

**EFFECT OF SALIVARY pH ON COATING DURABILITY OF
TWO DIFFERENT AESTHETIC ARCHWIRES COATINGS**

By

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Dissertation Submitted In Partial Fulfilment of the Requirements

For The Degree of Master of Science (Oral Science)

UNIVERSITI SAINS MALAYSIA

2018

ACKNOWLEDGEMENT

I dedicate my master thesis work to my parents for their boundless love, guidance, support, encouragement and faith on me.

To my life partner, my wife Baraah, for her endless sacrifice, support, love and encouragement during my educational journey.

To my son who will born soon to bringing the happiness to my life.

To all my family members for their care and support.

I am greatly thankful and appreciative for several people who make this project finished. Without their guidance and instruction, this project would have never been possible and it has been a pleasure working with them.

My sincere thanks to my supervisor, Dr. Anis Farhan Kamaruddin for her time and support during the journey of this thesis and advice in statistical analysis of this thesis. Under her guidance, my dreams became real.

I would also like to acknowledge Dr. Rabiatul Basria SMN Mydin for her support in the lab and academic work of this thesis.

I would also like to express my appreciation to Mr. Abdul Mutalib from Global Archaeological Research Centre for his support in surface observation using SEM for my study specimens.

Special thanks to Puan Zuraizah Zulkifli for her helping hands in dental lab work as well.

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LIST OF ABBREVIATION

AFM	Atomic force microscope/microscopy
Cr-Ni	Chromium nickel
DSLR	Digital single lens reflex
GIC	Glass ionomer cement
HaCaT	Permanent human keratinocytes
HGF	Primary human gingival fibroblasts
ICC	Intraclass correlation coefficient
NaOH	Sodium hydroxide
NiTi	Nickel titanium
pH	Potential of hydrogen
PMCR	Polyacid modified composite resin
PTFE	Polytetrafluoroethylene
SEM	Scanning electron microscope
SPSS	Statistical Package for the Social Sciences
SS	Stainless steel
TMA	Titanium molybdenum alloy
U2OS	Human osteogenic sarcoma cells
α	The probability of rejecting the null hypothesis when the null hypothesis is true.
μm	Micrometre
cm	Centimetre
ml	Millilitre
mm	Millimetre
$^{\circ}\text{C}$	Degree celsius
"	Inch
%	Percent

M

Molar

ABSTRAK

Kesan pH Air Liur Terhadap Ketahanan Salutan Dua Salutan Wayar Arkus Estetik Yang Berbeza

Wayar-wayar arkus ortodontik bersalut telah menunjukkan kehilangan salutan separa atau penuh seawal 21 hari penggunaan di dalam mulut. Interaksi di antara wayar arkus bersalut dan faktor oral seperti pH air liur dicadangkan telah menyebabkan fenomena ini berlaku. Penyelidikan ini telah menilai kesan pH terhadap ketahanan dua salutan wayar arkus yang berbeza dan memperhatikan topografi permukaan salutan-salutan. Wayar arkus nikel titanium (NiTi) berdiameter 0.018" yang telah sedia disalut dengan resin epoksi dan politetrafluoroetilina (PTFE) telah dipotong kepada segmen sepanjang 20 mm. Semua spesimen ($N = 102$) telah dibahagikan kepada enam kumpulan; empat kumpulan yang dirawat dengan dua pH yang berbeza dan dua kumpulan kawalan. pH air liur tiruan telah dilaraskan kepada 6.75 dan 3.5, masing-masing dengan menggunakan sodium hidroksida (NaOH) dan asid laktik. Setiap spesimen telah direndam di dalam tabung uji berasingan yang mengandungi 10 ml larutan terubahsuai yang ditutup dan disimpan di dalam takungan air selama 28 hari. Selepas masa berlalu, spesimen dikeluarkan dan dihadapkan kepada tekanan mekanikal menggunakan berus gigi elektronik selama 210 saat. Imej-imej diambil menggunakan kamera refleks lensa tunggal digital (DSLR) dan pengukuran kehilangan salutan dilakukan dengan menggunakan perisian Autodesk® AutoCAD®. Topografi permukaan spesimen dari setiap kumpulan dinilai menggunakan mikroskop electron pengimbas (SEM). Analisa statistik menunjukkan kehilangan salutan yang signifikan ($p < 0.001$) ditemui dalam kumpulan-kumpulan resin epoksi tanpa mengira nilai pH mereka, berbanding kumpulan-kumpulan PTFE. SEM mendedahkan terdapat ruptur, kekasaran dan kehilangan salutan di pelbagai lokasi pada permukaan salutan.

