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口腔中氟離子對金屬矯正裝置之機械與生物學效應研究(2/2)

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行政院國家科學委員會補助專題研究計畫期中進度報告

（計畫名稱）

口腔中氟離子對金屬矯正裝置之機械與生物學效應研究

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中文摘要

口腔是一個複雜的環境，氟化物常被用於預防蛀牙或牙菌斑之發生。金屬矯正裝置的種類依支架內金屬各種元素含量的不同以及廠牌不同特性的要求而有差異。本研究第一年目的：比較金屬矯正支架與金屬矯正線於含氟化物下之腐蝕性速率變化。第二年研究目的；由腐蝕之金屬矯正裝置釋出物（即金屬離子溶液），對於生物體之生物相容性。第一年研究方法與材料：本研究以不同之矯正支架與不同尺吋之矯正金屬線經表面處理後，分別以電化學腐蝕方法處理，以不同之電解液體（人工唾液、含氟溶液）分析其腐蝕之現象；以原子吸收光譜儀分析經電化學處理後之電解液體其金屬離子釋放量。結果以ANOVA分析，以Student newman Keul做事後分析比較。結果發現：於極化曲線下矯正支架與矯正金屬線均會發生腐蝕現象。不同廠牌之金屬矯正支架有不同量之金屬離子釋出。於pH 4人工唾液與pH3.5 APF條件下鎳離子、鉻離子與鐵離子之釋出量增加($P<0.05$)。於pH4 人工唾液和 pH 3.5 NaF溶液下， NiTi金屬線釋出之鎳離子濃度較不銹鋼線釋出之鎳離子濃度高($P<0.05$).。鉻離子濃度只於pH 3.5 NaF溶液下，呈現出差異性($P<0.05$)。鈦金屬離子只於pH 3.5 NaF溶液下，呈現出差異性($P<0.05$)。第二年研究方法與材料：將金屬矯正支架釋出溶液調整濃度為0.01, 0.1, and 1.0 μl/ml.分別加入到primary human oral gingival fibroblast (HGF)和Human osteogenic sarcoma cell line (U2OS)培養皿，以MTT分析細胞之存活率及觀察細胞之型態變化。結果發現：於pH4條件下，Unitek (p=0.003)和Ormco (p=0.000)之U2OS和HGF細胞存活率有差異性，於pH7條件下，則Unitek (p=0.03)和Dentaurum(p=0.021) 之U2OS和HGF細胞存活率有差異性。結語：含氟化物之溶液對於矯正金屬具有腐蝕效應，會將金屬離子釋出。而矯正支架釋出之金屬離子溶液隨廠牌不同而有不同之細胞毒性反應。後續之研究將可繼續進行該釋出物對於牙齦之癌性反應，以利了解矯正治療過程中牙齦發炎之機轉。

關鍵詞：金屬矯正支架、金屬矯正線、氟化物、腐蝕效應、金屬離子、細胞相容性

英文摘要

To prevent the dental caries and maintain the oral hygiene in orthodontic patient, fluoride prophylaxis material is routinely applied on them. The purpose of the first year study was to evaluate the corrosion of orthodontic metals under fluoride solution treatment. The purpose of the second year was to evaluate the cytotoxic effects of four different metal bracket immersion media on primary human oral gingival fibroblasts (HGFs) and one permanent human osteogenic sarcoma cell line (U2OS). Materials and methods: Different brands of metal bracket and different size of metal wire were treated by electrochemical method with various electrolytes. They were acidified NaF, pH4 and pH6.75 artificial saliva. The atomic absorption machine was to analyze the metal ions released from these corrosive electrolytes. In biocompatibility study, Four different metal brackets (Unitek, Tomy, Ormco, and Dentaurum) were immersed in buffer solutions of $NaHNO_3$ (1 mM) with a pH of 4 or 7, as well as artificial saliva. The concentrations for the experiments were 0.01, 0.1, and 1.0 μ l/ml. At the end of the period of bracket immersion, morphological observations were conducted using light microscopy. The tetrazolium reduction assay was used to detect the survival rate of the target cells. Data were analyzed by one way analysis of variance (ANOVA), with Student-Newman-Kewul test to detect the difference under $p<0.05$. Results: the present study showed all test samples existed corrosion under different medium. Different brands of metal brackets were released different amount of metal ions. In pH4 and pH3.5 NaF solution, Ni ion Fe ions and Cr ion concentrations increased.($p<0.05$) In pH4 and pH3.5 NaF solution wire test, Ni ions release higher in NiTi wire than that in stainless steel wire. ($p<0.05$) Cr ion exist statistic difference on pH3.5 NaF condition. ($p<0.05$) Similar result existed on Ti ion as in Cr ion group. ($p<0.05$) At pH 4, the survival rates of the U2OS cells and the HGFs differed statistically for the Unitek and Ormcogroups. ($P<0.05$) At pH 7, the survival rate for the HGFs and the U2OS cells differed statistically for the Dentaurum and Unitek groups ($P<0.05$). Conclusion: Fluoride material will corrode the orthodontic metal appliance and release different amount of metal ions. The elute solution will cause the HGF and U2OS cytotoxicity.

keywords:metal bracket, orthodontic wire, fluoride, corrosion, metal ions, biocompatibility, HGF, U2OS

Introduction

In the oral environment, orthodontic attachments are exposed to a number of potentially damaging physical and chemical agents, and such conditions possibly contribute to corrosion of the metal components of appliances.[1] Orthodontic bands, brackets, and wires are universally made of austenitic stainless steel containing approximately 8%~12% nickel and 17%~22% chromium.[2,3] Many studies on the corrosion resistance of metal appliances used for dental orthodontic applications have been conducted.[4-6] A previous study demonstrated that metal ions are released from an orthodontic metal bracket at pH 4.[7] A number of studies have also demonstrated that metal ions are released by all dental alloys *in vitro* and *in vivo*.[8,9]

Orthodontic patients are referred to general dentists for fluoride treatments once every 6 months during the course of orthodontic mechanotherapy. Increasingly, dental gels and resins containing fluoride are being applied to prevent dental caries. Fluoride levels in the oral cavity vary according to the prophylactic treatment. Fluoride levels in toothpastes and mouth rinses can reach 1%, and for eliminating enamel stains, can be close to 2%; these substances have a pH range of between about 3.5 and 7.0.[10] Since the oral environment is particularly favorable for the biodegradation of metal due to its ionic, thermal, microbiological, and enzymatic properties, it can be presumed that to a certain extent, patients are exposed to the products of corrosion processes.[11]

Nickel is one of the most-common causes of allergic contact dermatitis.[12] The incidence of nickel hypersensitivity within the general population is reported to be as high as 20%~30%, with case reports of nickel hypersensitivity commonplace within the biomedical literature.[12-14] Adverse reactions related to nickel-containing orthodontic devices such as arch wires, brackets, and buckles on headgear devices have been observed.[15-17] Particularly interesting were two clinical studies which claimed that the use of nickel-titanium arch wires can convert nickel-nonsensitive subjects into nickel-sensitive subjects, with an approximately 20% conversion rate.[18,19]

The purpose of the first year study was to evaluate the corrosion of orthodontic metals under fluoride solution treatment. The purpose of second year study was to compare the cytotoxic effects of four different orthodontic metal bracket immersion media on primary human oral gingival fi broblasts (HGFs) and one permanent human osteogenic sarcoma cell line (U2OS).

Materials and Methods

Four major orthodontic wires, namely, 0.016-in (0.41mm) nickel-titanium (NiTi) wire (Unitek, 3M, CA, USA), 0.016 x 0.022-in (0.41 x 0.56 mm) Ni-Ti wire (Unitek, 3M, CA, USA), 0.014-in (0.36 mm) stainless steel wire (SSW) (Unitek, 3M, CA, USA), and 0.016 x 0.022-in (0.41 x 0.56 mm mm) SSW (Unitek, 3M, CA, USA), and four major orthodontic brackets, namely, a Unitek DynaLock twin-torque bracket (Unitek, 3M, CA, USA), Tomy metal base bracket (Tomy Co, Tokyo, Japan), Ormco standard edgewise bracket (Ormco Co., CA, USA), and Dentarum Rickett bracket (Dentarum Co., Germany), were tested in this study. The wires were cut into 50-mm-long specimens. Each sample consisted of five pieces for testing. All materials were cleaned by swabbing with acetone and placing in an ultrasonic container with distilled water for 10 min before testing.

The electrochemical corrosive breakdown of the metal brackets and wires was initiated by applying a method based on Shih et al.'s description.[20] Three electrolytes were used in the corrosive reaction. First, artificial saliva (Table 1) containing 0.2% acidulated phosphate fluoride (NaF; 0.2% NaF + 0.17% H_3PO_4, adjusted to pH 3.5 with lactic acid) was used as the electrochemical corrosive electrolyte and was maintained at 37 °C. In the second and third cases, the electrochemical corrosive electrolytes were adjusted to pH 4 and pH 6 using lactic acid in artificial saliva. A cyclic potentiodynamic polarization machine was applied from -800 mV in the anodic direction with a scan rate of 1 mV/s after dipping the specimen into the electrolyte for 1 h. Each cyclic potentiodynamic polarization curve was printed out. The global polarization curves were assessed to determine the corrosion and breakdown potentials. One-way analysis of variance (ANOVA) was used to make the comparison with $p < 0.05$ accepted as showing a statistically significant difference. The Student-Newman-Keuls test was used for multiple comparisons. A scanning electron microscope (SEM) (ABT-150S, Topcon, Tokyo, Japan) was used to observe the surface morphologies of the metal brackets and wires.

Metal ions release analyses

The method was followed our previous study.[21] Extracts were added to polypropylene tubes containing SS wire or NiTi wire. The solution was analyzed for nickel (Ni), chromium (Cr) and titanium (Ti) ions. Standards were prepared in equivalent solutions to counteract any buffer effects. The pre-treatment and atomization temperatures as recommended by Perkin-Elmer were used in the furnace programs to ensure that linear standard curves were obtained for each element. Each sample was analyzed for all three ions and concentrations, measured as $\mu g/cm^2$, averaged across the five replicates. Results were compared using the one-way analysis of variance (ANOVA). Differences in treatment means were analyzed using the Student-Newman-Keul's test and were considered to be significant at $p < 0.05$.

Sample preparation

Four different brands of metal bracket were analysed (Table 1). The method of sample preparation followed that of a previous study (Huang *et al.* , 2004). For each brand, a total of 160 brackets were tested. The brackets were immersed in the relevant solutions and incubated at a temperature of 37°C for a period of 48 weeks. The buffer solutions included NaHNO 3 (1 mM), with a pH of 4 or 7, as well as artifi cial saliva (Sinphar, Taipei, Taiwan; Table 2). The concentrations for the experiments were 0.01, 0.1, and 1.0 μl/ml.

Cell cultures

Human primary gingival fi broblast culture. The research was approved by the ethical board of Chung Shan Medical University Hospital. Following informed consent, gingival tissues were obtained by excision of premolar gingiva from a 12-year-old female patient undergoing orthodontic treatment. The resultant tissue was cut into 1- to 2-mm 3 sized pieces, washed twice with phosphate-buffered saline supplemented with penicillin (100 U/ml; Sigma Chemical Co., St Louis, Missouri, USA), streptomycin (100 μ g/ml, Sigma Chemical Co.), and placed into 25 cm 3 tissue-culture fl asks. The explants were incubated with culture medium consisting of alpha minimum essential medium (Sigma Chemical Co.), 30 per cent foetal bovine serum (FBS; Sigma Chemical Co.), penicillin (100 U/ml), and streptomycin (100 μ g/ml), at 37°C in a humidified atmosphere of 5 per cent CO 2 in air. When outgrowth cells were observed in the cultures, the medium was replaced twice, sequentially, and the cells were then reincubated until the proliferating cells had reached confl uence. The cells were detached from the monolayer by brief treatment with 0.02 per cent trypsin/0.04 M ethylenediaminetetraacetic acid (EDTA) and recultured in 100 cm 2 tissue-culture fl asks until confl uent monolayers were again obtained. Cells between the fi fth and the seventh passages were used in the subsequent experiments.

Human osteogenic sarcoma cell culture.

The U2OS cell line (BCRC no. 60187, Food Industry Research and Development

Institute, Taiwan) was used. Briefl y, the cells were cultured in McCoy's medium (Sigma Chemical Co.) containing 10 per cent FBS and penicillin, streptomycin, and fungizone, and L -glutamine (1 per cent; Sigma Chemical Co.). The cultures were maintained at 37°C in a humidified atmosphere of 5 per cent CO_2 in air. Confl uent cells were detached with 0.025 per cent trypsin and 0.05 per cent EDTA for a period of 5 minutes, following which, aliquots of separated cells were subcultured. Cells were cultivated as monolayers in plastic culture fl asks.

