An *in vitro* assessment of the mechanical characteristics of nickel-titanium orthodontic wires in Fluoride solutions with different acidities

Shiva Alavi[1], Sara Barooti[1] and Ali Borzabadi-Farahani[2]

ABSTRACT

Objectives: The aim was to evaluate the *in vitro* effects of fluoride solutions with different acidities on load-deflection characteristics of nickel-titanium (NiTi) orthodontic wires.
Materials and Methods: In this study, which lasted 30 days, 36 (3 cm long, 0.016 × 0.022 inches, SENT 1622, G & H wire Company, Greenwood, Indiana, USA) NiTi wires, were divided into three experimental groups of 12 each. Two groups were subjected to 0.05 topical fluoride mouthwash with different acidities (G1, pH 4; G2, pH 6.6) for 90 s, twice a day, and kept in normal saline after that. The third group (G3, the control group) was kept in normal saline only. Load and unload forces were measured with three bracket bending test in a universal testing machine (Testometric Co, Rochdale, UK). Loading and unloading plateaus and hysteresis were also recorded. Data were then analyzed using analysis of variance and honestly significant difference Tukey at $P < 0.05$.
Results: During the loading phase, there was a significant difference between deflections ($P < 0.001$); but there was no interaction effect ($P = 0.191$) and no significant difference among three groups ($P = 0.268$). In the unloading phase, there was a significant difference between deflections ($P < 0.001$) and an interaction effect was also observed ($P = 0.008$). Further, significant differences noted among three groups ($P = 0.037$). Only in the unloading phase, at deflections of 2.2 through 0.2 mm, significant differences between the mean force values of the G1 and G3 groups were observed ($P = 0.037$).
Conclusion: Based on this *in-vitro* study, compared to neutral fluoride solution, daily mouthwash with a fluoride solution with more acidic pH of 4 affected the NiTi wires load-deflection characteristics during the unloading phase. This finding may have clinical implications and can be further validated by *in-vivo* studies.

Key words: Acidity, fluoride mouthwash, nickel-titanium, orthodontic wire

INTRODUCTION

Due to their desirable shape memory properties, nickel-titanium (NiTi) wires are increasingly used during the first stage of a comprehensive treatment in orthodontics that is, mainly for the alignment of teeth by correcting crowding and rotations.[1] These favorable characteristics include high spring back, flexibility, and ability to apply light and constant forces, as well as superelasticity, which are based on a martensitic phase transformation allowing the NiTi alloys to return to a previously defined shape when strained up to 8%.[2] Suboptimal oral health and biofilm formation are common issues in orthodontic treatment which can cause demineralization and caries[3] and for solving such problems, orthodontists may prescribe fluoride producs for their patients.[4] In the acidic products, such as prophylactic agents, the titanium protective film reacts with hydrofluoric acid to form sodium titanium fluoride[5,6] and the breakdown of the film reduces corrosion resistance because titanium indicates an intrinsically high activity.[7]

[1]Dental Materials Research Center, Departments of Orthodontics, School of Dentistry, Isfahan University of Medical Sciences, Isfahan, Iran, [2]Department of Orthodontics, Warwick Dentistry, Warwick Medical School, University of Warwick, Coventry, NHS England (Locum Consultant Orthodontist), UK

Address for correspondence: Dr. Ali Borzabadi-Farahani, Warwick Dentistry, Warwick Medical School, University of Warwick, Coventry, UK.
Email; faraortho@yahoo.com

Access this article online	
Quick Response Code:	Website: www.jorthodsci.org
	DOI: 10.4103/2278-0203.156030