ABSTRACT

Effect of Salivary pH On Coating Durability of Two Different Aesthetic Archwires

Coatings

Coated orthodontic archwires have demonstrated partial or full coating loss as early as 21 days of intraoral use. It is suggested that an interaction between the coated archwires and the oral factors such as salivary pH has caused this phenomenon to occur. This study evaluated the effect of pH on the durability of two different aesthetic archwire coatings and observed the surface topography of the coatings. Nickel titanium (NiTi) archwires of 0.018" in diameter, readily coated with epoxy resin and polytetrafluoroethylene (PTFE) were sectioned into 20 mm segments. All specimens (N = 102) were divided into six groups; four groups treated with two different pH and two control groups. The pH of artificial saliva was adjusted to 6.75 and 3.5 using sodium hydroxide (NaOH) and lactic acid, respectively. Each specimen was immersed in individual test tubes containing 10 ml of modified solution which were sealed and stored in a 37°C water bath for 28 days. After the time elapsed, the specimens were removed and subjected to mechanical stress using an electronic toothbrush for 210 seconds. Images were photographed using digital single lens reflex (DSLR) camera and coating loss measurement was done using Autodesk® AutoCAD® software. Surface topography of the specimens from each group was evaluated using scanning electron microscope (SEM). Statistical analysis showed significant ($p < 0.001$) coating loss was found in epoxy resin groups regardless of their pH value, than that of PTFE groups. SEM revealed rupture, roughness and loss of coating at multiple locations on the coating surface.

CHAPTER ONE

INTRODUCTION

1.1 Background of the study

Salivary fluid is an exocrine secretion that contains about 99% of water and the remaining composition consists of a wide range of electrolytes (sodium, potassium, calcium, phosphate, chloride, magnesium, bicarbonate) and proteins in the form of enzymes, immunoglobulin, antimicrobial agents and mucosal glycoproteins (Humphrey & Williamson, 2001). These components interact with each other and are responsible for the various functions attributed to saliva. The normal pH of the saliva ranges between pH 6 to pH 7 and is affected by factors such as secretion of organic and inorganic acids from salivary glands and acids resulting from oral microbes or acquired by nutrition (Humphrey & Williamson, 2001). Patients with poor oral hygiene are reported to have low saliva pH of 6.02 to 6.5 depending on age and sex (Pandey et al., 2015). Despite its known benefits to human, the saliva has been shown to show some undesirable effects towards resin-based dental materials that are used in dentistry.

In orthodontics, new generation of orthodontic archwires that are coated with tooth-coloured polymers have been marketed to address aesthetic concern of patients who undergo treatment with conventional stainless steel (SS) fixed appliance. This type of fixed appliance has demonstrated a negative impact on the quality of oral health related to the quality of life for patients in general (Johal et al., 2014) owing to its unsightly appearance.

The coated archwire (**Figure 1**) consists of core archwire of either NiTi or SS alloys that are coated with an outer layer of tooth-coloured polymers for instance PTFE, epoxy resin, parylene-polymer or less commonly silver-palladium. The most widely used coating polymers are PTFE or commercially known as Teflon® and epoxy resin (Kravitz, 2013).

PTFE is a polymer made up of carbon and fluorine. The coating process was carried out through surface treatment of the core archwire to support coating adhesion, followed by spraying of the atomised PTFE particles by using compressed air to coat the archwire and then placed in the kiln for heat treatment (Kravitz, 2013). The thickness of the coating layer varies from one manufacturer to another but is reportedly ranges about 20 to 25 μm (Choi et al., 2015). Apart from its excellent aesthetic property, PTFE also has physical properties that make it very acceptable in clinical practice, as it is nonreactive heat resistant and hydrophobic (Tang et al., 2016).

On the other hand, epoxy resin is a synthetic resin produced by mixing of epoxide using electrostatic coating technology to manufacture epoxy resin coated archwires. When a high voltage electrostatic charge is applied to the core archwire and an opposite charge is applied to the epoxy resin, the atomised liquid epoxy particles are sprayed onto the wire and placed in the kiln for heat treatment (Kravitz, 2013).

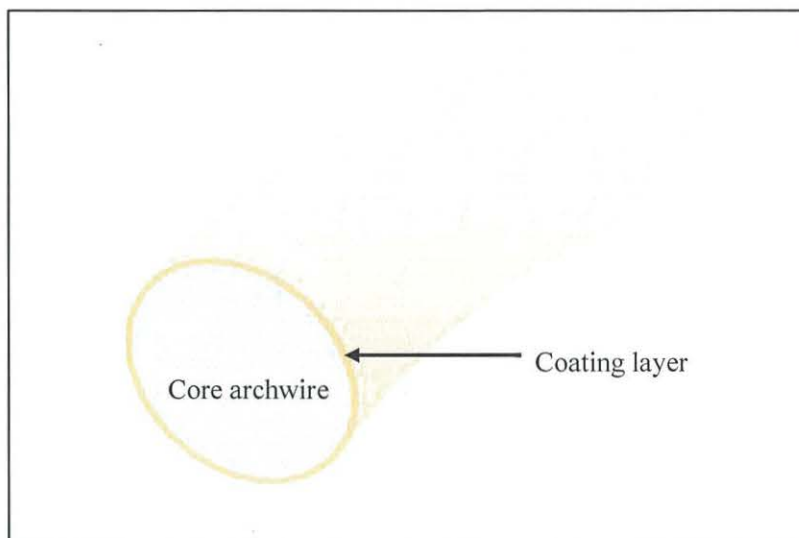


Figure 1: Cross section of coated arch wire.

1.2 Problem statement

Clinically, the coated archwires that are available in the market have demonstrated partial or full coating loss following intraoral exposure as early as 21 days (Argalji et al., 2017). It is suggested that this phenomenon occurred because of interaction that exists between the coated archwires and the surrounding oral environment such as salivary pH, enzymes, bacteria and others. However, there is no study has been done to investigate the effect of salivary pH.