Cell viability test — tetrazolium reduction assay

The tetrazolium reduction (MTT) [3-(4,5-dimethylthiazol- 2-yl)-2,5-diphenyl tetrazolium bromide] colorimetric assay, a measure of succinic dehydrogenase activity, was performed following the method of Mossman (1983) . HGF and U2OS cells were inoculated into 96-well plates (Falcon, Teterboro, New Jersey, USA) at a density of 4×10^3 cells per well, and incubated at 37°C, in 5 per cent CO_2 -in-air for a period of 2 days. The cells were then incubated under identical conditions to the above for a further 3 days. Subsequent to incubation, the cells were treated with various concentrations of metal bracket extracts, following which MTT dye (50 μ g/l) was added to each well. The plates were incubated at 37°C, and 5 per cent CO_2 in air for a period of 4 hours. For each well, the degree of light absorbance at 570 nm was then measured using an enzyme-linked immunosorbent assay reader (U2000, Hitachi, Tokyo, Japan). The cell viability results were presented as the ratio (in per cent) of the absorbance at 570 nm in the experimental wells to that detected in the control wells. Five replicates of each concentration were used for each test. All assays were repeated three times to ensure reproducibility. Statistical analysis was conducted using the SAS program for Unix 6.09 (SAS Institute, Cary, North Carolina, USA) using one-way analysis of variance, with a value of $P < 0.05$ showing statistical difference.

Result

Results of the electrochemical analysis of metal bracket cyclic potentiodynamic polarization curves are shown in figure 1. The corrosion tendencies of different brands of brackets were statistically compared, and the corrosion potential (voltage, mean ± standard deviation) sequence from strong to weak was: Ormco, NaF (-0.451 ± 0.087 V) = pH 4 (-0.438 ± 0.093 V) > pH 6 (-0.324 ± 0.118 V) ($F = 4.86$, $p = 0.016$); Unitek, NaF (- 0.412 ± 0.095 V) = pH 4 (0.417 ± 0.192) > pH 6 (-0.082 ± 0.018 V) ($F = 11.96$, $p = 0.001$); Dentaurum, NaF (-0.367 ± 0.069 V) = pH 4 (-0.407 ± 0.081 V) > pH 6 (-0.286 ± 0.052 V) ($F = 4.06$, $p = 0.045$); and Tomy, pH 4 (-0.346 ± 0.063 V) > NaF (-0.271 ± 0.051 V) = pH 6 (-0.256 ± 0.046 V) ($F = 4.02$, $p = 0.046$) (Figure 1).

Results of the electrochemical analysis of wires using the cyclic potentiodynamic polarization curves are shown in figure 2. The corrosion tendencies of the different wires are indicated by the corrosion potential (voltage, mean ± standard deviation) sequence from strong to weak: 0.016 x 0.022-in SSW, pH 4 (-0.402 ± 0.101 V) = pH 6 (-0.397 ± 0.189 V) > NaF (-0.09 ± 0.013 V) ($F = 10.39$, $p = 0.002$); 0.014-in SSW, pH 4 (-0.425 ± 0.105 V) = pH 6 (0.401 ± 0.101 V) > NaF (-0.121 ± 0.009 V) ($F = 20.11$, $p = 0.000$); 0.016-in NiTi, pH 4 (-0.358 ± 0.097 V) = pH 6 (-0.345 ± 0.106 V) > NaF (-0.126 ± 0.078 V) ($F = 9.54$, $p = 0.003$); and 0.016 x 0.022-in NiTi, pH 4 (-0.387 ± 0.095 V) = pH 6 (-0.316 ± 0.102 V) > NaF (-0.067 ± 0.019 V) ($F = 21.41$, $p = 0.000$) (Figure 2).

Results of the SEM morphological observations of the different brands of brackets in different media are shown in figure 3. The bracket surface of all brands of the normal artificial saliva group indicated that surface defects were related to the bracket milling, pickling, or electropolishing procedures performed during the manufacturing process. In the pH 6 group, surface defects were similar to those of normal artificial saliva group. In the pH 4 group, bracket surface defects and pitting corrosion were observed. In the NaF group, pitting corrosion and defects were similar to those of the pH 4 group.

Results of the SEM morphological observations of the different wires in different media are shown in figure 4. They indicated that the wire surface defects were related to the wire drawing, pickling, or electropolishing procedures performed during the manufacturing process. The wire surface of the normal salivary group, for both the stainless steel and nickel titanium wires, exhibited scratches and pits on the surface (Figure 4a). The surfaces of the nickel-titanium wires immersed in pH 4 and NaF media became rough and pitted because of corrosion. (Figure 4c and d for the 0.016 x 0.022-in NiTi and 0.016-in NiTi groups, respectively) The surfaces of the stainless steel wire groups exhibited scratches and pitting corrosion with pH 4 and NaF treatments (Figure 4).

Corrosive metal ions

The metal bracket release ions were shown in table 2 A to C. The results of atomic absorption analysis of different medium are presented in table 3 A to C. The release of ionic nickel was showed statistical difference in all groups ($P<0.05$). In pH 6.75 artificial saliva medium, the release of nickel ion was higher from the SSW than from the NiTi ($P<0.05$). In pH4 artificial saliva medium or pH 3.5 NaF medium, the release of nickel ion was higher from the NiTi than from the NiTi ($P<0.05$). Analysis of released chromium ions revealed that there was only difference in pH 3.5 NaF medium group ($P<0.05$). Analysis of released titanium ions revealed that there was only difference in pH 3.5 NaF medium group ($P<0.05$).

Morphological observation

The morphology of the cells appeared to have been maintained subsequent to metal bracket immersion media exposure. Cell membranes appeared to be intact with no obvious damage or apoptosis (Figures 5 and 6).

Cytotoxicity of pH 4 metal bracket immersion media evaluated by MTT assay

The dose-response curve showed a dose-dependent increase in toxicity for the Unitek (P = 0.03) (Figure 8) and Ormco (P = 0) (Figure 10) groups, but not for the Dentaurum (P = 0.667) (Figure 7) or Tomy (P = 0.138) groups (Figure 9). The greatest cell survival rate (in per cent) for the Unitek group was noted at a concentration of 0.01 μ l/ml for the HGF cell group (89.48 ± 5.37 per cent) while the lowest survival rate was observed at a concentration of 1 μ l/ml for the U2OS cell line (74.76 ± 4.89 per cent). The greatest cell survival rate (in per cent) for the Ormco group was noted at a concentration of 0.01 μ l/ml for the U2OS cell line (90.32 ± 8.99 per cent) while the lowest survival rate for the HGF cell group was observed at a concentration of 1 μ l/ml (69.42 ± 4.77 per cent).

Cytotoxicity of pH 7 metal bracket extracts evaluated by the MTT assay

The dose-response curve refl ecting the level of relative cytotoxicity of the metal bracket immersion media appeared Dentaurum (P = 0.021) and Unitek (P = 0.03) (Figure 8) groups but not for the Tomy (P = 0.054) (Figure 9) or Ormco (P = 0.06) groups (Figure 10). The greatest cell survival rate for the Dentaurum group (110.43 ± 8.38 per cent) (Figure 7) was observed at a concentration of 0.01 μ l/ml for the U2OS cell group, while the test cell survival rate appeared to be similar for the other remaining metal bracket groups. The greatest cell survival rate (in per cent) for the Unitek group was observed at a concentration of 0.01 μ l/ml for the U2OS cell group (104.83 ± 10.74 per cent).

Discussion

It has been reported that various brands of new brackets exhibit differences in corrosion behavior.[22] The present results revealed similar findings, i.e., the surfaces of brackets and wires in different media exhibited various degrees of roughness. AISI type 316L austenitic stainless steel alloy is currently used for bracket manufacturing.[23] Stainless steel owes its corrosion resistance property to chromium, a highly reactive base metal. The alloy's corrosion resistance depends on a passive film, which spontaneously forms (passivation) and reforms (repassivation) in air and under most tissue fluid conditions.[23] Oxygen is necessary to form and maintain the film, whereas acidity and chloride ions can be particularly detrimental to it. [24]

From the potentiodynamic polarization curves, it is apparent that most brands of metal brackets showed higher corrosion tendencies in the pH 4 and NaF media (Figure 1). It is known that corrosion of orthodontic alloys occurs in the intraoral environment, regardless of the alloy's metallurgic structure, and it is also known that the presence of manufacturing defects may accelerate the process.[24] The morphologies of the brackets by SEM showed already existing surface defects in the normal artificial saliva group in the present study (Figure 2). In an acidic condition, corrosion in the form of pitting was identified. Just as with Cl^- ions, fluoride ions (F^-) may penetrate into the metal/oxide film interface.[25] This evidence corresponds with our present results for the NaF group, in that defects and pitting corrosion also existed on the metal bracket surface (Figure 3). As it is known that stainless steel will release nickel ions after corrosion occurs, a disadvantage with stainless steel bracket corrosion concerns patients with allergies to nickel and other specific substances. From the present results, we recommend that substances with acidic or fluoride contents should be used with caution in patients undergoing orthodontic treatment. Titanium brackets were found to have reduced pitting and crevice corrosion.[25] Replacing stainless steel brackets with titanium ones should be considered.

Surface irregularities observed on the NiTi arch wires may arise from the manufacturing process.[26] Thus it was found in the normal salivary group that the NiTi wire surface showed irregularities or roughness in the SEM observations (Figure 4). The present electrochemical studies indicated that pitting corrosion of NiTi wires occurred in a pH 4 solution. The mechanism of hydrogen penetrating the NiTi wire was proven. [27] Acid treatment causes the wire to become brittle, and under stress, the NiTi wire may fracture.[27] Usually titanium forms several oxides (during passivation, it forms TiO_2, TiO, and Ti_2O_5). The NiTi wire shows increased corrosion resistance. When Ti is exposed to water, TiO_2 is expected to form according to the reaction, $Ti + 2H_2O \rightarrow TiO_2 + 2H_2$. During this reaction, H+ ions are produced, increasing the pH. The resulting OH^- anions are adsorbed onto the surface, where they create an electrical field for ion migration and subsequent oxide growth.[28] This mechanism can explain the present results of SEM morphological observations, as the surfaces of the NiTi wires in the pH 4 group showed defects (Figure 4). The improper acid corrosion of NiTi wires increases the risk of wire fracture under the stresses of orthodontic treatmetn.

Fluoride ions can cause the breakdown of the protective passivation layer that normally exists on titanium and its alloys, leading to pitting corrosion.[29] In the present study, the NaF corrosion potential was lower than pH 4 or pH 6 on the potentiondynamic curves (Figure 2). This indicates that wires in the NaF medium still corroded, but the corrosion resistance was stronger than that of the NiTi wire in the pH 4 or pH 6 groups. But according to the SEM observations, the NiTi wire corrosion defects in the NaF group were more obvious than those in the pH 4 or pH 6 groups. That is probably because titanium easily dissolves in hydrofluoric acid [HF] which creates surface defects.[30]

The present results showed that the surface of the stainless steel wire groups exhibited scratches and pitting corrosion under pH 4 and NaF treatments. This is similar to reports that 316 stainless steel in an acetic acid solution containing F^- ions showed pitting corrosion.[31] The mechanism involves penetration of F^- ions into the metal/oxide film interface.[29] In the present study, the surfaces of the brackets or wires showed roughness or defects before testing, and the corrosive potentials of stainless steel and nickel titanium were similar. Thus after specimens were treated in the corrosive media, the surface defects became more severe and obvious. These results are the same as other reports.[32,33] The reason might be that metallic materials are not susceptible to corrosion as long as the surface oxide film is intact. But when the breakdown potential of an alloy is reached, the oxide layer dissolves, and surface corrosion and pitting begins.

It was reported that NiTi superelastic alloy exhibits good corrosion resistance in saliva and saline solutions.[34] The titanium alloy forms titanium oxide which resists corrosion. Certain nickel-titanium arch wires are manufactured using an ion implantation technique. Nitrogen ions are introduced into the near-surface region of the arch wires in an attempt to reduce the amount of friction occurring between brackets and arch wires. The coating probably also increases the corrosion resistance of the wire. [35-37] But Yokoyama et al.'s study showed that when stress is applied to NiTi, corrosion can still occur.[34]

The corrosion resistances of metal brackets and wire were analyzed by electrochemical methods in the present study. Most brands of metal bracket were easily corroded in the NaF and pH 4 environments. Potentiodynamic curves showed that NiTi and stainless steel wires were easily corroded in pH 4 artificial saliva. According to the SEM morphological observations, the bracket and wire surfaces showed defects or pitting corrosion in all tested media. The extent of surface roughness might influence the friction. How the rough corroded surfaces of brackets and wires influence the orthodontic tooth sliding movement is the next step for further investigation. Care must be exercised when fluoride-containing prophylactic agents are used on orthodontic patients.