Kaneko *et al.*,[7] showed degradation in performance of four major orthodontic alloys (NiTi, β-titanium, stainless steel, cobalt chromium) caused by hydrogen absorption during short-term immersion (1 h) in 2.0% acidulated phosphate fluoride. Walker *et al*.[8] also found that mechanical properties and surface characterization of orthodontic archwire decreased during short-term immersion (1.5 h) in topical fluoride treatment with either an acidulated fluoride agent or a neutral fluoride agent compared with distilled water (as a control group). Kwon *et al*.[9] investigated mechanical properties of β-titanium orthodontic wires in the presence of four sodium fluoride solutions (0.05% pH = 6, 0.05% pH = 4, 0.2% pH = 6, 0.2% pH = 4) for l or 3 days. He found that the tensile strength of the immersed wires was significantly reduced compared to as received one, and ion released had much increased for higher sodium fluoride (NaF) concentration and lower pH. Walker *et al*.[10] immersed stainless steel and β-titanium orthodontic archwires in either an acidulated fluoride agent, a neutral fluoride agent or distilled water (control) for 1.5 h and found that unloading mechanical properties were significantly decreased for both fluoride solutions. Kaneko *et al*.[11] examined hydrogen embrittlement of β-titanium orthodontic wires in acidic and neutral aqueous solutions and concluded that the immersion in fluoride solutions led to the degradation of the mechanical properties and fracture of beta titanium alloy associated with hydrogen absorption. Similarly, they stated that when immersion in acidic fluoride solutions was carried out, Ni-Ti superelastic alloy underwent general corrosion and absorbed substantial amounts of hydrogen.[12] In another investigation, after the tensile test (after immersion) and hydrogen thermal desorption analysis of of Ni-Ti superelastic alloy, they hypothesized that one reason that Ti and its alloys fractured in the oral cavity is because hydrogen was absorbed in a fluoride solution, such as prophylactic agents.[13]

Toniollo *et al*.[14] evaluated the effects of fluoride-containing solutions on the commercially pure titanium (Cpti) using three solutions as follows: Distilled water, 0.05% NaF, 0.2% NaF during different times and concluded that fluoride containing solutions (pH = 7) used as mouthwashes did not damage the surface of Cpti and could be used in patients with titanium-based restorations.

Ramalingam *et al*.[15] determined the *in vivo* effect of fluoride agents on the mechanical properties of NiTi and copper NiTi archwires. Thirty patients were divided into three groups. First group used no topical fluoride agent. The second group used fluoride mouthrinse and the third group used fluoride gel. Then arch wires were examined and it was concluded that unloading properties of orthodontic wires fell significantly in gel group. Apparently, topical fluoride agents altered the mechanical properties of NiTi wire and hence could prolong orthodontic treatment.

The widespread use of NiTi wires in orthodontics and the importance of mechanical properties of orthodontic wires in the treatment efficiency as well as the probable effect of fluoride mouthwashes resulting from the presence of hydrogen and fluoride ions on the orthodontic wires, demand further research on the possible interaction between fluoride use and NiTi archwire properties. The aim of this *in vitro* study was to assess the effect of daily fluoride mouthwashes on the mechanical characteristics of orthodontic NiTi orthodontic archwires.

MATERIALS AND METHODS

For the present *in vitro* study, 36 NiTi wires (3 cm length) with the dimensions of 0.016 × 0.022 inches (SENT 1622, G & H wire Company, Greenwood, Indiana, USA) were used. The samples were divided into three groups of 12 each as follows:

- G1, the pH = 4 group, samples were immersed in the normal saline solution for 30 days, and were immersed in the 0.05% fluoride solution (pH = 4) twice a day for 90 s. Subsequently, they were returned into normal saline.
- G2, the pH = 6.6 group, samples were immersed in the normal saline solution for 30 days, but were immersed in the 0.05% commercial fluoride mouthwash (Advantage Mouth Rinse, Oral B, South Boston, UK) with the pH = 6.6 twice a day for 90 s; subsequently they were returned into normal saline solution.
- G3, the normal saline group; samples were only immersed in normal saline solution (pH = 7) for 30 days.

Assessment of Load-Deflection Characteristics

After 30 days, the wires were removed from the solutions and Universal Testometric Machine (Testometric Co, Rochdale, UK) measured the load-deflection characteristics of the samples in the 37°C water bath using a setup made for this purpose. In order to produce the load-deflection curves, three copper cylinders with a length of 3 cm and a rectangular cube bar with dimensions of 10 cm × 12 cm × 1 cm with completely smooth surfaces were prepared, two copper cylinders with the central distance of 14 mm were screwed, and the third cylinder was connected to the crosshead of the instrument. On each of the three cylinders, a stainless steel maxillary canine bracket was placed (Equilibrium® 2, Dentarum, Inspringen, Germany) with the slot size of 0.018 inches, and fixed with the cianoacrylate glue. The wire was subsequently placed passively in the slot of the brackets so that the wire maintained the straight form; subsequently wires were placed in the bracket slots and tied by elastomeric ligatures (Safe-T-Tie Silver, Ortho Organizer, Carisbad, CA, USA) and the three bracket bending test was performed on each sample.