1.3 Objectives

1.3.1 General objective

To investigate the effect of salivary pH on coating durability of two different aesthetic archwire coating materials.

1.3.2 Specific objectives

- 1) To investigate the effect of normal and acidic salivary pH on the coating durability of the epoxy resin and PTFE coated NiTi archwires.
- 2) To compare the coating durability of treated archwires with the untreated archwires.
- 3) To observe the surface topography using SEM.

1.4 Research hypotheses

1.4.1 Null hypothesis

Different salivary pH do not affect different types of coated archwires.

CHAPTER TWO

LITERATURE REVIEW

2.1 The impact of salivary pH on dental materials

Chemical, physical, microbiological and enzymatic changes in the oral cavity have resulted in biodegradation and erosion effect on dental materials (Kuhta et al., 2009). Apart from these factors, salivary pH also has been reported to have a strong negative effect on the properties of dental materials (Abey et al., 2014).

Kuhta et al. (2009) conducted a study on three types of orthodontic archwires with various composition and properties which included NiTi, thermo-NiTi and SS archwires. This study evaluated the effect of different pH and exposure time on ion leaching mechanism and determined the types and quantities of the released ions. They used artificial saliva with different pH values, 6.75 (average pH in the oral cavity) and lowest pH of 3.5 (found under the mature dental plaque). The pH of the artificial saliva was modified using 10 M sodium hydroxide and lactic acid. The lactic acid was selected as it is a by-product of plaque metabolism in the oral cavity. The specimens were divided into two groups before being submerged in the solutions with different pH at 37°C. The specimens were collected after the following 1, 7, 14 and 28 days. They used a mass spectrophotometer to determine the amount of ions. The result revealed that the release of ions was affected by the archwire composition, pH and exposure time. The acidic medium demonstrated higher release of ions sufficient to cause allergic reactions.

Sheibaninia (2014) studied the effect of thermocycling on the release of nickel from orthodontic NiTi archwires placed in the artificial saliva with different pH values. The study included four groups of 10 specimens that were submerged in a water bath at 37°C for 48 hours. Each specimen was placed in an individual capillary tube filled with the artificial saliva. The first and second groups had a pH value of 7.4 in which the latter was subjected to thermocycling. The third and fourth groups had pH value of 4.5 which was reduced using ascorbic acid and the latter was subjected to thermocycling. The

thermocycling process was between 5°C to 55°C for 500 cycles. This study reported similar findings from Kuhta's study (Kuhta et al., 2009) in which the release of ion was accelerated by the acidic environment.

The metals are widely used in dentistry as restorative materials, orthodontic devices, implants and others. The electrochemical properties of these metals are affected by pH value of saliva (Opydo-Szymaczek et al., 2015). They studied the effects of saliva and pH on electrochemical potential of various biomaterials used in dentistry. Seven metal alloys that are commonly used for making crowns, bridges and orthodontic devices (Wironit Extrahard, Wiron® 99, Gialloy PA, Remanium GM 380+, Gialloy CB/H, Remanium 2000+ and Gialloy CB/N), including amalgam and chromium nickel Cr-Ni steel were tested. The electrochemical conductivity of these materials in the artificial saliva was measured with pH ranging from 5.5 to 11.2. The results indicated that all materials were negatively affected by the reduced pH value except for amalgam, where the impact was weak.

Escobar et al. (2015) evaluated the effect of different salivary pH on the surface and roughness of two different types of orthodontic wires which were titanium-molybdenum alloy (TMA) and Cr-Ni steel. The specimen included 270 wire segments of 1.5 cm long. The specimens were divided equally into three groups based on the wire type and were evaluated thrice; before treatment which acted as control, followed by one month later and finally two months after treatment. The specimens were also subjected to three different values of pH 2, 5 and 7.2. Profilometer and SEM were used to measure roughness and observe the surface morphology of the wires, respectively. The result showed that the TMA was negatively affected in pH 2 and 5. The decrease in pH led to increased roughness. The Cr-Ni steel wires did not show any superficial changes, indicating that these wires have greater resistance against acidic pH.

Moon et al. (2015) studied the chromatic stability of composite resin with different shades when exposed to water with varying degrees of pH. Four different

specimens of nanohybrid composite resin were submerged in the water at pH 3, 6 and 9 for 14 days. The colour of the specimens was evaluated before and after water immersion. The results indicated that the colour of the composite resin changes differently, depending on the shade and pH factors. These changes were within the minimum limits that could be observed by the naked eye, which, in the researcher's view does not represent an aesthetic problem clinically.

Silva et al. (2007) investigated the effect of two different solutions (demineralising and re-mineralising) with varying pH (pH 4.3, 4.6, 5.0, 5.5 and 6.2) on glass ionomer cements (GIC) and polyacid modified composite resin (PMCR) that are characterised by their fluoride release. After one hour of materials preparation, the initial surface hardness was assessed. The specimens were then sent for pH cycling and submerged in the solutions for 15 days. Subsequently, the surface properties were reassessed. They concluded that low pH had reduced the release of fluoride for both materials. They also stated that the difference in pH values had reduced the hardness and modified the properties of the surface of the GIC without affecting the other.