Dental materials used in the oral environment are subject to electrochemical and chemical reactions, mechanical forces of mastication, and wear. Since orthodontic metal brackets are typically located proximate to periodontal tissue in the oral environment, it is critical to determine the relative level of biocompatibility of the various metal brackets in such an environment. The previous our study found that metal brackets may corrode in such an oral environment and metal-ion leaching may occur.[35] The biologic reaction of the metal bracket extracts is needed to evaluate. The extraction assay described above would appear to be one of the most frequently used methods to investigate the mechanism of intra-oral cytotoxicity in regard to the study of dental materials and their oral environmental interaction[36] The MTT assay is often used to evaluate the activity of mitochondrial succinic dehydrogenase by measuring the amount of formazan produced by this enzyme. [37,38] In present study, we chose this method to evaluate the relative toxicity to tested cells of orthodontic metal-bracket extracts.

Various authors have shown that human primary gingival cells can provide a more-sensitive and discriminating cultured-cell model for the cytotoxic assessment of dental materials than various permanent cell lines originally derived from animal tissue.[39,40] In 1994, Andreotti et al. did note that the resistance to dental material toxicity of normal cells is likely to be greater than that for cell lines,[41] it being suggested that this is due to the high growth-rate conditions in which cell lines are cultured. In present study we chose the primary cultured gingival fibroblast cell and the U2OS cell line which we believe was a representation of the alveolar bone, in order to detect the biocompatibility of the four different orthodontic metal bracket extracts. Comparing the results for the Ormco HGF group with those for the Ormco U2OS group, the latter's survival rate (83.44 ± 5.38 %) proved to be statistically greater than the corresponding result for the HGF group ($p<0.05$). Our findings have demonstrated that cells of different origins reveal a different cellular reaction to contact with foreign bodies. From this result, HGF would appear to be more sensitive to Ormco metal-bracket extracts, it revealing similarity to the 1994 findings of Andreotti et al..[39]

Our previous study revealed that a greater number of metal ions were released into solution from the metal brackets placed in the pH = 4 extract than was the case for the pH = 7 extract. [42] The present results revealed that HGFs treated with Ormco metal-bracket extract at a concentration of 1 μl/ml and at a pH of four reflected the overall lowest cell survival rate (69.42 ± 4.77 %). Interestingly, form our previous study showed that the immersed Ormco metal bracket group was responsible for eliciting the most-substantial nickel concentration of all tested metal bracket groups. (260.5 ± 17.9µg/ml).[42] Viewing other studies, it has been revealed that nickel ions present in bracket extracts were able to enter test cells in a number of different ways.[38-40] Essentially, under such circumstances, the nickel ions would bind with several biological compounds, and thus decrease the extent of a number of cellular functions,[43,44] including succinic dehydrogenase activity and protein synthesis.[44] The higher nickel content of the Ormco metal bracket should be the cause of the detected lowest survival rate for HGF cells exposed to metal-bracket extracts.

The metal bracket fabrication may be welded or brazed together. Since the brazing alloys generally consist of silver and cooper and sometimes palladium. The research showed that brazing alloy is more cytotoxic than stainless steel on gingival fibroblast.[44] The present result has revealed that for the low concentrations of Tomy metal-bracket extract (0.01 and 0.1 μl/ml), under either pH = 4 or pH = 7 conditions, the survival rate for HGF and U2OS cells was greater than 100%, such results suggesting that the Tomy metal bracket actually contributed to a minor exposure-related proliferative response to the test cells. Our previous study revealed that the Tomy metal bracket released lower concentrations of metal ions such as nickel, chromium and copper into the immersing solution than was the case for the Unitek, Ormco and Dentaurum metal bracket.[35] Such a result can explain why the Tomy metal bracket was relatively biocompatible with U2OS cells and HGFs.

The morphological changes revealed by the U2OS cells and by HGFs following treatment with the four different metal bracket extracts did not appear to include any obvious cellular alterations. From microscopic observation, there appeared to be no evidence of any apoptotic change or necrosis, neither cell membrane demonstrating any evidence of bulb formation or destruction in the treated cells. The result suggesting that these four types of metal bracket are biocompatible. Further, according to the results obtained from mitochondrial activity and morphology investigations, the four different types of orthodontic metal bracket demonstrated a good biocompatibility with the U2OS cells and HGF.

CONCLUSION

The metal bracket can release the different concentrations of metal ions. The biocompatibility of the four types of metal bracket tested was evaluated using two kinds of cell. The study demonstrated that cells of different origins exhibit different cellular responses to exposure to metal bracket extracts, although the four kinds of brackets do appear to be biocompatible with HGF and U2OS cells.

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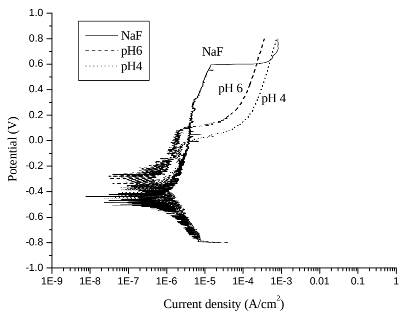
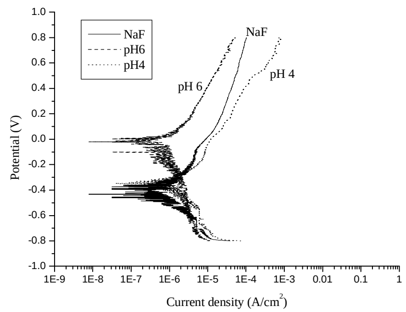
圖 1. 金屬矯正支架於電化學腐蝕試驗—極化曲線圖。

Figure 1.The electrochemical analysis. Polarization curves of metal brackets in pH4, pH6.75 , and pH 3.5 NaF artificial saliva.

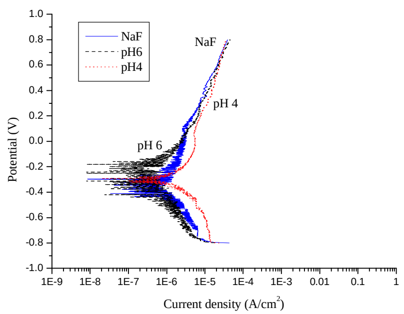
Ormco



Unitek



Dentaurum



Tomy

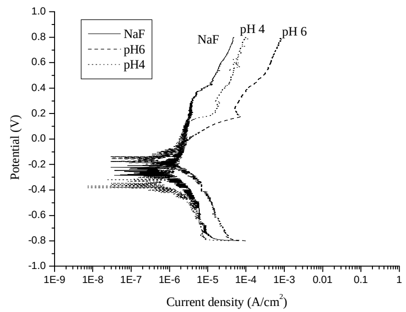
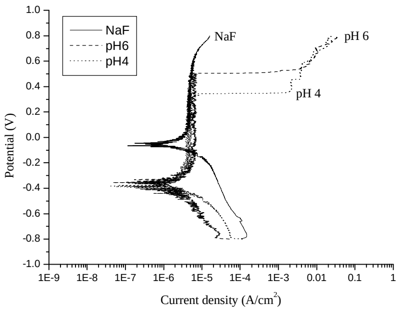


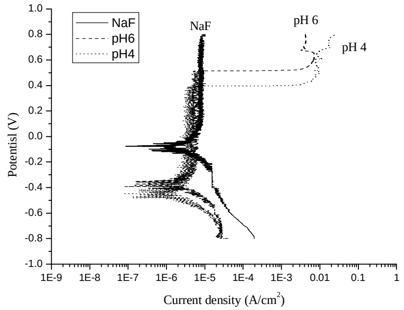
圖 2. 金屬矯正線於電化學腐蝕試驗。

Figure 2.The electrochemical analysis. Polarization curves of stainless steel wire and nickel titanium wires in pH4, pH6.75 , and pH 3.5 NaF artificial saliva.

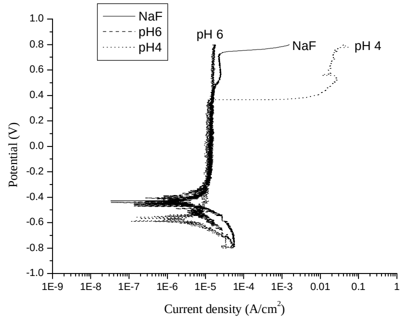
0.016x0022' SSW



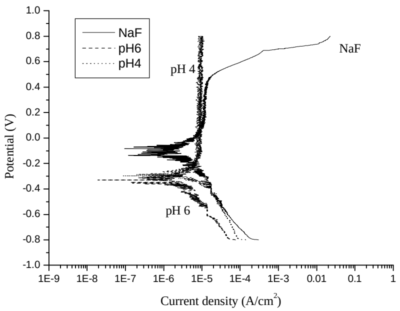
0.014' SSW



0.010' SSW



0.016' NiTi



0.016 x 0.022' NiTi

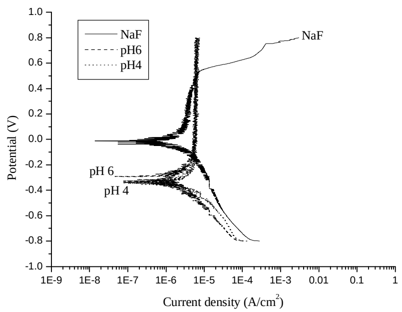


圖 3. 金屬矯正支架於電化學腐蝕試驗後經電子顯微鏡（SEM）觀察。

Figure 3. The scaner electron microscope surface morphologic observation of different brands bracket in different medium treatment. A. in normal artificial saliva. B. in pH6.75 artificial saliva. C. in pH 4 artificial saliva. D. in pH3.5 NaF artificial saliva.

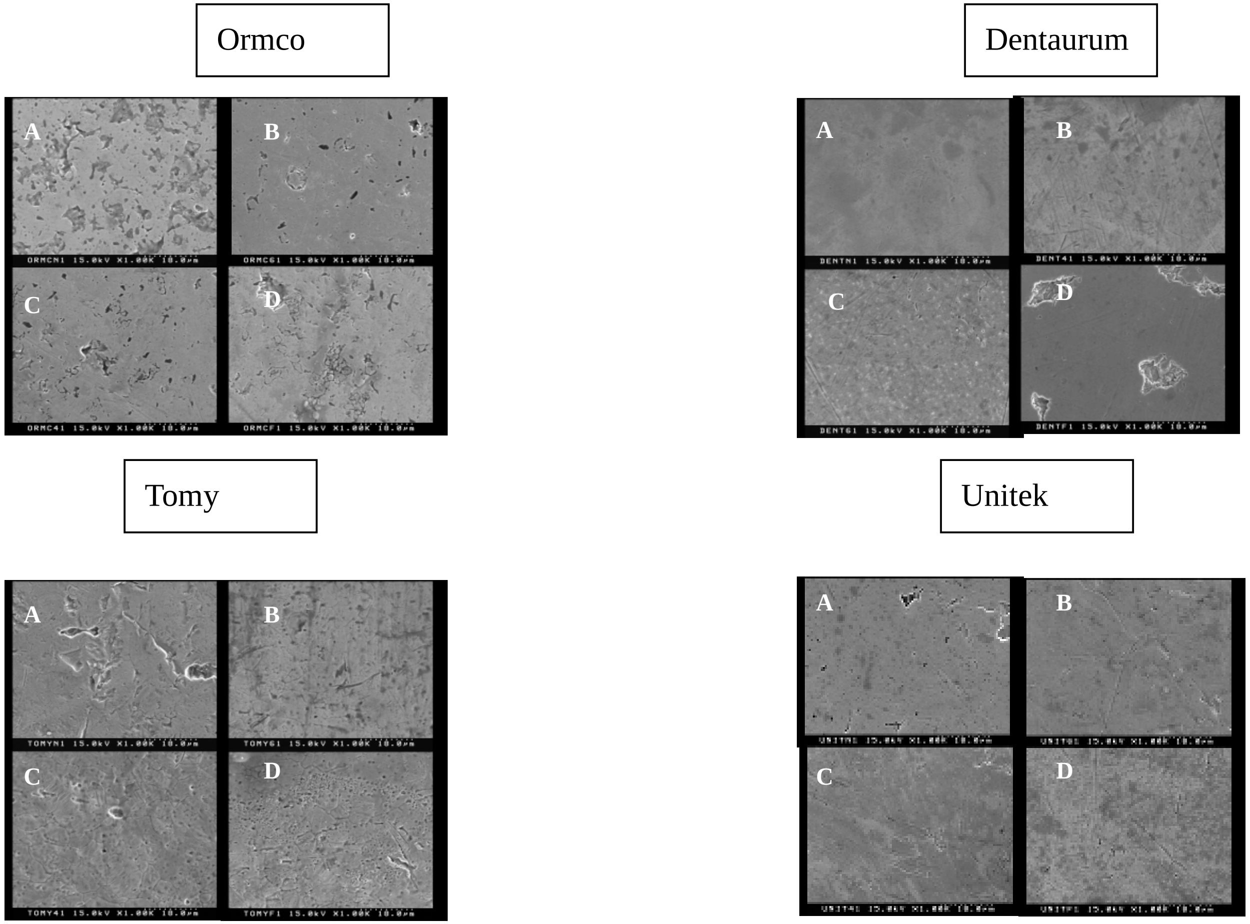


圖 4. 金屬矯正線於電化學腐蝕試驗後經電子顯微鏡（SEM）觀察。

Figure4 . The scaner electron microscope surface morphologic observation of stainless steel wire and nickel titanium wire in different medium treatment. A. in normal artificial saliva. B. in pH6.75 artificial saliva. C. in pH 4 artificial saliva. D. in pH3.5 NaF artificial saliva.