The instrument began to move with a speed of 0.5 mm/min with the range of 2.4 mm; the loading and unloading forces were registered, and the load-deflection curves plotted. Loading forces at 2.4–0.2 mm deflection (L2.4 to L0.2) and unloading forces at 2.2–0.2 mm deflection with 0.2 mm deflection intervals (L2.2 to L0.2) were recorded. Further, the unloading (between UL0.6 and UL1.6) and loading plateau means (between L 0.6 and L 1.6) and the difference between them (hysteresis) was estimated for each sample.

Statistical Analysis

Data were analyzed using SPSS 15 for windows (SPSS Inc, Chicago, III). The repeated measurement of ANOVA and Tukey's *post-hoc* tests were used for statistical analysis. All statistical analysis were undertaken at the $P < 0.05$ level of significance.

RESULTS

The measured force values (mean and standard deviation) for loading and unloading of all groups are shown in Table 1. The loading and unloading plateau means and the hysteresis relevant to three groups are shown in Table 2 and Figure 1.

During the loading phase, there was a significant difference between the deflections ($P < 0.001$). There was also no interaction effect ($P = 0.191$); the changes occurred in three groups at various deflections were similar. Overall, there was no significant difference among three groups ($P = 0.268$).

In the unloading phase, firstly, there was a significant difference between the deflections ($P < 0.001$). Secondly, there was an interaction effect ($P = 0.008$), that is, the changes occurred in three groups at various deflections were not similar. Further, there were significant differences among three groups ($P = 0.037$).

Honestly significant difference Tukey was also applied to determine which groups were statistically different. It became clear that this difference was related to the unloading phase between the G1 and G3 groups ($P = 0.037$) and at UL0.2, UL0.4, UL1.4, UL1.6, UL1.8, UL2, UL2.2 ($P < 0.05$). There was not any significant differences between the mean values of unloading plateau ($P = 0.324$), loading plateau ($P = 0.464$) and hysteresis ($P = 0.566$) for three groups.

DISCUSSION

The present in-vitro study demonstrated that the fluoride and hydrogen ions could affect the load-deflection characteristics of NiTi wires, particularly, the hydrogen embrittlement effect, which has been discussed previously.[16] Despite the presence of a protective film, metal ions such as titanium and nickel could leak out of the alloy and presenting with relatively lower corrosion resistance and higher amount of Ion release, which can be cytotoxic.[17-23] A component of the NiTi alloy, Nickel, is also allergic and cytotoxic.[24] Although the released titanium is not cytotoxic, the lower pH can result in higher release of nickel ions.[25] It is possible to examine surface roughness and topographic characteristics of the wires with scanning electronic microscope reported by other researches

Table 1: Mean (SD) of measured force values during the loading/unloading in three groups

Group	L2.4 (g)	L2.2 (g)	L2 (g)	L1.8 (g)	L1.6 (g)	L1.4 (g)	L1.2 (g)	L1 (g)	L0.8 (g)	L0.6 (g)	L0.4 (g)	L0.2 (g)
Control	1055	982	919	860	810	764	108	684	664	640	575	453
	(82)	(62)	(44)	(31)	(24)	(20)	(19)	(20)	(21)	(19)	(16)	(16)
pH=4	993	964	905	857	809	757	708	685	631	603	518	417
	(84)	(82)	(76)	(61)	(47)	(38)	(35)	(32)	(96)	(28)	(32)	(18)
pH=6.6	1011	950	913	861	811	764	715	690	663	623	539	429
	(71)	(59)	(42)	(39)	(33)	(30)	(26)	(25)	(25)	(39)	(47)	(26)

Group	UL2.2 (g)	UL2 (g)	UL1.8 (g)	UL1.6 (g)	UL1.4 (g)	UL1.2 (g)	UL1 (g)	UL0.8 (g)	UL0.6 (g)	UL0.4 (g)	UL0.2 (g)
Control	427	272	179	133	126	120	118	113	116	126	160
	(75)	(62)	(47)	(37)	(27)	(23)	(16)	(16)	(15)	(18)	(23)
pH=4	324	202	137	104	99	110	113	112	123	146	133
	(81)	(59)	(37)	(24)	(14)	(11)	(11)	(11)	(11)	(18)	(31)
pH=6.6	355	220	143	105	98	109	113	115	123	101	152
	(72)	(61)	(39)	(27)	(20)	(20)	(18)	(17)	(15)	(17)	(29)

SD – Standard deviation; L – Loading; UL – Unloading

Table 2: Mean (SD) of force values related to loading plateau, unloading plateau, and Hys of three groups