Another *in vitro* study has evaluated the biocompatibility of orthodontic mini screws in saline solution of different pH values (pH 7 and 4) with several cell lines which consist of human osteogenic sarcoma cells (U2OS), permanent human keratinocytes (HaCaT) and primary human gingival fibroblasts (HGF). These mini screws were immersed for 1, 7, 14, 21, 28 and 84 days. Then, the liquid was taken after the expiration of the period and the cell lines were rinsed with this solution. After 24 hours, mitochondrial dehydrogenase activity was measured to assess the toxicity. The results indicated that at pH 7, there was excellent biocompatibility while at pH 4, the result demonstrated a significant toxicity response by the cells (Galeotti et al., 2013).

2.2 The drawbacks of the coated archwires

Several studies (da Silva et al., 2013; Elayyan et al., 2008; Zegan et al., 2012) have reported that there are changes in the surface topography of the coated archwires. The major changes included surface rupture, roughness, coating loss and discolouration. These changes indicate that the properties of the archwires used in orthodontics are altered by several factors including the oral environment.

The surface of the coated archwire roughened and lost its aesthetic value when 25% of the coating layer was reportedly came off within 33 days of *in vivo* use accompanied by an acute damage in the surface morphology (Elayyan et al., 2008). This study compared the mechanical and physical properties of coated archwires after intraoral use for four to six weeks with the unused ones. Ten new archwires were submitted to a three-point bending test. The surface roughness was assessed by means of contact stylus profilometer whereas the surface topography was assessed using electron and optical microscope. The same tests were performed on ten specimens after being used for an average of 33 days within the vivo and the percentage of the residual coating was calculated by digital photography.

da Silva et al. (2013) conducted a research aiming to compare the surface characteristics and coating stability for four brands of coated archwires with the characteristics of SS and NiTi archwires. Two of the coated archwires were partially coated on the labial surface and another two were fully coated. A specimen consisted of 164 segments with 20 mm length, 68 of them were unused and 96 segments were retrieved after 21 days of use within the oral cavity. The surface was observed using the SEM and the surface roughness was evaluated by atomic force microscope (AFM). Measurement of the coating loss was done by computer software after digital imaging of the specimens. This study concluded that the coated archwires showed low aesthetic value due to the unstable coating layer. After oral exposure, there was a coating loss ranging from 100% for labially coated archwires and 28.71% to 72.90% for fully coated

archwires. Additionally, the roughness of the coated archwire surface was greater than the control groups of both SS and NiTi wires following clinical use.

Another study that was carried out by Zegan et al. (2012) investigated the effect of saliva on the surface characteristics of the coated and uncoated NiTi archwires and evaluated the friction between the archwires and the brackets after *in vivo* use for two to three months. The archwires were retrieved during follow-up visits for orthodontic patients who were randomly selected within certain criteria. The specimens were sterilised after retrieval (washing with distilled water and immersing in alcohol in an ultrasonic bath for five minutes) and dried for microscopic tests. Various cross sections of the specimens were examined using dual-beam electron microscopy and spectroscopy analysis before and after clinical use. It was found at the end of this study that there was manufacturing error in the coating layer for archwires that were examined prior to clinical use. The retrieved archwires showed deposition of amorphous organic materials on the surfaces of coated NiTi archwires and peel in the coating layer was caused by the friction on the bracket-archwire interface. Changes in the atomic ratios and density of the chemical elements on the retrieved archwire surfaces were also observed by X-ray microanalysis. In addition, the depletion of nickel and titanium ions and the appearance of extra elements occurred due to interaction with saliva.

Ulhaq et al. (2017) conducted a multicentre randomised clinical trial of a relatively large specimen for four coated NiTi archwires (Bio Cosmetic, Titanol Cosmetic, TP Aesthetic and Tooth Tone) aimed to assess the alignment efficiency and its aesthetic performance. The study consisted of 150 patients (300 archwires) from four hospitals (three in the UK and one in Italy). The patients were called for follow-up after four weeks to re-tie the archwires and after eight weeks to remove the archwires. They analysed 258 archwires for discolouration and 273 for coating loss (the rests of the archwires were excluded due to lost to follow-up or fractures). They found that there was a considerable alteration in colour, which varied between the four types and higher in male patients than females. Additionally, the coating loss was reported to occur at 22.7%,

44.4%, 38.9% and 37.5% for Bio Cosmetic, Titanol, TP Aesthetic and Tooth Tone, respectively.

An *in vivo* study aimed to compare the mechanical, structural and aesthetic properties of two types of coated NiTi archwires to their uncoated counterparts in the form of as-received (the wires as-received from the manufacturer) and retrieved (the wires after clinical use). The study included 121 specimens (61 retrieved, 60 as-received). 61 patients were randomly selected and divided into four groups based on the type of NiTi archwires used during treatment. The archwires were only used in the upper jaw and were left for 4 to 12 weeks to achieve the clinical goal of treatment. After retrieving, the archwires were washed with running water, dried with gauze and placed in a coded plastic bag. The scanned archwire images was used to determine the percentage of coating loss. All retrieved and as-received specimens were subjected to a three-point bending test and all patients were given a set of questionnaires to answer. Bradley et al. (2013) concluded that all coated archwires in the test groups lost a considerable amount of the coating layer after clinical use and this loss was noticeable for patients who felt dissatisfied with this loss. The metallic properties of the as-received coated archwires were better their uncoated counterparts.