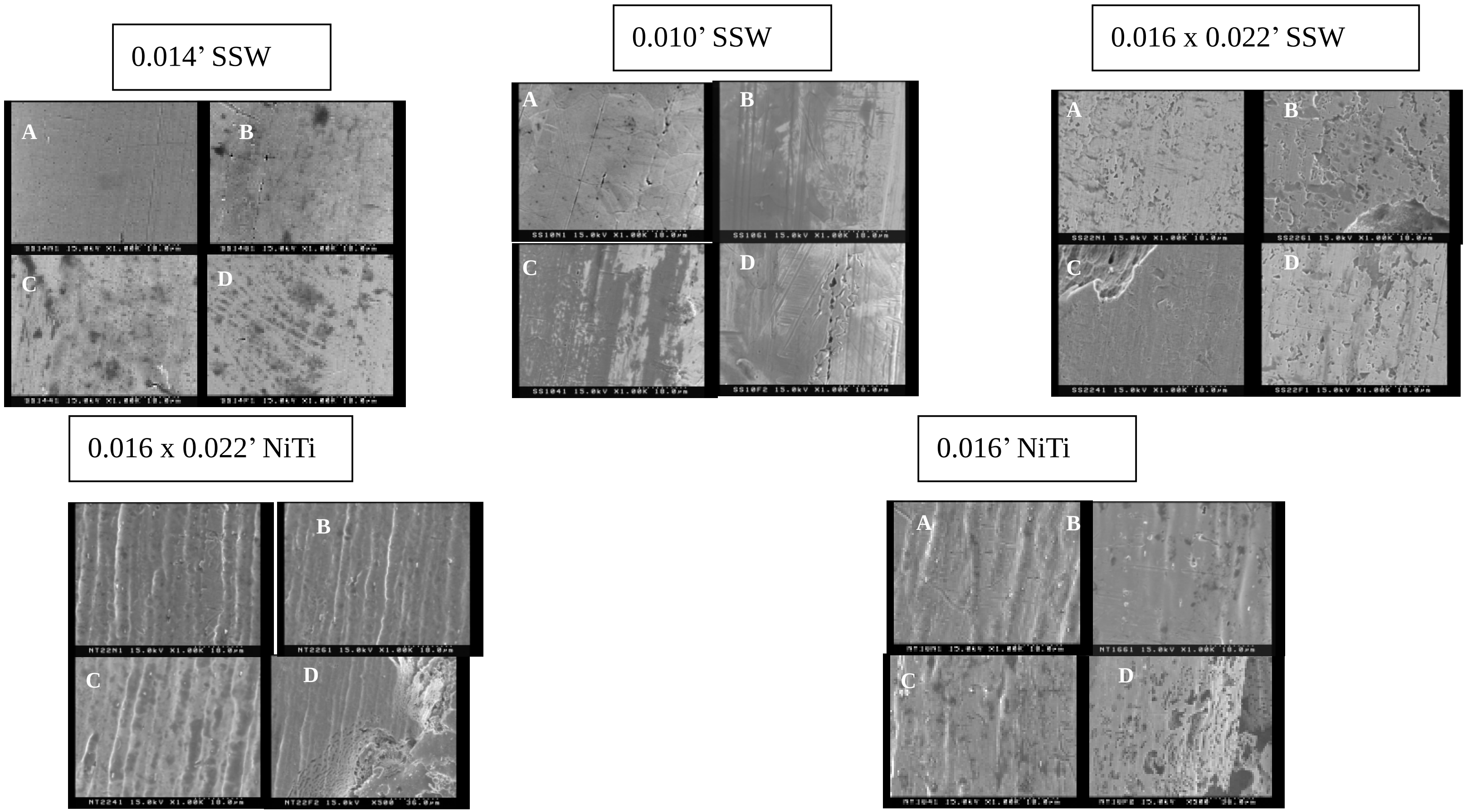


Table I. Contents of the artificial saliva.

Company	Sinphar Pharm, Taipei, Taiwan	
Content	Sali Lube (Saliva substitute)	
	Sodium Chloride	0.844 mg
	Potassium Chloride	1.2 mg
	Calcium Chloride Anhydrous	0.146 mg
	Magnesium Chloride 6 H2O	0.052 mg
	Potassium Phosphate dibasic	0.34 mg
	Sorbitol Solution 70%	60 mg
	Methyl Paraben	2 mg
	Hydroxyethyl Cellulose	3.5 mg

表 **2A.** 矯正支架之原子吸收光譜分析

2A. pH 6.75 AS metal bracket (μg/L)

		Ni	Cr	Fe	Co	Ti	Cu
Dentaurum	Mean	38.65	12.98	8.32	1.23	--	14.34
	SD	4.38	2.23	1.26	0.22	--	2.98
Unitek	Mean	56.83	13.13	7.99	0.57	--	12.77
	SD	3.71	1.45	1.12	0.12	--	3.24
Tomy	Mean	45.31	11.98	9.56	0.10	--	13.13
	SD	5.29	2.31	2.95	0.03	--	2.96
Ormco	Mean	86.73	13.86	12.28	0.55	--	17.96
	SD	8.34	2.32	1.37	0.18	--	3.92
Control		0.28±0.15	0.24±0.13	0.73±0.65	0	--	0.32±0.13

表 **2B.** 矯正支架之原子吸收光譜分析

2B. pH 4 AS metal bracket

		Ni	Cr	Fe	Co	Ti	Cu
Dentaurum	Mean	82.98	22.78	12.38	3.45	--	16.60
	SD	7.26	4.12	2.93	0.83	--	2.37
Unitek	Mean	102.65	28.96	15.38	1.35	--	20.32
	SD	20.21	7.32	3.21	0.21	--	2.98
Tomy	Mean	129.65	17.89	16.28	1.73	--	21.28
	SD	10.22	8.44	2.98	0.27	--	3.22
Ormco	Mean	289.74	21.76	21.57	2.36	--	41.27
	SD	24.37	7.21	3.19	0.30	--	6.35
Control		0.28±0.15	0.24±0.13	0.73±0.65	0	--	0.32±0.13

表 **2 C.** 矯正支架之原子吸收光譜分析

2C. pH 3.5 APF metal bracket

		Ni	Cr	Fe	Co	Ti	Cu
Dentaurum	Mean	201.26	24.32	21.29	2.18	--	58.39
	SD	24.82	0.74	7.28	0.32	--	9.20
Unitek	Mean	130.67	25.23	20.66	0.25	--	67.35
	SD	21.54	2.45	6.90	0.02	--	14.93
Tomy	Mean	153.92	19.65	25.28	1.13	--	48.32
	SD	9.78	1.68	3.76	0.18	--	11.86
Ormco	Mean	372.27	49.83	38.52	2.43	--	94.37
	SD	29.36	2.66	12.19	0.28	--	13.28
Control		0.28±0.15	0.24±0.13	0.73±0.65	0	--	0.32±0.13

表 **3A.** 矯正金屬線之原子吸收光譜分析

3A. pH 6.75 AS metal wire (μg/cm^2)

		Ni	Cr	Fe	Co	Ti	Cu
0.016-in NiTi	Mean	18.27	--	0	--	0	--
	SD	3.28	--	0	--	0	--
0.016 x 0.022-in NiTi	Mean	20.73	--	0	--	0	--
	SD	5.43	--	0	--	0	--
0.014-in SSW	Mean	21.38	12.63	2.73	--	--	0.92
	SD	5.32	5.27	0.26	--	--	0.01
0.016 x 0.022-in SSW	Mean	26.21	11.27	3.29	--	--	0.89
	SD	5.43	4.47	0.32	--	--	0.01
0.010 in SSW		38.24	15.21	3.10	--	--	0.97
		8.35	5.48	0.29	--	--	0.01
Control		0.28±0.15	0.24±0.13	0.73±0.65	--	--	--

表 **3B.** 矯正金屬線之原子吸收光譜分析

2B. pH 4 AS metal wire

		Ni	Cr	Fe	Co	Ti	Cu
0.016-in NiTi	Mean	58.21	--	0	--	21.42	0
	SD	4.37	--	0	--	8.32	0
0.016 x 0.022-in NiTi	Mean	69.27	--	0	--	26.73	0
	SD	5.49	--	0	--	9.28	0
0.014-in SSW	Mean	45.32	17.28	3.74	--	0	2.10
	SD	6.43	9.20	0.32	--	0	0.53
0.016 x 0.022-in SSW	Mean	50.48	21.20	2.82	--	0	1.18
	SD	8.39	11.27	0.29	--	0	0.47
0.010 in SSW		43.93	29.51	4.21	--	0	1.92
		5.68	10.63	0.10	--	0	0.55
Control		0.28±0.15	0.24±0.13	0.73±0.65	--	--	--

表 **2C.** 矯正金屬線之原子吸收光譜分析

2C. pH 3.5 APF metal wire

			Ni	Cr	Fe	Co	Ti	Cu
0.016-in NiTi	Mean		63.28	--	--	--	53.39	--
	SD		7.39	--	--	--	4.56	--
0.016 x 0.022-in NiTi	Mean		73.90	--	--	--	67.83	--
	SD		8.37	--	--	--	8.47	--
0.014-in SSW	Mean		68.38	43.39	21.38	--	--	2.12
	SD		8.87	6.30	5.32	--	--	0.39
0.016 x 0.022-in SSW	Mean		74.39	37.71	32.19	--	--	1.95
	SD		9.93	4.38	8.34	--	--	0.27
0.010 in SSW			74.54	49.37	25.31	--	--	2.38
			8.38	6.49	6.38	--	--	0.41
Control			0.28±0.15	0.24±0.13	0.73±0.65	--	--	--

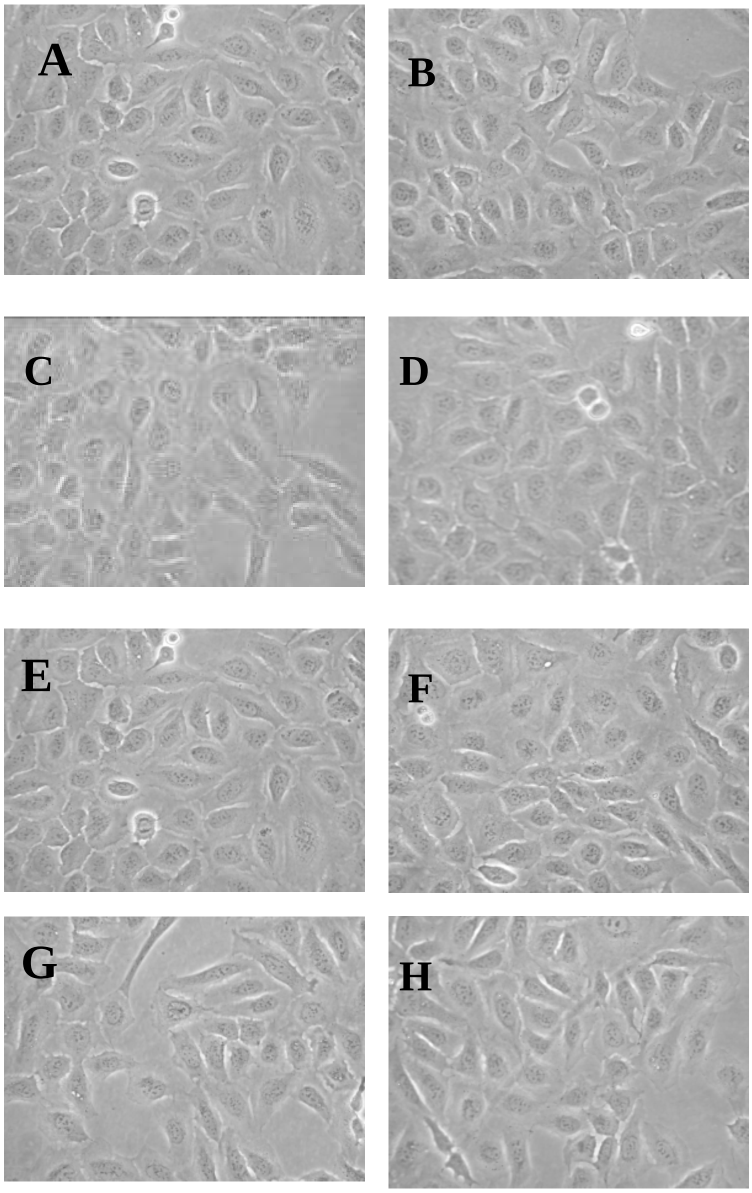


Figure 5. MTT assay photographs under examining light microscope (200x magnified) showing U2OS cells treated with various concentrations of immersed metal brackets solutions. A. the Dentaurum group, pH4, 1 ul/ml. B. the Dentaurum group, pH7, 1 ul/ml. C. the Unitek group, pH4, 1 ul/ml. D. the Unitek group, pH7, 1 ul/ml. E. the Tomy group, pH4, 1 ul/ml. F. the Tomy group, pH7, 1 ul/ml. G. the Ormco group, pH4, 1 ul/ml. H. the Ormco group, pH7, 1 ul/ml. Cell membranes appeared to be intact and no obvious cell damage or apoptotic body appeared.

