the sterilization process. (2)

Presoaking the endodontic files in NaOCl is a proven method for complete removal of organic and necrotic debris before placing them in an autoclave or dry heat sterilizer. The strength of NaOCl solution must be balanced against potential damage to instruments by corrosion as it is highly corrosive to metals. (3)

Unfortunately, NaOCl could cause corrosion for endodontic instrument in a form of pitting which potentially weakening the structure of the instruments. (4,6) Reuse involves repeated exposure to sterilization between uses (autoclave or dry heat sterilization). The physical properties of these instruments (flexibility, resistance to fracture by twisting and cutting efficiency) should not be significantly altered by sterilization procedures.

The hypothesis which stated that multiple sterilizations of endodontic nickel titanium instruments will lead to a continuous decrease in the resistance of the instruments to separation was tested by many investigators. Some studies showed that multiple sterilizations do not determinately affects mechanical properties of NiTi alloy (7,9), while other studies showed that multiple sterilizations could influences NiTi alloy properties (5,6,10).

Sattapan et al documented two main types of file fracture: torsional fracture and flexural fatigue. Torsional fracture occurs when the tip or any part of the instrument is locked in a canal while the shaft continues to rotate; the instrument exceeds the elastic limit of the metal and shows

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plastic deformation followed by fracture, while flexural or cyclic fracture occurs at the point of maximum flexure when the instrument is freely rotating in a curved canal (11). A few studies were made to evaluate the effect of NaOCl cleaning and sterilization procedures (autoclave and dry heat) concomitantly on the torsional properties of rotary NiTi instrument as these two factors could influence the mechanical properties of NiTi instrument and lead to undesirable and unexpected file fracture during root canal instrumentation.

MATERIAL AND METHOD
A total of 90 protaper rotary NiTi files (finisher files F3) (Dentsply Maillefer, Switzerland) were withdrawn from sealed boxes, and then randomly divided into three experimental groups (Figure 1): Group 1: consisted of ten instruments. This group was assigned as the control group which was not exposed to neither cleaning nor sterilization procedures. Group 2 and group 3 were subjected to five and ten cleaning and sterilization cycles respectively each group was divided into two subgroups: subgroup A: 20 instruments soaked within 1% NaOCl solution. Again subgroup A was divided into: a-ten instruments were sterilized using autoclave sterilizer and b- ten instruments were sterilized using dry heat sterilizer - Subgroup B: consisted of 20 instruments which were soaked within 5.25% NaOCl solution. This subgroup was divided into: a- ten instruments were sterilized using autoclave sterilizer and b- ten instruments were sterilized using dry heat sterilizer.

Cleaning procedure. Each cleaning cycle involved immersion of the instruments in NaOCl solution. Instruments were soaked for 10 minutes for each cleaning cycle (12). Then they were thoroughly rinsed under distilled water immediately after each cycle and allowed to dry 30 minutes before proceeding the sterilization cycle.

Sterilization procedure. Sterilization was carried out according to the International Organization for Standardization specification 3630 (International Organization for Standardization 1992) (13) and ANSI/ADA specification 28 (American National Standards Institute 1988) (14) with the use of an autoclave and a dry heat sterilizers. Sterilization parameters were a pressure of 30 PSI at 136 ± 2°C maintained for 30 ± 0.5 minutes for the autoclave and a temperature of 180 ± 5°C maintained for 30± 0.5 minutes for the dry heat. After the instruments were allowed to cool down to room temperature for 30 minutes before starting the next cleaning and sterilization cycle.

Torsional strength and angular deflection tests. The torque testing device (Figures 2) which was custom designed for this study was previously described by Sattapan et al, a platform was custom designed for this purpose, at one end, the gearbox unit was mounted, and at the other end, the torque meter was placed (15).

A driving chuck was presented at each end. A chuck was attached firmly to the output shaft of a manually driven reversible gearbox unit. The reduction ratio for the gearbox unit was 60:1 and had a revolution counter on its top and input handwheel (each revolution for the input handwheel produces 6° deflection for a driving chuck).