Another *in vitro* study was carried out by Iijima et al. (2011) which aimed to determine the effect of coating layer on two types of coated NiTi archwire. Two brands were selected from partially coated archwires on the labial surface. The uncoated posterior surface of the archwires and uncoated wires were used for comparison. They used the SEM to observe the cross-sectioned and outer surfaces. In addition, all ten specimens were subjected to the three-point bending test. The results indicated that the coating layer affected the behaviour of the archwire in the three-point bending test and the coated surfaces were rougher than their uncoated counterparts in both brands.

Ryu et al. (2015) investigated the surface roughness and mechanical characteristics of three types of partially coated NiTi archwires (silver-platinum, epoxy resin and PTFE coated archwires). The study consisted of a total of 45 specimens (15

specimens for each type). Uncoated parts of the archwire were used as controls. The surface was assessed using an AFM and modified three-point bending test to evaluate the mechanical properties. They reported that the PTFE group showed the lowest deviation amongst the three groups. In silver-platinum group, the coated surface was rougher than the uncoated area, while the PTFE coated archwire showed softness in the coating area more than the uncoated metal area. In addition, the epoxy group showed no significant difference between the coated and uncoated areas.

Argalji et al. (2017) compared the coating dimensions and the surface properties of two NiTi archwires coated with PTFE and unknown polymeric coating, in as-received state and after oral exposure for 21 days. The thickness and dimension of the coating layer of the as-received specimens were measured using the stereomicroscope. After 21 days of oral exposure, the specimens were removed and images were taken by a stereomicroscope. The coating loss was measured and its percentage calculated. The labial surface of both types of specimen was observed under SEM. This study concluded that both archwire types showed a significant loss of coating layer with PTFE demonstrated higher coating loss. In both types, the coating layer was thinner than those reported by manufacturers (0.002 inches).

Modification of the surface of the archwire and adding the coating layer affected the frictional property of the coated archwires than that of the uncoated one. This coating layer did not only impact the frictional property but also the aesthetic property, corrosion characteristics and mechanical durability of the archwires (Husmann et al., 2002). In contrast, plastic coatings reduced the friction ratio than that of the uncoated archwires manufactured by the same company (Elayyan et al., 2010). The coated archwires were routinely destroyed after three weeks of intraoral exposure as a result of oral enzymes activity and chewing forces (Lim et al., 1994; Kusy, 2002). In terms of frictional property, coated NiTi archwires generated lower loading and unloading forces than their uncoated counterparts that have the same dimensions (Elayyan et al., 2010).

There is also roughness in the coated archwire surface (Elayyan et al., 2008; da Silva et al., 2013) and loss of coating layer after being used *in vivo* (Elayyan et al., 2008; da Silva et al., 2013; Ulhaq et al., 2017). These coated archwires are still marketed by the manufacturers and are used clinically by orthodontists, despite the unfavourable results reported from various studies with regards to coating durability and surface changes, as well as the lack of scientific information to provide a clear understanding of their mechanical properties and vital interactions (Elayyan et al., 2010).

CHAPTER THREE

METHODOLOGY

3.1 Study design

This study was conducted using an experimental laboratory design.

3.2 Sample size calculation

There is no study has been done to look at the effect of salivary pH on the durability of coated archwires. Hence, the sample size was done based on the coating loss of the archwires. The sample size was calculated using PS software (Dupont & Plummer, 1997) based on comparing two mean formula. The power was set at 80% and the alpha, α , was at 0.05. To detect a significant coating loss of 25% (Elayyan et al., 2008), 17 archwire specimens are required in each group based on the standard deviation of 0.25 (da Silva et al., 2013). Therefore, a total of 102 archwire specimens (N=102) was required in this study.

3.3 Preparation of specimen

3.3.1 Preparation of archwire specimen

NiTi archwires of 0.018" in diameter, which were readily coated with epoxy, resin (OrthoForce® Ultraesthetic™, G&H Orthodontics®, Franklin, USA) and PTFE (Euroform™ Cosmetic, Ortho-Care LTD, Yorkshire, UK) were used. The archwires were measured using digital calliper and sectioned into smaller specimens of 20 mm long using orthodontic archwire cutter (3M Unitek, Minnesota, USA).

3.3.2 Preparation of treatment solution

Artificial saliva (Biotene®, GlaxoSmithKline, Victoria, Australia) of different pH values was prepared based on methods described by Kuhta et al. (2009) which used an average pH 6.75 and lowest pH 3.5. The pH values was adjusted using 10 M sodium hydroxide (NaOH) and lactic acid (Sigma-Aldrich®, Kuala Lumpur, Malaysia). The actual pH value was measured using pH meter (827 pH Lab, Metrohm, Herisau, Switzerland).

3.3 Treatment of archwire specimen

17 archwires specimens from each type were immersed in individual test tubes containing 10 ml artificial saliva of pH 6.75 and 3.5. Each test tube was incubated in a water bath at 37°C for 28 days. At the end of the incubation period, all archwire specimens were collected and washed with normal saline before being subjected to mechanical stress test from tooth brushing.

The archwire specimens for the control groups were washed with normal saline and ligated to acrylic jigs to receive the mechanical stress from tooth brushing.