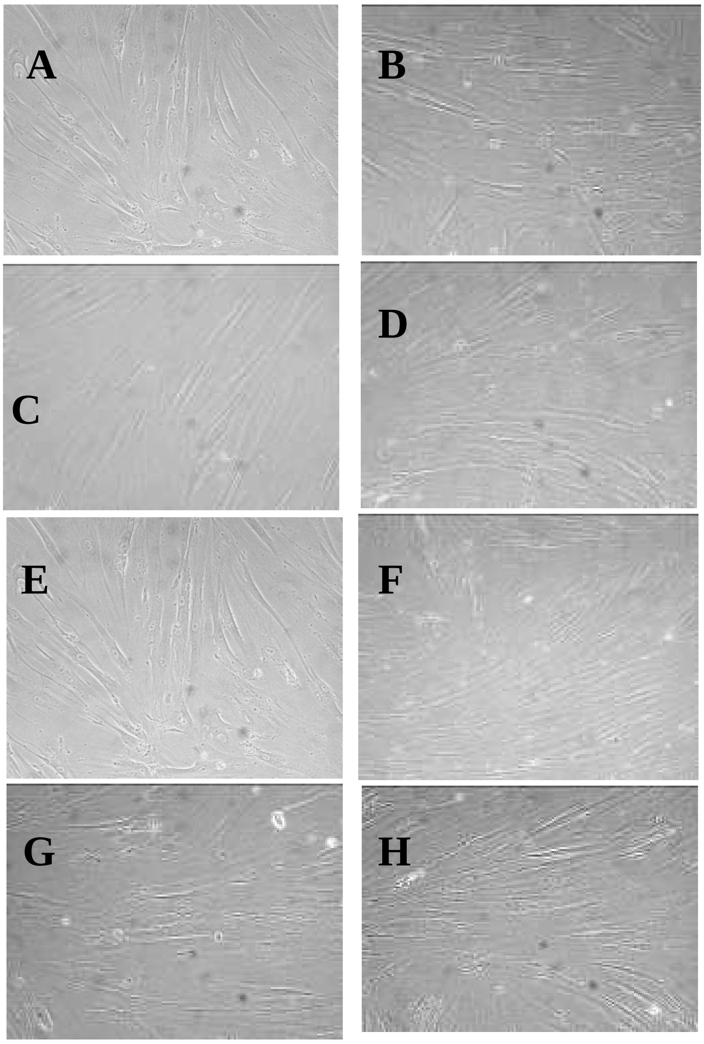


Figure 6. MTT assay photographs under examining light microscope (200x magnified) showing HGF treated with various concentrations of immersed metal brackets solutions. A. the Dentaurum group, pH4, 1 ul/ml. B. the Dentaurum group, pH7, 1 ul/ml. C. the Unitek group, pH4, 1 ul/ml. D. the Unitek group, pH7, 1 ul/ml. E. the Tomy group, pH4, 1 ul/ml. F. the Tomy group, pH7, 1 ul/ml. G. the Ormco group, pH4, 1 ul/ml. H. the Ormco group, pH7, 1 ul/ml. Cell membranes appeared to be intact and no obvious cell damage or apoptotic body appeared.

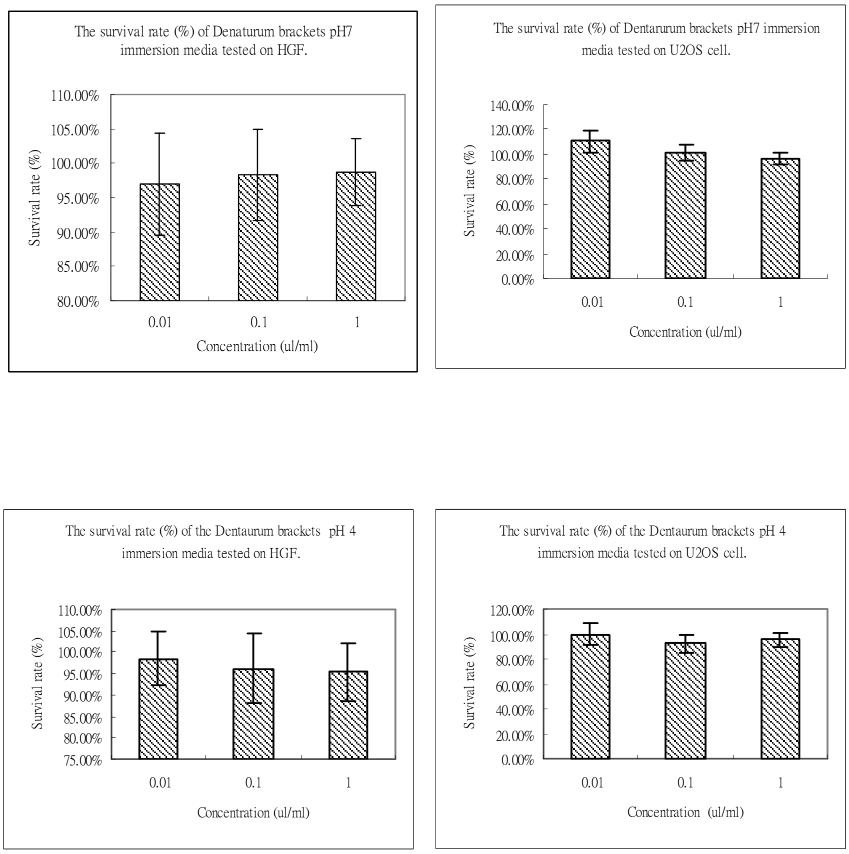


Figure 7. The survival rate of the Dentaurum metal-bracket immersion media treated on U2OS and HGF cells. Survival rate (%)= (absorbance of experiment / absorbance of control) X 100%

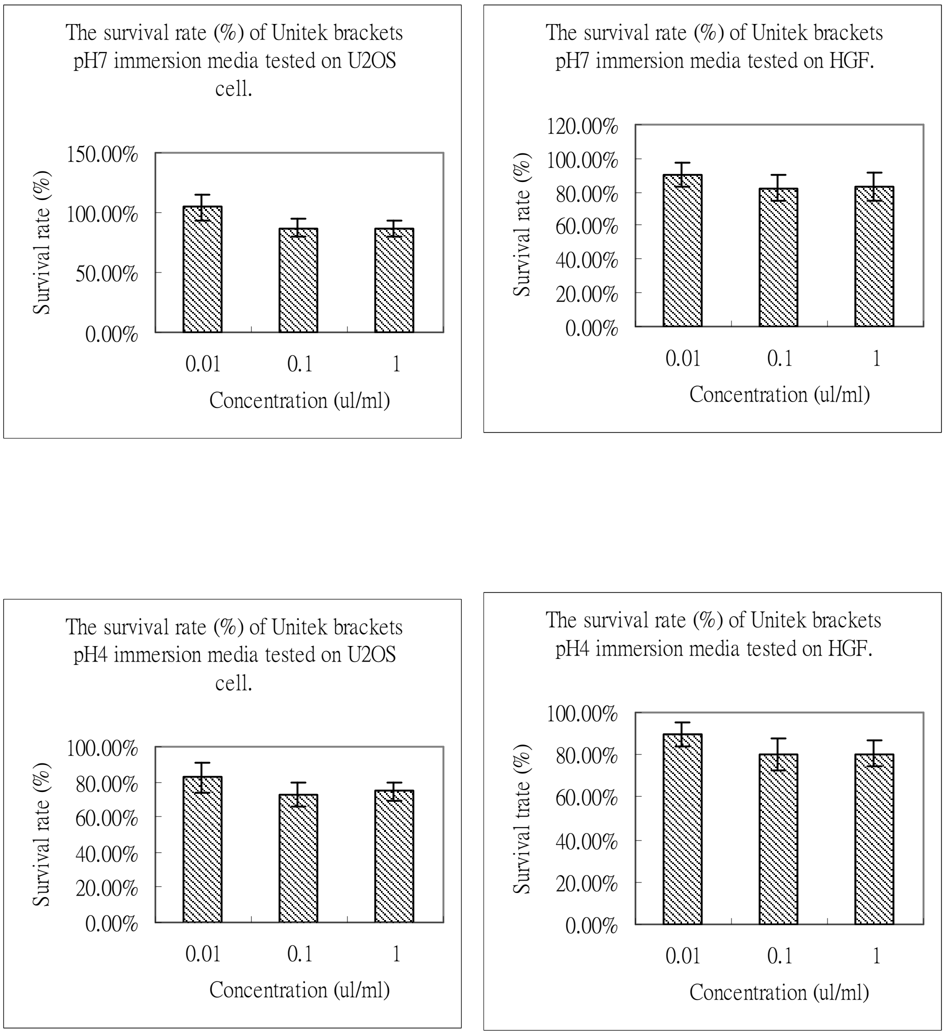


Figure 8. The survival rate of the Unitek metal-bracket immersion media treated on U2OS and HGF cells. Survival rate (%)= (absorbance of experiment / absorbance of control) X 100%.

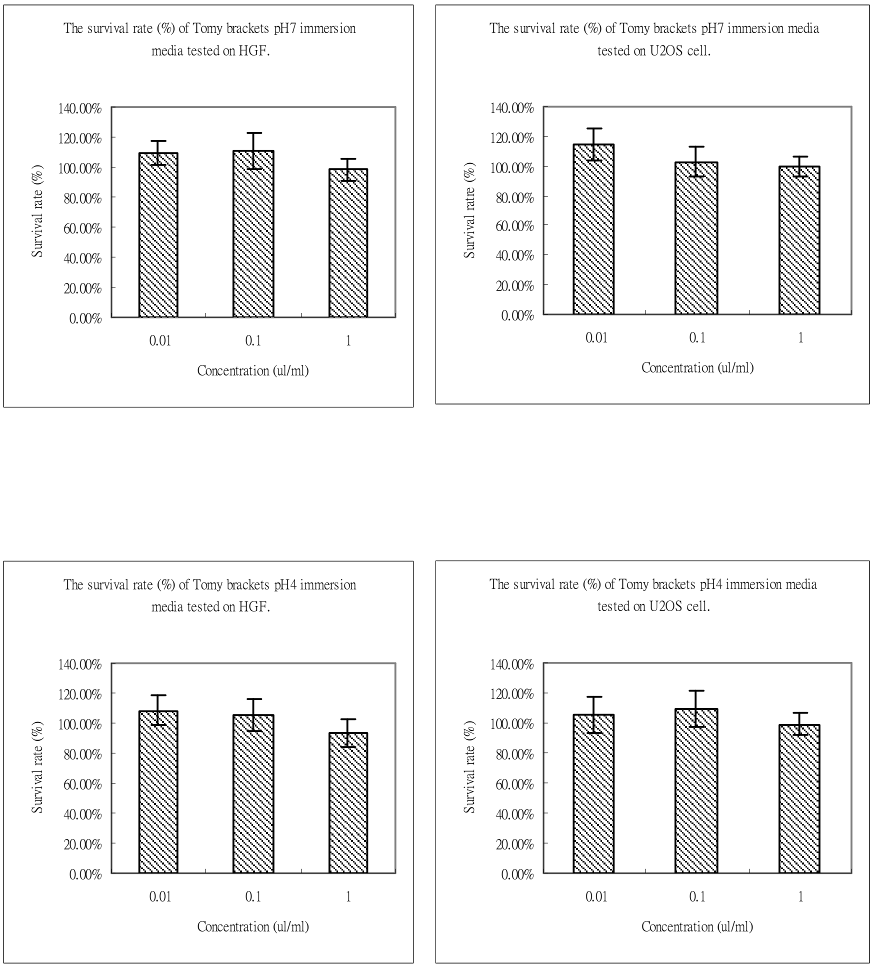


Figure 9. The survival rate of the Tomy metal-bracket immersion media treated on U2OS and HGF cells. Survival rate (%)= (absorbance of experiment / absorbance of control) X 100%.