At the other end a torque meter was placed, which is a metal shaft, which had a support and was freely rotating a small chuck was attached to the shaft of torque meter. The lever arm, 5 cm radius (R), was attached perpendicularly to the long axis of the shaft. At one end of the lever arm, it was rested on a 500 g capacity within 0.1 g accuracy load cell (Diamond, model 500, China).

Testing method
Resistance to fracture by twisting (torsional moment) and resistance to angular deflection were
The effect of cleaning and …..

The torsional moment (in g.cm) represented the moment when instrument failure occurred during a clockwise rotation and angular deflection (in degrees) represented the twist angle for breakage. The methods used for measuring these parameters have been described in a previous study. Briefly, the handles were removed with wire cutters at the point where the handle is attached to the instrument shaft. One millimeter of the instrument shaft was secured in a driving chuck that was attached to the gearbox unit, while the last 3 mm of the working part of the instrument were held in a small chuck attached to the shaft of the torque meter. A jig was constructed to ensure reproducible positioning of the tip of the instrument in the chuck (Figures 3).

Figure 3: Jig used to determine the clamping point in a reproducible manner (3 mm from tip of the instrument).

Figure 4: Grasping of two ends of the instrument.

The last 3 mm of the working end of each tested instrument was grasped first and then the handle was grasped in the chuck attached to the gearbox unit chuck. This allowed the investigator that was loading the files to accurately load 3 mm of working end into the chuck (Figure 4).

Torsional loading was performed manually through driving the input handwheel in a manner that reduces a clockwise rotation for the output shaft of the gearbox unit until the instrument fractured. During test the readings of the load cell were recorded continuously. The largest reading (peak) of the load cell in grams (G) at the moment of the fracture was recorded.

The maximum torque at the fracture was calculated through the formula: $\text{Maximum Torque (G.cm)} = \text{Radius (cm)} \times \text{Maximum Load (G)}$

The results were obtained from this formula represented the torsional moment. Angular deflection was obtained by multiplication of the number at the revolution counter at the time of the fracture by six (degree of deflection obtained by a single revolution of the input handwheel).

Fractographic examination

The fracture instruments were evaluated for signs of distortion using the microscope at ×40 magnification. Microphotographs were taken for the separated instruments using a camera obtained from the same manufacturer of the microscope, inserted into to the eyepiece tube of the microscope.

RESULTS

Torque at fracture

The results of this study were collected and analyzed statistically. The mean, standard deviation, minimum and maximum values of the torque at fracture in g.cm of all groups are presented in figure 5 and table 1.

Table 1: Descriptive statistics results of Torque (g.cm) for all groups

<table>
<thead>
<tr>
<th>Torque</th>
<th>N</th>
<th>Mean</th>
<th>SD</th>
<th>Min</th>
<th>M</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>190.77</td>
<td>4.95</td>
<td>183.75</td>
<td>199.50</td>
</tr>
<tr>
<td>2Aa</td>
<td>10</td>
<td>188.40</td>
<td>5.60</td>
<td>183.75</td>
<td>199.50</td>
</tr>
<tr>
<td>2Ab</td>
<td>10</td>
<td>185.40</td>
<td>1.68</td>
<td>183.75</td>
<td>187.50</td>
</tr>
<tr>
<td>2Ba</td>
<td>10</td>
<td>186.05</td>
<td>2.04</td>
<td>182.00</td>
<td>189.00</td>
</tr>
<tr>
<td>2Bb</td>
<td>10</td>
<td>186.60</td>
<td>1.44</td>
<td>185.50</td>
<td>189.00</td>
</tr>
<tr>
<td>3Aa</td>
<td>10</td>
<td>185.92</td>
<td>2.16</td>
<td>182.00</td>
<td>189.00</td>
</tr>
<tr>
<td>3Ab</td>
<td>10</td>
<td>187.12</td>
<td>0.96</td>
<td>186.00</td>
<td>188.50</td>
</tr>
<tr>
<td>3Ba</td>
<td>10</td>
<td>186.70</td>
<td>1.02</td>
<td>185.50</td>
<td>188.50</td>
</tr>
<tr>
<td>3Bb</td>
<td>10</td>
<td>185.80</td>
<td>2.36</td>
<td>180.00</td>
<td>189.00</td>
</tr>
</tbody>
</table>