3.4 Mechanical stress test

Prior to mechanical stress test, an acrylic block of 3 mm width x 20 mm length x 3 mm thickness was prepared with two orthodontic brackets were embedded on each end, at the distance of 10 mm (**Figure 2**). Each specimen was tied to the orthodontic brackets embedded on an acrylic block using the elastomeric module. The experimental region was subjected to mechanical stress from an electronic toothbrush (Guardian®, Selangor, Malaysia) for 210 seconds continuously. The tooth brushing duration was determined by taking into account manufacturer's recommendation of 120 seconds per brushing, twice daily for 28 days of experiment which was divided by 32 teeth. This gave a total of 210 seconds of tooth brushing per tooth (**Figure 3**).

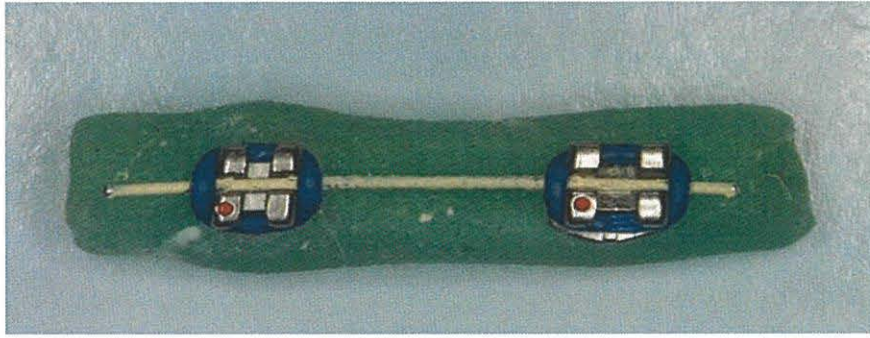
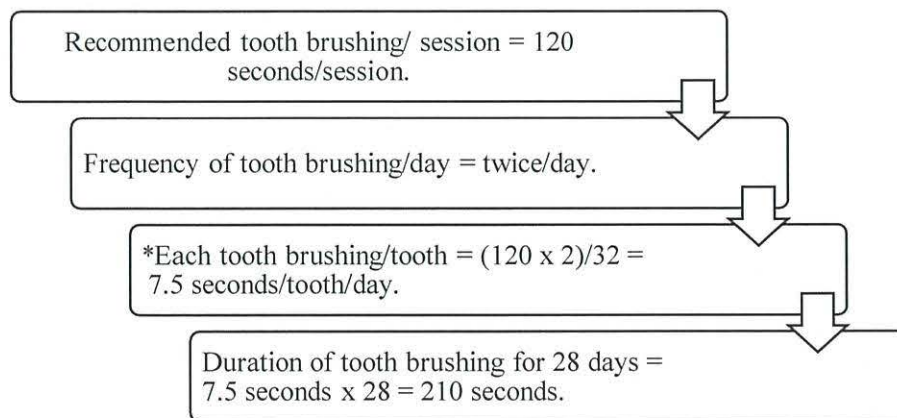


Figure 2: The specimen after ligated to acrylic bar.



*Assuming all 32 sets of teeth are present.

Figure 3: Simulation the tooth brushing routine.

3.5 Archwire photography

Each specimen was photographed using DSLR camera with 60 mm macro lens using a standardised intraoral photography using the following set up: Aperture set at f/36, shutter speed at 1/200 and the distance from camera to specimen is set at 0.1 meter (using the manual lens focus).

3.6 Research flow chart

Figure 4 shows the research pathway from the beginning by dividing the groups and treatment procedures into data analysis.