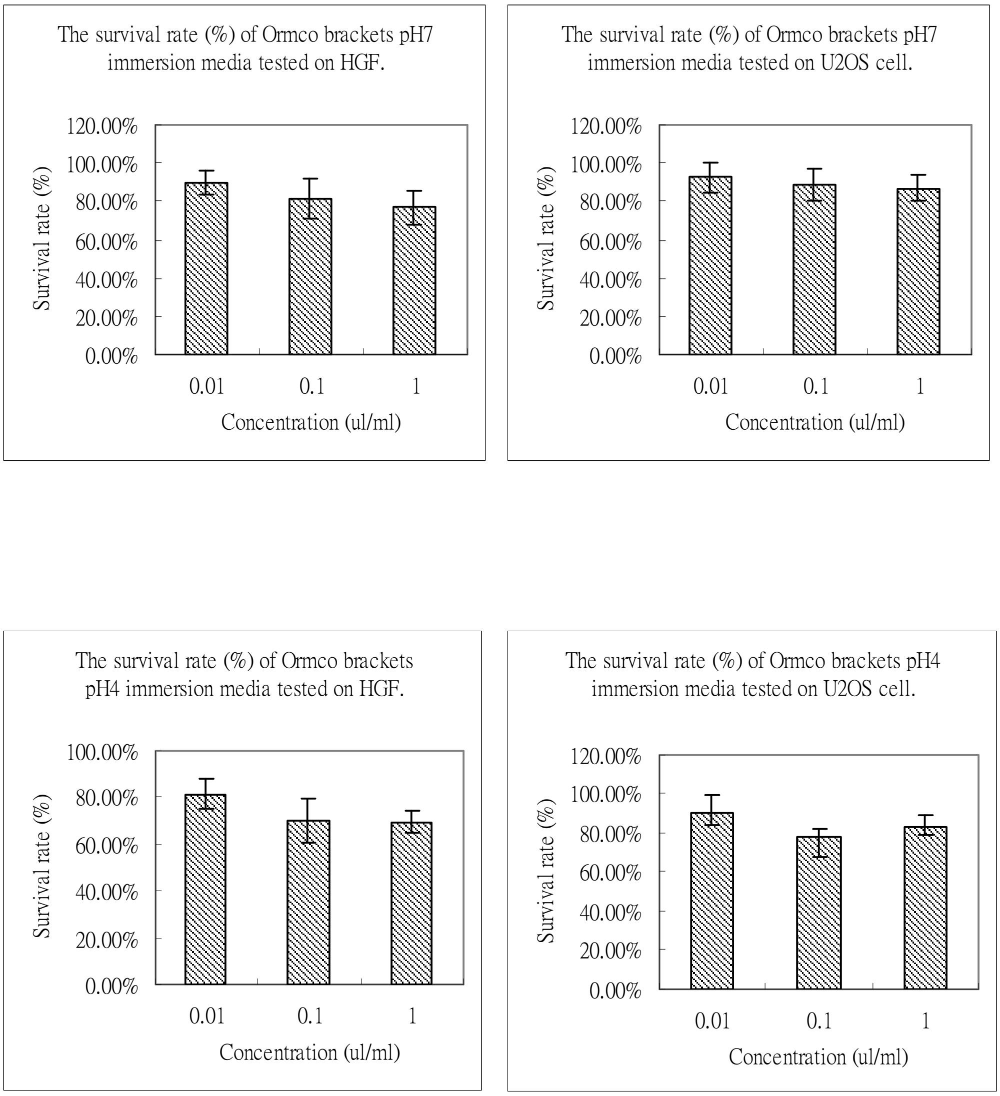


Figure 10. The survival rate of the Ormco metal-bracket immersion media treated on U2OS and HGF cells. Survival rate (%)= (absorbance of experiment / absorbance of control) X 100%.

(六)計畫成果自評部份，

請就研究內容與原計畫相符程度：內容與計劃大致上相符合

達成預期目標情況：有達到第一與第二年年計畫的 研究目的

研究成果之學術或應用價值：已投稿期刊接受刊登

European Journal of Orthodontics. 2007 29: 198 – 203

Angle Orthodontics.2007, 77(2):349-354

綜合評估:

1. 口腔健康含氟預防用品對於矯正患者之裝置會有腐蝕現象出現,使用時應審慎。
2. 腐蝕之釋出物就目前研究結果發現會對細胞具有毒性,目前尚無證據顯示會造成人體傷害，值得進一步研究。
3. 如何再改質矯正支架，以減少不必要之物質釋出，仍為可進衣不發展之方向。

出席國際學術會議心得報告

計畫編號	95-2314-B-040-004-
計畫名稱	口腔中氟離子對金屬矯正裝置之機械與生物學效應研究(2/2)
出國人員姓名 服務機關及職稱	高嘉澤、中山醫學大學口腔材料科學研究所教授
會議時間地點	美國 NewOrleans
會議名稱	第八十五屆國際牙醫學會學術研究大會(85th General session and Exhibition of the International Association of Dental Research)
發表論文題目	**The study of friction force on orthodontic appliance**

一、參加會議經過

本屆國際牙醫學會學術研究大會於三月二十一日至三月二十四日於美國路易斯安那州的紐奧爾良(NewOrleans)舉辦，隨者學校一起參加人員，經過多次轉機終於到達此二年前才經過災患摧殘的都市，外觀感覺似乎已重建恢復。此屆大會參與人數也許因為上述原因人數較少，海報展是約有 3000 篇，其中不乏撤回者。口頭報告與專題演講的主題仍是一樣，聆聽人數較往年減少。另外單獨特別的節目也有人參加。個人挑選有興趣之主題去聆聽，並與發表者討論。晚上結束時參加姐妹校(UAB)之招待茶會，認識一些新朋友。

二、與會心得

每年的 IADR 大會是世界上許多國家的口腔醫學方面研究者都來聚會的會議，主辦單位應已至少八十四次辦理之經驗，大會之秩序與流程順暢，各項工作者僅然有序，讓參加者感受到尊重與舒適，此會議之辦理直得學習，以作為日後舉辦國際會議時之重要參考。

大會中最讓人感覺得有趣的還是於海報論文方面展示，可以詳細的與報告者討論，分享別人的經驗，甚至交個朋友，作為日後交流討論之對象。而口頭報告則因時間較短，報告完的醫師，隨即消失，不易有太多的收穫。另外會場中之廠商展示，可以看到新的材料和儀器設備，也可知道當今之發展潮流。隨行中，有研究生之參與，也趁機給與教導，讓後輩看到世界的脈動，刺激他們的興趣。

此種大會是值得鼓勵有志於於研究的人員參加，除了學術上經驗之交流外，人際之交流也是參加會議之最大收穫。

發表論文部份

Abstract

Purpose: This study investigated and compared the levels of friction resistance between metal brackets and orthodontic wires after immersion in an acidified phosphate fluoride (APF) agent. **Materials:** Three types of lower incisor stainless-steel metal brackets with β-titanium alloy wire (TMA), heated-activated NiTi wire (NiTi), and 2 sizes of stainless steel wires (SSWs) were immersed in 0.2% APF and pH 6.75 artificial saliva solutions for 24 hours. The total specimen population was composed of 480 brackets/wire specimens. The frictional resistance was measured on an EZ-test machine (Shimadazu, Tokyo, Japan) with a 5 N load cell. An elastic modulus ligated to the bracket was attached to the crosshead of the machine and pulled at a speed of 10 mm/min for a distance of 5 mm. A completely randomized design (one-way) ANOVA was used to test for significant differences among the 3 bracket/wire specimens after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions. This was followed by the Student-Newman-Keuls multiple comparison of means ranking at $P < 0.05$ to determine differences between the different groups. **Results:** In the APF-immersed group, the static frictional force was higher than the kinetic frictional force. The orthodontic wires had frictional forces that statistically significantly differed ($P < 0.05$) in this progressive order: TMA, NiTi, and SSW. Similar frictional force results were obtained in the pH 6.75 saliva group ($P < 0.05$). The frictional force values of the APF group were higher than those of the pH 6.75 saliva group ($P < 0.05$). **Conclusions:** This study demonstrates that the frictional force of orthodontic brackets and wires are influenced by contact with

fluoride-containing solutions.

Tooth movement associated with sliding mechanics has been noted as a series of steps involving tooth tipping and uprighting, rather than a continuous, gliding process.[1-3] In a preadjusted edgewise system, smoothly sliding mechanics can increase the treatment efficiency. The resistance to sliding in the preadjusted edgewise system is a combination of classical friction, archwire-bracket binding, and archwire notching.[4] It is important that frictional forces be eliminated or minimized when orthodontic tooth movement is being planned. As is well known, friction is defined as a force that delays or resists the relative motion of 2 materials in contact, and its direction is tangential to the common interface of the 2 surfaces.[5-7] There are 2 types of friction: kinetic (dynamic), which occurs during motion, and static, which resist motion.[8-10]

Frictional forces develop when sliding mechanics are employed. Friction is a function of the dynamic relationship between archwires, brackets, and the ligation type in the oral environment. During sliding mechanics, the biologic tissues respond, and tooth movement occurs only when the optimal forces applied exceed the friction on the bracket wire interface. High levels of frictional forces can result in the loss of the bracket bonding, associated with either small dental movement or no movement at all. When friction prevents the tooth to which the bracket is attached form moving, friction can reduce the available force by almost 40%, resulting in anchorage loss.[6,11,12] Many studies have been conducted on orthodontic archwires and brackets, and the following factors influencing friction have been identified: ligation type, the force applied, bracket-wire clearance, wire size and morphology, bracket dimensions, torque at the bracket-wire interface, types of motion at the bracket-wire interface, and the type of bracket and wire materials used.[1-5,10,13] But among these factors which influence friction, some of them are conflicting. Such discrepancies may be due to differences in the test set-up or in the surface conditions of the samples tested (such as being oxidized, degreased, oiled, dry, wet, rough, smooth, etc.). There is one factor that influences friction which has seldom been discussed: the application of fluoride on orthodontic appliances in the oral environment.

The fluoride level in the oral cavity varies according to the prophylactic treatment. Fluoride is used at concentrations of up to 1% in toothpastes and mouthwashes and at close to 2% when the aim is to eliminate enamel stains,[14] and these substances have a pH range of between about 3.5 and 7.0.[15] It is known that dental hygiene products containing fluoride ions can attack the oxide film formed on titanium surfaces, and this suggests problems regarding the dental use of Ti.[16] In an acidic medium, a low quantity of fluoride induces the formation of hydrofluoric acid (HF) according to the following reaction:[17,18]

$NaF + H^+ = HF + Na^+$. (1)

Hydrofluoric acid is known to dissolve the surface oxide layer by the following reactions:

$Ti_2O_3 + 6HF = 2TiF_3 + 3H_2O$, (2)

$TiO_2 + 4HF = TiF_4 + 2H_2O$, or (3)

$TiO_2 + 2HF = TiOF_2 + H_2O$. (4)

Among these titanium fluorides or titanium oxyfluoride, titanium (IV) fluoride (TiF_4) is a soluble compound.[19] Due to the formation of higher concentrations of HF, the corrosion of Ti is enhanced in an acidic environment. Therefore, the frictional effect of orthodontic appliances after fluoride corrosion is important in orthodontic treatment.

The purpose of the present study was to investigate the friction resistance changes of metal brackets and wires after these materials were immersed in an acidulated phosphate fluoride solution and in a pH 6.4 artificial saliva solution.

MATERIAL AND METHODS

Sample population

In total, 480 bracket-wire samples were studied. Each test condition contained 10 bracket-wire samples. Three types of lower incisor stainless steel metal brackets were used: Unitek DynaLock standard edgewise brackets (Unitek, 3M, Monrovia, CA, USA), Tomy standard edgewise brackets (Tomy, Tokyo, Japan), and Ormco standard edgewise brackets (TMA, SDS Ormco, Orange, CA, USA). Two orthodontic stainless steel wires (SSWs) were used: 0.018 in (0.46 mm) and 0.019 x 0.025 in(0.48 x 0.64 mm) (3M, Unitek). Two titanium-based orthodontic wires were used: 0.019 x 0.025 in heat-activated Ni-Ti (NiTi, 3M, Unitek) and 0.017 x 0.025 in(0.44 x 0.64 mm) β-titanium alloy wire (TMA, Ormco). A 0.2% acidulated phosphate fluoride (APF; 0.2 mass % NaF + 0.17 mass % H_3PO_4, pH 3.5) solution and pH 6.75-adjusted artificial saliva solution (Table I) were used in the study. The ligation between the bracket and wire used a clear modulus (Quik-Stik Clear, A-1 Alastik, 3M Unitek).