In comparison among the groups, a statistical analysis of the torque values of all groups, using analysis of variance (ANOVA) test, was performed and the results are shown in Table 2, it is obvious that there was a statistically significant difference among the tested groups, regarding their torque values.
The effect of cleaning and Restorative Dentistry on the angular deflection and torque of dental restorations

Table 2: Analysis of variance (ANOVA) of the Torque of all groups

<table>
<thead>
<tr>
<th>Torque (g.cm)</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>225.287</td>
<td>8</td>
<td>28.161</td>
<td>3.279</td>
<td>0.003</td>
</tr>
<tr>
<td>Within Groups</td>
<td>695.594</td>
<td>81</td>
<td>8.588</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>920.881</td>
<td>89</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Further statistical analysis was performed to examine the differences between the paired groups. Student’s t-test was used for the comparison between the group 1 (control group) and the other groups, in regard to their torque values (Table 3).

Table 3: Student’s t-test results of group 1 (control group) regarding the torque (in g.cm).

<table>
<thead>
<tr>
<th>Compared groups</th>
<th>t-test</th>
<th>P-value</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Vs. 2Aa</td>
<td>1.005</td>
<td>0.328</td>
<td>NS*</td>
</tr>
<tr>
<td>1 Vs. 2Ab</td>
<td>3.251</td>
<td>0.004</td>
<td>**</td>
</tr>
<tr>
<td>1 Vs. 2Ba</td>
<td>2.789</td>
<td>0.012</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs. 2Bb</td>
<td>2.560</td>
<td>0.020</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs. 3Aa</td>
<td>2.838</td>
<td>0.011</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs. 3Ab</td>
<td>2.288</td>
<td>0.034</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs. 3Bb</td>
<td>2.549</td>
<td>0.020</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs.3Bb</td>
<td>2.867</td>
<td>0.010</td>
<td>S</td>
</tr>
</tbody>
</table>

*P<0.05 Significant  
**P>0.05 Non significant

Table 4: Descriptive statistics results of Angular deflection for all groups.

<table>
<thead>
<tr>
<th>Angular deflection (in degrees)</th>
<th>N</th>
<th>M</th>
<th>SD</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>548.40</td>
<td>10.156</td>
<td>534.00</td>
<td>570.00</td>
</tr>
<tr>
<td>2Aa</td>
<td>10</td>
<td>530.70</td>
<td>6.3953</td>
<td>522.00</td>
<td>540.00</td>
</tr>
<tr>
<td>2Ab</td>
<td>10</td>
<td>534.00</td>
<td>8.4853</td>
<td>522.00</td>
<td>546.00</td>
</tr>
<tr>
<td>2Ba</td>
<td>10</td>
<td>541.20</td>
<td>9.7160</td>
<td>522.00</td>
<td>552.00</td>
</tr>
<tr>
<td>2Bb</td>
<td>10</td>
<td>534.60</td>
<td>7.1833</td>
<td>516.00</td>
<td>540.00</td>
</tr>
<tr>
<td>3Aa</td>
<td>10</td>
<td>480.60</td>
<td>11.815</td>
<td>462.00</td>
<td>492.00</td>
</tr>
<tr>
<td>3Ab</td>
<td>10</td>
<td>478.20</td>
<td>10.086</td>
<td>456.00</td>
<td>492.00</td>
</tr>
<tr>
<td>3Bb</td>
<td>10</td>
<td>479.10</td>
<td>8.9994</td>
<td>456.00</td>
<td>486.00</td>
</tr>
</tbody>
</table>

Regarding the degree of the angular deflection, another analysis of variance (ANOVA) test, was performed, in comparison among the groups, and the results of this second (ANOVA) test showed a very highly statistically significant difference among the tested groups as shown in Table 5.

Table 5: Analysis of variance (ANOVA) of the Angular deflection of all groups.

<table>
<thead>
<tr>
<th>Angular deflection (in degrees)</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>77759</td>
<td>8</td>
<td>9719</td>
<td>115.11</td>
<td>.000</td>
</tr>
<tr>
<td>Within Groups</td>
<td>6839</td>
<td>81</td>
<td>84.438</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>84598</td>
<td>89</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Since the (ANOVA) test showed a very highly statistically significant difference, Student’s t-test was carried out for the comparison between the group 1 (control group) and the other experimental groups, regarding to their angular deflection (in degrees) (Table 6).