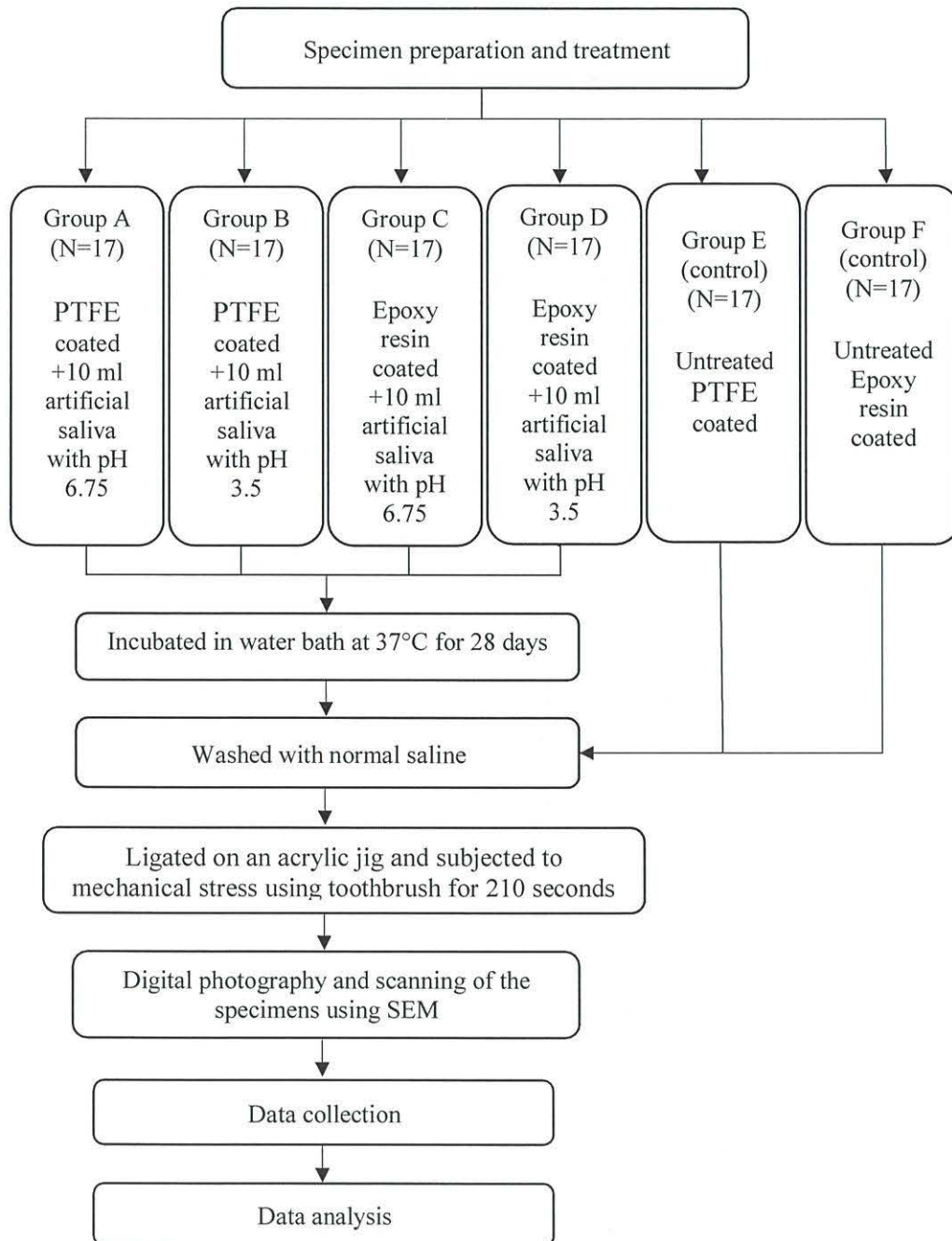


Figure 4: Flow chart of the study process.

3.7 Reliability of measurements

Prior to the actual measurement, the first examiner was trained and calibrated with the second examiner (the orthodontist) beforehand to reduce the measurement error. Ten archwire specimens were randomly selected. For intra-examiner reliability, the first examiner had performed coating loss measurement which was repeated after two weeks interval under the same set up. This measurement had been confirmed with the second examiner for inter-examiner reliability of the measurement.

3.8 Data collection

3.8.1 Coating loss measurement

Photographed images were uploaded to Autodesk AutoCAD® software version 2018 (Autodesk Inc, California, USA) and coating loss measurement was performed. The coating loss was recorded in percentage.

3.8.2 Surface topography using SEM

SEM images were obtained with two different magnifications (x200 and x400) for 12 specimens, which were (1) PTFE coated archwire treated with pH 3.5 and subjected to mechanical stress, (2) PTFE coated archwire treated with pH 6.75 and subjected to mechanical stress, (3) Epoxy resin coated archwire treated with pH 3.5 and mechanical stress, (4) Epoxy resin coated archwire treated with pH 6.75 and mechanical stress, (5) PTFE coated archwire treated with pH 3.5 without mechanical stress, (6) PTFE coated archwire treated with pH 6.75 without mechanical stress, (7) Epoxy resin coated archwire treated with pH 3.5 without mechanical stress, (8) Epoxy resin coated archwire treated with pH 6.75 without mechanical stress, (9) PTFE coated archwire subjected to mechanical stress only, (10) Epoxy resin coated archwire subjected to mechanical stress only, (11) PTFE coated archwire as-received and (12) Epoxy resin coated archwire as-received.

3.9 Statistical analyses

Data analysis was carried out using IBM Statistical Package for the Social Sciences (SPSS) version 22.0. The intra-examiner and inter-examiner reliability of the measurements were calculated using the Intraclass Correlation Coefficient (ICC) test based on a single rating for intra-examiner reliability and mean rating ($k = 2$) for inter-examiner reliability, absolute-agreement, 2-way mixed-effects model. The coating loss variable was described using percentage. The distribution of the data were tested for normality using Shapiro-Wilk test. To compare the coating loss amongst groups, non-parametric Kruskal-Wallis test was applied. This was followed by multiple pairwise comparison using Dunn's test to detect which group that was significant of all.

CHAPTER FOUR

RESULTS

4.1 Reliability of measurements

Table 1: Intra-examiner and inter-examiner reliability using ICC test

Average measures	ICC ^a	95% Confidence Interval	
		Lower Bound	Upper Bound
Intra-examiner	0.993	0.805	0.988
Inter-examiner	0.952	0.972	0.998

^a Values less than 0.5 are indicative of poor reliability, values between 0.5 and 0.75 indicate moderate reliability, values between 0.75 and 0.9 indicate good reliability, and values greater than 0.90 indicate excellent reliability.

ICC test showed excellent agreement was obtained for both intra- and inter-examiner reliability testing with ICC scores for both measures are greater than 0.90.