Method of testing

Brackets and archwires were cleaned with an alcohol wipe before the modules were tied with mosquito forceps, 25 mm from the lower end of the archwire, to form a test unit. All units in the experimental groups were soaked in 0.2% acidulated phosphate fluoride for 24 hours before testing. The control groups were immersed in a pH 6.4 artificial saliva solution for 24 hours before testing.

The test procedure was modified from a previous design.[20] Testing was performed on an EZ-test machine (Shimadazu, Tokyo, Japan), with a crosshead speed of 10 mm/min over a 5-mm stretch of archwire (Fig. 1). A plumb line was hung to ensure that the bracket mount was parallel with the vertical line scribed on the steel bar base of the bracket mount assembly.

The 5 N load cell was calibrated to between 0 and 5 N, and the archwire was drawn through the bracket as the crosshead moved inferiorly at a rate of 10 mm/min (Fig. 2). This crosshead speed was selected as a previous study found no significant difference when using crosshead speeds of 0.5 to 50 mm/min.[21]

Care was taken to align the archwire so that the sample was parallel with the vertical framework of the machine. The bracket was pulled in a vertical direction by a loop of 0.018-in stainless-steel wire, and the force required to initiate and maintain movement of the bracket over the 5-mm test distance was measured. The program was set to highlight the maximum frictional force at initial movement, which was taken to represent the peak static frictional resistance. For each bracket-wire combination, a new wire and bracket were used.

Data acquisition and Statistical analysis

The load cell registered the force levels needed to move the wire through the bracket, and these values were stored on a computer hard disk. The data were then analyzed using a statistical package (Primer, McGraw Hill, New York, NY, USA).

A completely randomized design (one-way) ANOVA was used to test for significant differences among the 3 bracket/wire types. This was followed by the

Student-Newman-Keuls multiple comparison of means procedure at $P < 0.05$ to determine differences between the different groups. Unpaired *t*-test was used to detect the frictional difference between the APF and pH 6.75 groups with $P < 0.05$ accepted as showing a significant statistical difference.

RESULTS

The frictional force values (mean ± SD of static and kinetic friction) of the stainless-steel, nickel-titanium, and TMA wires with different types of brackets were compared after immersion in the 0.2% APF solution. The static and kinetic forces in all test groups showed statistical differences ($P < 0.05$). (Tab. II) Table II shows that the static force of TMA was the highest, while the 0.018-in round stainless steel wire was the lowest. The same result was found for the kinetic force measurements.

The frictional force values of the stainless-steel, nickel-titanium, and TMA wires with different types of brackets were compared after immersion in the pH 6.75 artificial saliva solution. The static and kinetic forces in all test groups showed statistical differences ($P < 0.05$) (Tab. III). Table III shows that, except for the Tomy group, the static force of TMA was the highest, while the 0.018-in round stainless steel was the lowest. The same result was found for the kinetic force measurements.

The frictional force values of the stainless-steel, nickel-titanium, and TMA wires with Ormco brackets were compared after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions (Tab. IV). The results showed that in the 0.2% APF solution, the static force was higher than the kinetic force ($P < 0.05$). Comparison of the static forces between the 0.2% APF and pH 6.75 saliva solutions showed no statistical difference between the 0.019 x 0.025-in SSW ($P = 0.0853 > 0.05$) and the 0.019 x 0.025-in NiTi ($P = 0.057 > 0.05$). The kinetic force comparisons among the 0.2%

APF, pH 6.75 saliva, TMA , NiTi, and SSW groups all showed statistical significant differences ($P < 0.05$). The kinetic force of the APF group was higher than that of the pH 6.4 group.

The frictional force values of the stainless-steel, nickel-titanium, and beta-titanium wires with Tomy brackets were compared after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions (Tab. V). The result showed that in the 0.2% APF solution, the static force was higher than the kinetic force in stainless-steel wire group (0.018-in SSW and 0.019 x 0.025 in SSW) ($P < 0.05$), but no difference was observed between the TMA and NiTi wire groups ($P > 0.05$). The static forces of TMA, NiTi, and SSW in the 0.2% APF and pH 6.75 saliva groups showed statistical differences ($P < 0.05$). The 0.2% APF group showed a higher static force than the pH 6.75 saliva group for TMA wire, and showed lower static forces for the NiTi and SSW wires. The kinetic forces of TMA, NiTi, and SSW with the 0.2% APF and PH 6.75 saliva solutions showed statistical differences ($P < 0.05$). The 0.2% APF group showed a higher kinetic force than the pH 6.75 saliva group with the TMA wire, but had lower static forces with the NiTi and SSW wires.

The frictional force values of the stainless-steel, nickel-titanium, and beta-titanium wires with Unitek brackets were compared after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions (Tab. VI). In the 0.2% APF solution, the static force was higher than the kinetic force with the TMA, NiTi, and SSW wires ($P < 0.05$). With the static force comparisons in the 0.2% APF and pH 6.75 saliva solutions, except for the TMA group, the static force of the NiTi and SSW groups showed statistical differences ($P < 0.05$). For the kinetic force comparisons in the 0.2% APF and pH 6.75 saliva solutions, only the 0.019 x 0.025-in NiTi group showed a statistical difference ($P = 0.000 < 0.05$).

DISCUSSION

There is little information available regarding the frictional effects of fluoride-containing prophylactic agents on actual titanium-based and stainless-steel orthodontic wires with metal brackets. The present study is the first to use an acidified fluoride solution to treat wire and bracket combinations to evaluate the frictional effects. Previous studies evaluating friction only analyzed materials in a dry or wet state.[1-5] However, orthodontists and patients mostly worry about oral hygiene care. This is obviously an important thing for orthodontic patients. For daily oral hygiene protection, fluoride is commonly used, in such products as toothpaste, mouthwashes, etc. Appliances often contact fluoride products. The effects of fluoride on orthodontic appliances need to be evaluated. In the present study, wire and bracket combinations were immersed in a fluoride solution. This was used to simulate the oral environment. Although the frictional test system in the present study used a dry state, it was assumed that the friction values might be larger than that in a wet state.

Reley et al. studied friction with respect to diverse material compositions of ligation auxilaries.[22] They found that steel ligatures generated greater frictional forces than plastic modules and that moistening caused an insignificant increase in friction in the case of steel ligatures and was irrelevant to the plastic modules.[22] To avoid having too many side effects that might influence the frictional forces of the wire and bracket, an elastic modules was used as the ligation method in the present study.

When examining diverse brackets and archwire materials on a simulated canine-retraction assembly, Garner et al. indicated that friction increased in the order of stainless steel, nitinol, and beta titanium (TMA).[23] In present study, a similar result was found in that the TMA group's frictional force was higher than that of the NiTi or SSW group. The static frictional force of TMA was higher in the 0.2% APF than that in the pH 6.75 saliva solution in the present study (Tabs. II and III). Al-Mayouf et al.[24] indicated that corrosion of Ti and its alloys are enhanced in an acidic environment, because F^- ions in the solution combine with H^+ ions to form HF, even at low fluoride concentrations. The fluoride ions in the prophylactic agents have been reported to cause corrosion and discoloration of titanium and its alloys.[25] Thus the increased frictional force of TMA in the 0.2% APF then in pH 6.75 solution may be a reflection of the severity of the corrosion due to the acidified fluoride. The NiTi wire group showed similar frictional force results as for the TMA wire in the Ormco and Unitek groups (Tabs. IV and VI). But the results differed in the Tomy group (Tab. V), for which the frictional values, both static and kinetic, of the APF group were lower than those of the pH 6.75 saliva group. Watanabe et al. reported that the metal content of the alloy influences the surface roughness.[26] This might have been the reason why the frictional values of the Tomy group differed from those of the Ormco and Tomy groups.

In orthodontic teeth movement, titanium alloy wires provide a light and continuous force with large amounts of activation for long periods. They are very useful as initial or intermediate wires between the first alignment and finishing stages of treatment. In these stages, friction is regarded as an influential factor on tooth alignment. Recently, passive self-ligating brackets combined with small copper NiTi wire were found to enhance the low friction and to hasten tooth alignment.[27] From this point of view, if a Ti-containing wire is corroded by fluoride products, then there will be an increase in the frictional resistance, regardless of what kind of bracket is used.

Clinically, when the sliding mechanics are applied to teeth movement, the friction becomes an important factor in the sliding of the teeth, as it influences the treatment results and efficiency. With regard to the archwires, TMA wires generally resulted in less-efficient sliding mechanics than SSW, and the frictional resistance generally increased with an increase in archwires size, with rectangular wires generally producing more friction than round wires.[23,28-32] From Tables I and II, data demonstrated that the TMA friction force was higher than that of the SSW, and as the SSW size increased from 0.018 to 0.019 x 0.025 in, the friction forces also increased.

The SSW frictional force in the acidified APF groups statistically differed from that of the pH 6.75 groups (Tabs. IV-VI). Stainless steel is easily corroded in chloride-containing solutions and an acidic environment.[33-35] The sliding mechanism usually uses a stainless steel wire as the working wire, in order to reduce the frictional resistance. In the present study, the SSW might have been corroded by the acid. Therefore, the results showed that SSW group’s frictional resistance in the acidified APF condition was higher than that in the pH 6.75 condition. From this point of view, the corrosion of the wire or bracket influenced the frictional force change. One needs to be cautious as to the acid and fluoride states in the mouth.

CONCLUSIONS

The ideal orthodontic treatment is for the teeth to slide or align smoothly when wired into the bracket. From the present study, it is clear that fluoride-containing prophylactic material used in the orthodontic treatment process can influence the frictional forces. The frictional resistance of the wires and brackets increased in the acidic 0.2% APF solution. The result of increased frictional levels might be an increased treatment period and loss of anchorage control. Methods for carefully applying fluoride in orthodontic patients should be evaluated.

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Table I. Content of the artificial saliva

Company	Sinphar Pharm, Taipei, Taiwan	
Content	Sali Lube (Saliva substitute)	
	Sodium Chloride	0.844 mg
	Potassium Chloride	1.2 mg
	Calcium Chloride Anhydrous	0.146 mg
	Magnesium Chloride 6 H2O	0.052 mg
	Potassium Phosphate dibasic	0.34 mg
	Sorbitol Solution 70%	60 mg
	Methyl Paraben	2 mg
	Hydroxyethyl Cellulose	3.5 mg

Table II. Comparison of frictional force values (mean±SD of static and kinetic forces) of the stainless steel (SSW), nickel-titanium NiTi), and beta-titanium (TMA) wires with different types of brackets after immersion in a 0.2% APF solution. One-way ANOVA was used to test for significant differences among the three bracket/wire types. The Student-Newman-Keul (SNK) multiple comparison of means procedure at *P* < 0.05 was used to show differences. SNK ranking with the same letters do not significantly differ at *P* = 0.05.

	Static force							*Kinetic force*					
	Ormco		*Tomy*			*Unitek*		*Ormco*		*Tomy*		*Unitek*	
	Mean ± SD	*SNK*	*Mean ± SD*	*SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*
0.017 x 0.025 TMA	3.10±0.25		2.83±0.35	0.35		2.84±0.21	A	2.80±0.20		2.63±0.39		2.610±0.22	
0.018 SSW	1.27±0.05	A	0.93±0.05	0.05		1.15±0.04		1.15±0.06	A	0.78±0.06		0.99±0.04	
0.019 x 0.025 NiTi	2.60±0.13		2.13±0.16	0.16		2.74±0.24		2.33±0.12		1.89±0.16		2.39±0.20	
0.019 x 0.025 SSW	1.40±0.05	A	1.34±0.06	0.06		1.39±0.06	A	1.26±0.07	A	1.20±0.08		1.25±0.07	
	F=380.51	**P=0**	**F=180.4**		**P=0**	**F=291.04**	**P=0**	**F=428.62**	**P=0**	**F=137.97**	**P=0**	**F=285.48**	**P=0**

n=10, movement speed 10mm/min

Table III. Comparison of frictional force values of the stainless steel (SSW), nickel-titanium (NiTi), and beta-titanium (TMA) wires with different types of brackets after immersion in a pH 6.75 artificial saliva solution. One-way ANOVA was used to test for significant differences among the three bracket/wire types. The Student-Newman-Keul (SNK) multiple comparison of means procedure at *P* < 0.05 was used to show differences. The SNK ranking with the same letters do not significantly differ at *P* = 0.05.