The angular deflection
The descriptive statistic of the results regarding the angular deflection (in degrees), which included the mean, standard deviation, minimum and maximum values of all groups are presented in Figure 6 and Table 4.
Table 6: Student’s t-test results of group 1 (control group) regarding the angular deflection (in degrees).

<table>
<thead>
<tr>
<th>Compared groups</th>
<th>t-test</th>
<th>P-value</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Vs. 2Aa</td>
<td>4.663</td>
<td>0.000</td>
<td>HS***</td>
</tr>
<tr>
<td>1 Vs. 2Ab</td>
<td>3.441</td>
<td>0.003</td>
<td>S**</td>
</tr>
<tr>
<td>1 Vs. 2Ba</td>
<td>1.620</td>
<td>0.123</td>
<td>NS*</td>
</tr>
<tr>
<td>1 Vs. 2Bb</td>
<td>3.508</td>
<td>0.003</td>
<td>S</td>
</tr>
<tr>
<td>1 Vs. 3Aa</td>
<td>13.761</td>
<td>0.000</td>
<td>HS</td>
</tr>
<tr>
<td>1 Vs. 3Ab</td>
<td>15.509</td>
<td>0.000</td>
<td>HS</td>
</tr>
<tr>
<td>1 Vs. 3Ba</td>
<td>16.149</td>
<td>0.000</td>
<td>HS</td>
</tr>
<tr>
<td>1 Vs. 3Bb</td>
<td>16.251</td>
<td>0.000</td>
<td>HS</td>
</tr>
</tbody>
</table>

*P<0.05 Significant  
**P>0.05 Non significant  
***P<0.001 High significant

Fracture characteristics

The fractured instruments showed a plastic deformation of spirals, unwinding, reverse winding for the original direction of spirals, reverse winding with tightening of the flutes adjacent to the fractured site or a combination of them (Figures 7).

Most of the fractured instruments which showed unwinding and reverse winding for the original direction of spiral were belong to the group 2 and group 3 while most of the fractured instruments which showed reverse winding with tightening of the flutes were belong to the control group.

Figures 7: Top left new instrument. Top right, distorted with unwinding. Bottom left, distorted instrument with reverse winding. Bottom right, with reverse winding with tightening of the flutes x40

DISCUSSION

More instruments were failed in torsion than from flexural fatigue (11). Many studies showed that Protaper files has a higher fracture rate than other rotary NiTi systems (17,18). Finisher files were tested as they used to shape the apical third of the canal, they undergo fairly high bending and pure torsional stress in their thinner and weaker part (apical 3 mm) as the life expectancy of finisher files less than shapers as the latter accumulates more torsional stress, (19).

The angular deflection is a sign of the instrument flexibility in contrast to the torsional moment which is a sign of strength of a material (20).

The maximum angular deflection is a very important parameter, as it acts as a safety factor in relation to the fracture of the instrument. Larger the angular deflection of an endodontic instrument, the larger will be its toughness and plastic deformation before starting the fracture (16).

Clinical recycling exposes endodontic files to changes in their physical properties, when the files are subjected to repeated sterilization cycles with dry heat or an autoclave (6). In the present study the effect of combined effect of both corrosive NaOCl and sterilization on the torsional properties of NiTi files was studied. A fractographic analysis was made also for the fractured instruments.

Torque at fracture

Torque at fracture limit was defined as the maximum force that an instrument can withstand before fracture, when twisted (15).

The range of average torque at failure for the new and treated instruments was between 185.5 and 190.8 g.cm was higher than that recommended by the manufacturer who advise to preset torque levels for the motor at 1.3 - 1.1 Ncm for the protaper finisher files (21). These results showed that these instruments could be used safely at this range of torque without fearing from fracture regarding to torsional strength.

The results of our study showed a significant reduction in the torsional strength of rotary NiTi instruments after exposure to NaOCl and sterilization. This may come in agreement with the findings of other studies (6,22-24), which was the results of a combined adverse effects of corrosive NaOCl and sterilization on the mechanical properties of NiTi instruments. as upon torsion test, samples subjected to combined tension compression shear stress, fracture caused by fatigue failure mechanism occurs due to crack initiation at the cutting surfaces and propagation toward the file’s axial centre (25-27).