Group	PUL (g)	PL (g)	Hys (g)
Control	120 (20)	712 (19)	592 (24)
pH=4	110 (11)	699 (36)	588 (33)
pH=6.6	110 (18)	711 (27)	600 (23)

PUL – Plateau unloading; PL – Plateau loading; Hys – Hystresis; SD – Standard deviation

and these studies had shown that the immersed wires as compared with the control group also showed a difference in this regard.[7,9,25] Overall, a protective layer is formed on NiTi and the titanium surface when these contact with the aqueous environment.[26,27] For the titanium surface the protective film is constituted by titanium oxide (TiO_2), but on NiTi the passive film has also smaller amounts of nickel oxide or metallic Ni, making it more susceptible to chemical attack.[26,27] Further, issues associated with poor corrosion resistance can affect the treatment effectiveness as well as toxic and allergic reactions due to nickel release, creating biocompatibility issues for of NiTi alloys.[28] The present study, however, did not assess the corrosive characteristics of the NiTi wires.

Change in mechanical characteristics of wires immersed in the acidic electrolytes could be related to different factors such as the concentration of fluoride ions present in the solution, the pH of the solution, the manufacturing characteristics of the wires, and the duration of immersion, that had been referred to in the previous studies.[29] In this study, three bracket bending test was used, which is the alternative to the three point bending test,[30] and the most precise design for examining mechanical properties of orthodontic wires. As brackets were used in this procedure, a relatively comprehensive simulation was obtained in terms of the inter bracket distance, wire length, bracket type, and the friction between the wire and bracket.[31] The central distance between two brackets, which were installed on the fixed cylinders, was selected to be 14 mm, that is, the inter-bracket distance was 7 mm, which was more consistent with the bracket distance in the patient's mouth. The speed of the middle cylinder connected to the crosshead of the machine was 0.5 mm/min with a range of 2.4 mm, which lies within the scope of previous studies.[30]

In this study, incubation and three bracket bending test was performed in the humid environment at 37°C, which simulated the mouth condition. There were controversies in various studies regarding the effects of the acidity rate on the performance and characteristics of orthodontic alloys. Kwon *et al.*[9,32] reported that through increasing the acidity (reducing) pH, elements released from the alloy had been increased. While according to Harris *et al.*,[33] the acidity of the environment did not have any effect on the properties of the alloy. The NiTi wires were used due to their increasing use in clinical practice and their desirable properties. In addition, regarding the crucial importance of the mechanical characteristics of the wires in efficiency and duration of orthodontic treatment, it was decided to use this kind of wire in the present study. Further, three solutions were used. The reason for using 0.05% fluoride was that this concentration is present in the commercial fluoride mouthwashes, which are used daily as part of home health care. Moreover, using various pH values allowed us to examine the effect of acidity on the properties of NiTi wires. In most studies on the mechanical characteristics, yield strength and modulus of elasticity and tensile strength were examined.[9,10,18, 33]

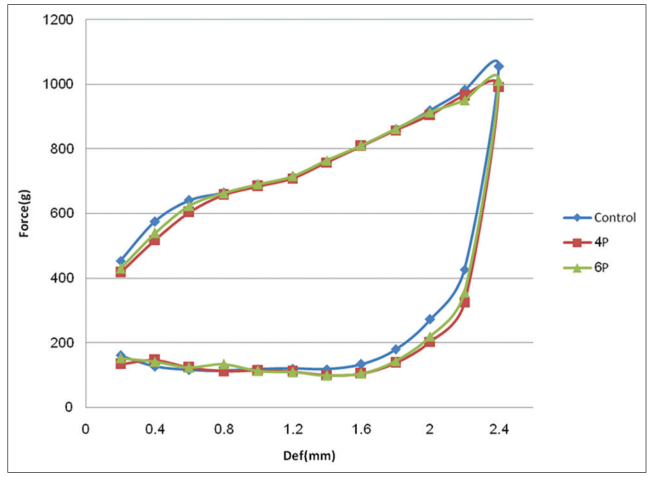

Figure 1: Load-deflection diagram related to three groups

Our findings were consistent with Ramalingam's *et al*. report[15] in that the mechanical characteristics of NiTi wires during unloading was dropped using professional fluoride gel, but did not change with mouthwash. Walker *et al.*[10] found that the acidity caused the unloading of the mechanical characteristics of NiTi alloy to be reduced. This finding verified our research but regarding the effect of fluoride ions on the NiTi alloys, they reported a reduction in mechanical characteristics while we did not observe such an effect statistically. Moreover, one probable reason can be that they have used high fluoride concentration of 1.1% in their research. Toniollo's *et al.*[14] findings suggested that fluoride ion in the concentrations of 0.05% daily did not damage the surface of the titanium alloys. Findings of Kao and Huang[34] indicated that NiTi alloy corroded in the artificial saliva with pH = 4 more than NaF solution, this may explain the results of present study concerning the effect of acidity. Similarly, Yokoyama *et al.*[16] stated that acidic fluoride solutions adversely affected the properties of superelastic alloys that show some similarity with our findings.