	Static force (N/mm²)						*Kinetic force (N/mm²)*					
	Ormco		Tomy		Unitek		Ormco		Tomy		Unitek	
	Mean ± SD	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*	*Mean ± SD*	*SNK*
0.017 x 0.025 TMA	2.83±0.12		2.31±0.12		2.83±0.23		2.59±0.15		2.10±0.16		2.56±0.24	
0.018 SSW	1.06±0.08		1.07±0.10		1.10±0.03		0.92±0.08		0.85±0.07		0.99±0.07	
0.019 x 0.025 NiTi	2.43±0.24		2.42±0.10		2.27±0.16		2.09±0.03		2.10±0.15		1.97±0.09	
0.019 x 0.025 SSW	1.39±0.16		1.63±0.17		1.57±0.19		1.19±0.06		1.43±0.12		1.35±0.15	
	F=264.20	**P=0**	**F=249.54**	**P=0**	**F=204.47**	**P=0**	**F=741.75**	**P=0**	**F=223.16**	**P=0**	**F=205.45**	**P=0**

Table IV. Comparison of frictional force values of the stainless steel (SSW), nickel-titanium (NiTi), and beta-titanium (TMA) wires with Ormco brackets after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions. Unpaired *t*-test was used to detect the frictional differences between the APF and pH 6.4 solutions, with *P* < 0.05 indicating a significant statistical difference.

	APF		APF			
	Static force	*(N/mm^2)*	*Kinetic force*	*(N/mm^2)*		
	Mean	SD	Mean	SD	t value	P value
0.017 x 0.025 TMA	3.10	0.25	2.80	0.20	**3.035**	**0.007**
0.018 SSW	1.27	0.05	1.15	0.06	**1.971**	**0.064**
0.019 x 0.025 NiTi	2.60	0.13	2.33	0.12	**4.899**	**0.000**
0.019 x 0.025 SSW	1.40	0.05	1.26	0.07	**5.640**	**0.000**
	APF		pH 6.75			
	Static force	*(N/mm^2)*	*Statistic force*	*(N/mm^2)*		
	Mean	SD	Mean	SD	t value	P value
0.017 x 0.025 TMA	3.10	0.25	2.31	0.12	**3.135**	**0.006**
0.018 SSW	1.27	0.05	1.07	0.10	**7.445**	**0.000**
0.019 x 0.025 NiTi	2.60	0.13	2.42	0.10	**2.037**	**0.057**
0.019 x 0.025 SSW	1.40	0.05	1.63	0.17	**0.188**	**0.853**
	APF		pH 6.75			
	Kinetic force	*(N/mm^2)*	*Kinetic force*	*(N/mm^2)*		
	Mean	SD	Mean	SD	t value	P value
0.017 x 0.025 TMA	2.80	0.20	2.10	0.16	**2.605**	**0.018**
0.018 SSW	1.15	0.06	0.85	0.07	**7.641**	**0.000**
0.019 x 0.025 NiTi	2.33	0.12	2.10	0.15	**6.060**	**0.000**
0.019 x 0.025 SSW	1.26	0.07	1.43	0.12	**2.271**	**0.036**

Table V. Comparison of the frictional force values of the stainless steel (SSW), nickel-titanium (NiTi), and beta-titanium (TMA) wires with Tomy brackets after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions. Unpaired *t*-test was used to detect the frictional differences between the APF and pH 6.4 solutions, with *P* < 0.05 indicating a significant statistical difference.

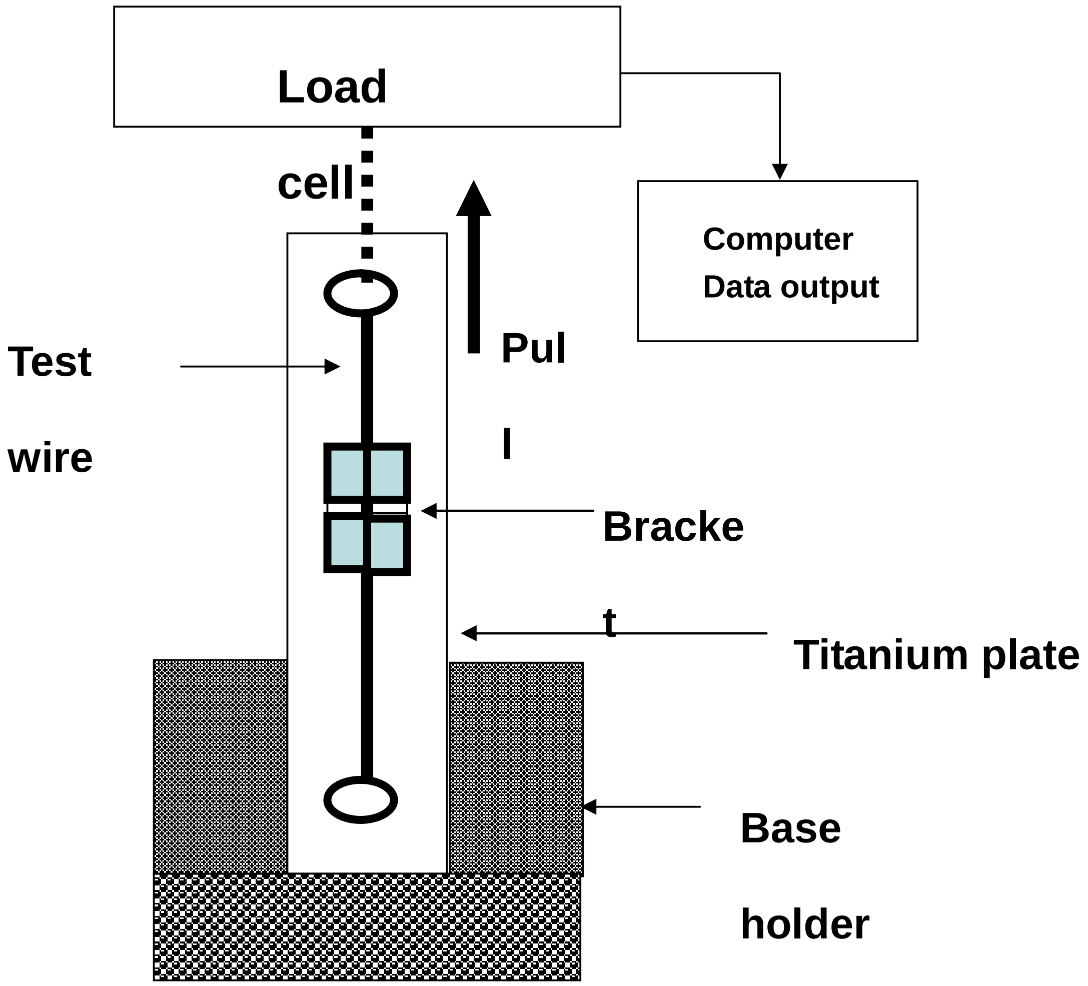
	APF		APF			
	Static force	*(N/mm²)*	*Kinetic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.83	0.35	2.63	0.39	**1.176**	**0.255**
0.018 SSW	0.93	0.05	0.78	0.06	**6.049**	**0.000**
0.019 x 0.025 NiTi	2.13	0.16	1.89	0.16	**3.290**	**0.104**
0.019 x 0.025 SSW	1.34	0.06	1.20	0.08	**4.285**	**0.000**
	APF		pH 6.75			
	Static force	*(N/mm²)*	*Statistic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.83	0.35	2.31	0.117	**4.513**	**0**
0.018 SSW	0.93	0.05	1.065	0.104	**-3.647**	**0.002**
0.019 x 0.025 NiTi	2.13	0.16	2.4175	0.1014	**-4.778**	**0**
0.019 x 0.025 SSW	1.34	0.06	1.6325	0.1708	**-5.182**	**0**
	APF		pH 6.75			
	Kinetic force	*(N/mm²)*	*Kinetic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.63	0.39	2.103	0.1622	**3.933**	**0**
0.018 SSW	0.78	0.06	0.8475	0.06609	**-2.385**	**0.028**
0.019 x 0.025 NiTi	1.89	0.16	2.098	0.146	**-2.982**	**0.008**
0.019 x 0.025 SSW	1.20	0.08	1.433	0.1155	**-5.286**	**0**

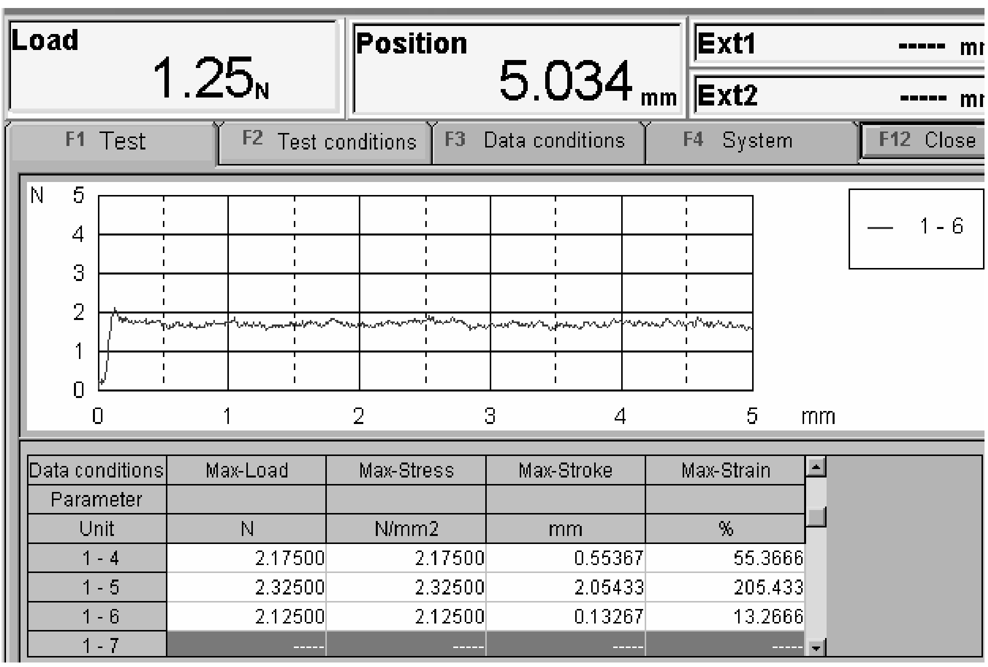
Table VI. Comparison of the frictional force values of the stainless steel (SSW), nickel-titanium (NiTi), and beta-titanium (TMA) wires with Unitek brackets after immersion in the 0.2% APF and pH 6.75 artificial saliva solutions. Unpaired *t*-test was used to detect the frictional difference between the APF and PH 6.4 solutions, with *P* < 0.05 indicating a significant statistical difference.

	APF		APF			
	Static force	*(N/mm²)*	*Kinetic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.84	0.21	2.61	0.21	**2.397**	**0.028**
0.018 SSW	1.15	0.04	0.99	0.04	**9.323**	**0.000**
0.019 x 0.025 NiTi	2.74	0.24	2.39	0.20	**3.560**	**0.002**
0.019 x 0.025 SSW	1.39	0.06	1.25	0.07	**4.742**	**0.000**
	APF		pH 6.75			
	Static force	*(N/mm²)*	*Statistic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.84	0.21	2.84	0.23	**0.000**	**1.000**
0.018 SSW	1.15	0.04	1.10	0.03	**3.903**	**0.001**
0.019 x 0.025 NiTi	2.74	0.24	2.27	0.16	**5.233**	**0.000**
0.019 x 0.025 SSW	1.39	0.06	1.57	0.19	**-2.797**	**0.012**
	APF		pH 6.75			
	Kinetic force	*(N/mm²)*	*Kinetic force*	*(N/mm²)*		
	Mean	SD	Mean	SD	t value	P value
0.017x0.025 TMA	2.61	0.21	2.56	0.24	**0.452**	**0.657**
0.018 SSW	0.99	0.04	0.99	0.07	**0.213**	**0.833**
0.019 x 0.025 NiTi	2.39	0.20	1.97	0.09	**6.044**	**0.000**
0.019 x 0.025 SSW	1.25	0.07	1.35	0.15	**-1.953**	**0.067**

Fig 1. Diagrammatic representation of the experimental setup.

Fig 2. Representative graphic curve of resistance force vs. displacement output.





Data conditions	Max-Load	Max-Stress	Max-Stroke	Max-Strain
Parameter				
Unit	N	N/mm2	mm	%
1 - 4	2.17500	2.17500	0.55367	55.3666
1 - 5	2.32500	2.32500	2.05433	205.433
1 - 6	2.12500	2.12500	0.13267	13.2666
1 - 7	-----	-----	-----	-----