It is likely that pitting or crevice corrosion might occur first which serves as weak points and sites for the cracks initiation and subsequently propagation (28). Corrosion mechanism might be activated during...
chemomechanical preparation, chemical disinfection or sterilization.

There are no significant differences between different experimental groups regarding the number of cycles when other variables were fixed (e.g., NaOCl concentration and type of the sterilization). This was consistent with the results of another study (28).

The present study showed no significant differences between different experimental groups regarding the type of the sterilization (dry heat or autoclave), when other variables such as number of the cleaning and sterilization cycles and NaOCl concentration were fixed. This finding agreed with the findings of other studies (6,26). The explanation for this situation is that torsional strength mainly affected as a result of exposure to NaOCl rather than to the heat, which was confirmed by the results of Hoy et al, who showed lower means of torsional strength of NiTi instrument after prolong exposure to 1% NaOCl alone (23).

The torsional strength for the treated instruments not severely deteriorated regarding to the torque at fracture values, even after 10 cycles. The possible explanation for this phenomenon was not clear, but may be related to the beneficial effect of the heat treatment on the alloy strength. However, considering propagation of fatigue cracks is directly associated with the mechanical strength of the material, the increase of instrument fatigue resistance was found in many studies after heat sterilization procedures which may be a direct consequence of the observed increase in hardness. Serene et al suggested an improvement in the alloy strength following heat treatment (5).

Angular deflection

For instance, the present study results showed that values of angular deflection at failure was dramatically decreased after 5 and 10 sterilization cycles as a trend towards higher angle of rotation at fracture was observed with the new instruments compared to the sterilized ones. These findings are in full agreement with the results of other studies (6,8,31,32). This may be explained by fact that heat treatment of NiTi rotary endodontic instrument will make them stiffer and less flexible (5). These results also was confirmed by a study found that 5 times sterilized and used profile instruments were less flexible than new instruments when exposing them to the bending test (33).

Under the parameter of this study, there are no significant effects regarding NaOCl concentration and type of sterilization on the angular deflection values, when comparing different experimental groups at a fixed number of cycles, as these two parameters having a little effect on the instrument flexibility. The most influencing factor was the number of the cycles and it is obvious that the angular deflection was more affected than the torque at failure regarding the number of the cycles. The type of sterilization procedure does not exhibit a significant influence despite the deference in the temperature, used for the autoclave and dry heat sterilizers. This finding is of a general agreement with the results of other studies (6,8).

The explanation of the deterioration is physical, as an excessive numbers of sterilization cycles may have a cumulative effect on annealing of the instrument, this confirmed by a study showed that heat treatment for orthodontic NiTi wire at 500 °C for 10 minutes caused a minimal effect on cantilever bending force, but 2 hours of heat treatment at the same temperature increased dramatically the superelastic bending moment (34).

It was clear that there is no linear relationship between the torsion and the angular deflection values for the control group and the experimental groups. This finding is in consistence with the findings of the other studies (35,36). The best explanation for this situation is that NiTi alloy of rotary instrument can posses a significant ductility in torsion without experiencing separation to a certain limit (37). According to the results of our study this limit may be reduced (ductility) as heat treatment causing a material stiffer.

Fractographic analysis

The fractured instruments showed the typical signs of torsional failure such as plastic deformation of spirals, unwinding, reverse winding for the original direction of spirals, reverse winding with tightening of the flutes adjacent to the fractured site or a combination of them. This has been in close agreement with the results of other studies (11,25,38-40). This might be explained by the fact that the direction of the applied force (clockwise) was an apposite to the spiral direction of the flutes so the flutes will tend to be widened (opened).

Most of the fractured instruments which showed unwinding and reverse winding for the original direction of spiral were belong to the group 2 and group 3 while most of the fractured instruments which showed reverse winding with tightening of the flutes were belong to the control group. This may be explained as the control group is more flexible, and so on it was subjected to a higher degree of distortion before it is breakage, while the other experimental groups where stiffer so, they were fractured with a little distortion as they are stiffer than control group.
REFERENCES