CONCLUSIONS

- The NiTi mechanical characteristics were affected in the acidic solutions
- Compared to neutral fluoride solution, a fluoride mouthwash with more acidic pH of 4 affected the NiTi wires mechanical characteristics adversely during unloading phase.

REFERENCES

1. Jian F, Lai W, Furness S, McIntyre GT, Millett DT, Hickman J, *et al*. Initial arch wires for tooth alignment during orthodontic treatment with fixed appliances. Cochrane Database Syst Rev. 2013 Apr 30;4:CD007859. doi: 10.1002/14651858.CD007859.

2. Dayananda GN, Subba Rao M. Effect of strain rate on properties of superelastic NiTi thin wires. Mater Sci Eng A Struct Mater. 2008;486:96–103.
3. Ren Y, Jongsma MA, Mei L, van der Mei HC, Busscher HJ. Orthodontic treatment with fixed appliances and biofilm formation--a potential public health threat? Clin Oral Investig. 2014;18:1711-8
4. Alexander SA, Ripa LW. Effects of self-applied topical fluoride preparations in orthodontic patients. Angle Orthod 2000;70:424-30.
5. Nakagawa M, Matsuya S, Udoh K. Effects of fluoride and dissolved oxygen concentrations on the corrosion behavior of pure titanium and titanium alloys. Dent Mater J 2002;21:83-92.
6. Huang HH. Effects of fluoride concentration and elastic tensile strain on the corrosion resistance of commercially pure titanium. Biomaterials 2002;23:59-63.
7. Kaneko K, Yokoyama K, Moriyama K, Asaoka K, Sakai J. Degradation in performance of orthodontic wires caused by hydrogen absorption during short-term immersion in 2.0% acidulated phosphate fluoride solution. Angle Orthod 2004;74:487-95.
8. Walker MP, Ries D, Kula K, Ellis M, Fricke B. Mechanical properties and surface characterization of beta titanium and stainless steel orthodontic wire following topical fluoride treatment. Angle Orthod 2007;77:342-8.
9. Kwon YH, Seol HJ, Kim HI, Hwang KJ, Lee SG, Kim KH. Effect of acidic fluoride solution on beta titanium alloy wire. J Biomed Mater Res B Appl Biomater 2005;73:285-90.
10. Walker MP, White RJ, Kula KS. Effect of fluoride prophylactic agents on the mechanical properties of nickel-titanium-based orthodontic wires. Am J Orthod Dentofacial Orthop 2005;127:662-9.
11. Kaneko K, Yokoyama K, Moriyama K, Asaoka K, Sakai J, Nagumo M. Delayed fracture of beta titanium orthodontic wire in fluoride aqueous solutions. Biomaterials 2003;24:2113-20.
12. Yokoyama K, Kaneko K, Moriyama K, Asaoka K, Sakai J, Nagumo M. Delayed fracture of Ni-Ti superelastic alloys in acidic and neutral fluoride solutions. J Biomed Mater Res A 2004;69:105-13.
13. Yokoyama K, Kaneko K, Moriyama K, Asaoka K, Sakai J, Nagumo M. Hydrogen embrittlement of Ni-Ti superelastic alloy in fluoride solution. J Biomed Mater Res A 2003;65:182-7.
14. Toniollo MB, Tiossi R, Macedo AP, Rodrigues RC, Ribeiro RF, Mattos Mda G. Effect of fluoride-containing solutions on the surface of cast commercially pure titanium. Braz Dent J 2009;20:201-4.
15. Ramalingam A, Kailasam V, Padmanabhan S, Chitharanjan A. The effect of topical fluoride agents on the physical and mechanical properties of NiTi and copper NiTi archwires. An *in vivo* study. Aust Orthod J 2008;24:26-31.
16. Yokoyama K, Kaneko K, Moriyama K, Asaoka K, Sakai J, Nagumo M. Hydrogen embrittlement of Ni-Ti superelastic alloy in fluoride solution. J Biomed Mater Res A 2003;65:182-7.
17. Staffolani N, Damiani F, Lilli C, Guerra M, Staffolani NJ, Belcastro S, *et al*. Ion release from orthodontic appliances. J Dent 1999;27:449-54.
18. Belcastro S, Staffolani N, Guerra M. Effects of organic acids on corrosion of orthodontic appliances. Minerva Stomatol 2001;50:15-20.
19. Huang HH. Corrosion resistance of stressed NiTi and stainless steel orthodontic wires in acid artificial saliva. J Biomed Mater Res A 2003;66:829-39.
20. Huang HH, Wang CC, Chiu S-M, Wang JF, Liaw YC, Lee TH, *et al*. Ion release from NiTi orthodontic wires in artificial saliva with various acidities. Biomaterials 2003;24:3585-92.
21. Ahn HS, Kim MJ, Seol HJ, Lee JH, Kim HI, Kwon YH. Effect of pH and temperature on orthodontic NiTi wires immersed in acidic fluoride solution. J Biomed Mater Res B Appl Biomater 2006;79:7-15.
22. Kao CT, Ding SJ, He H, Chou MY, Huang TH. Cytotoxicity of orthodontic wire corroded in fluoride solution in vitro. Angle Orthod 2007;77:349-54.
23. Kao CT, Ding SJ, Min Y, Hsu TC, Chou MY, Huang TH. The cytotoxicity of orthodontic metal bracket immersion media. Eur J Orthod 2007;29:198-203.
24. Amini F, Borzabadi Farahani A, Jafari A, Rabbani M. *In vivo* study of metal content of oral mucosa cells in patients with and without fixed orthodontic appliances. Orthod Craniofac Res 2008;11:51-6.
25. Huang HH, Chiu YH, Lee TH, Wu SC, Yang HW, Su KH, *et al.* Ion release from NiTi orthodontic wires in artificial saliva with various acidities. Biomaterials 2003;24:3585-92.
26. Kassab E, Neelakantan L, Frotscher M, Swaminathan S, Maaß B, Rohwerder M, *et al*. (2014), Effect of ternary element addition on the corrosion behaviour of NiTi shape memory alloys. Materials and Corrosion, 65: 18–22.
27. Kassab EJ, Gomes JP. Assessment of nickel titanium and beta titanium corrosion resistance behavior in fluoride and chloride environments. Angle Orthod. 2013;83:864-9.
28. Bogdanski D, Köller M, Müller D, Muhr G, Bram M, Buchkremer HP, *et al.* Easy assessment of the biocompatibility of Ni-Ti alloys by *in vitro* cell culture experiments on a functionally graded Ni-NiTi-Ti material. Biomaterials. 2002;23:4549–4555.
29. Huang HH. Surface characterizations and corrosion resistance of nickel-titanium orthodontic archwires in artificial saliva of various degrees of acidity. J Biomed Mater Res A 2005;74:629-39.
30. Kasuya S, Nagasaka S, Hanyuda A, Ishimura S, Hirashita A. The effect of ligation on the load deflection characteristics of nickel titanium orthodontic wire. Eur J Orthod 2007;29:578-82.
31. Santoro M, Nicolay OF, Cangialosi TJ. Pseudoelasticity and thermoelasticity of nickel-titanium alloys: A clinically oriented review. Part II: Deactivation forces. Am J Orthod Dentofacial Orthop 2001;119:594-603.
32. Kwon YH, Cheon YD, Seol HJ, Lee JH, Kim HI. Changes on NiTi orthodontic wired due to acidic fluoride solution. Dent Mater J 2004;23:557-65.
33. Harris EF, Newman SM, Nicholson JA. Nitinol arch wire in a simulated oral environment: Changes in mechanical properties. Am J Orthod Dentofacial Orthop 1988;93:508-13.
34. Kao CT, Huang TH. Variations in surface characteristics and corrosion behaviour of metal brackets and wires in different electrolyte solutions. Eur J Orthod 2010;32:555-60.

How to cite this article: Alavi S, Barooti S, Borzabadi-Farahani A. An *in vitro* assessment of the mechanical characteristics of nickel-titanium orthodontic wires in Fluoride solutions with different acidities. J Orthodont Sci 2015;4:52-6.

Source of Support: Nil, **Conflict of Interest:** None declared.