4.2 Normality distribution

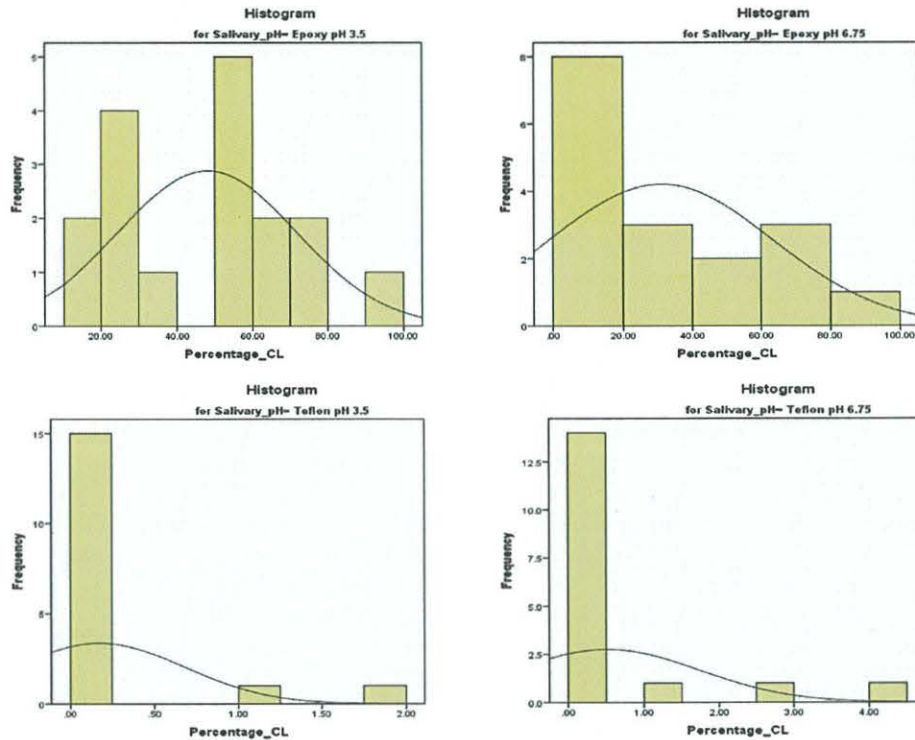


Figure 5: The distribution of the data.

The data distribution skewed to left except the data of the epoxy resin group treated with pH 3.5 distributed normally according to Shapiro-Wilk test.

4.3 Comparison of coating loss between all groups

Table 2: The percentage of coating loss between six groups using Kruskal-Wallis test.

Group (N=17)	Mean $\pm$ SD	Median $\pm$ IQR ^a	χ^2 statistic (df) ^b	P value ^b
Epoxy resin pH 3.5	48.1 $\pm$ 23.55	-	75.38 (5)	< 0.001
Epoxy resin pH 6.75	-	23.0 $\pm$ 62.62		
Epoxy resin (control)	-	0.00 $\pm$ 0.00		
PTFE pH 3.5	-	0.00 $\pm$ 0.00		
PTFE pH 6.75	-	0.00 $\pm$ 0.00		
PTFE (control)	-	0.00 $\pm$ 0.00		

^aThe distribution is skewed to the left.

^bKruskal-Wallis test.

There was a considerable loss found within treated epoxy resin groups. The epoxy resin group treated with pH 3.5 showed the highest percentage of coating loss (48.1% $\pm$ 23.55) compared to the rest of the groups. The Kruskal-Wallis test indicated that there was a statistically significant difference between the groups ($p < 0.001$). However, since Kruskal-Wallis test could not specify which group was significantly different, further post hoc multiple pairwise comparison test was carried out.

4.4 Multiple pairwise comparison

Table 3: Post hoc comparison using Dunn's test. Mean differences shown.

	Epoxy resin pH 3.5	Epoxy resin pH 6.75	Epoxy resin (control)	PTFE pH 3.5	PTFE pH 6.75	PTFE (control)
Epoxy resin pH 3.5	-	1.000	< 0.001*	< 0.001*	< 0.001*	< 0.001*
Epoxy resin pH 6.75		-	< 0.001*	< 0.001*	0.001*	< 0.001*
Epoxy resin (control)			-	1.000	1.000	1.000
PTFE pH 3.5				-	1.000	1.000
PTFE pH 6.75					-	1.000
PTFE (control)						-

*indicates the mean difference is significant at 0.05 level.

Groups in vertical column considered as significant groups.

Pairwise comparison of epoxy resin and PTFE groups treated with pH 3.5 shows that the epoxy resin group has significant changes ($p < 0.001$) compared to PTFE. The same result was observed when comparing epoxy resin and PTFE groups treated with pH 6.75 while there were no significant changes when comparing the epoxy resin group treated with pH 3.5 and pH 6.75 as well as when comparing the PTFE group treated with pH 3.5 and pH 6.75. The epoxy resin control group showed a significant difference when compared to the treated groups. Meanwhile, the PTFE control group did not show any significant difference when compared to the treated PTFE groups. These differences indicate that salivary pH does not influence these changes.

4.5 Surface topography

4.5.1 Intra-group comparison

Both treated epoxy resin groups showed severe surface changes (**Figure 6A, 5B**). The severity of these changes varies depending on the pH value. The group treated with pH 3.5 demonstrated significant coating loss whilst the group treated with pH 6.75 showed noticeable change to the surface morphology evidenced by presence of cracks and ruptures. The control group **Figure 6C** showed localised abrasion along the wire specimen which was caused by friction from the toothbrush. In addition, **Figure 6D** showed manufacturing defects which can be observed on the epoxy resin.

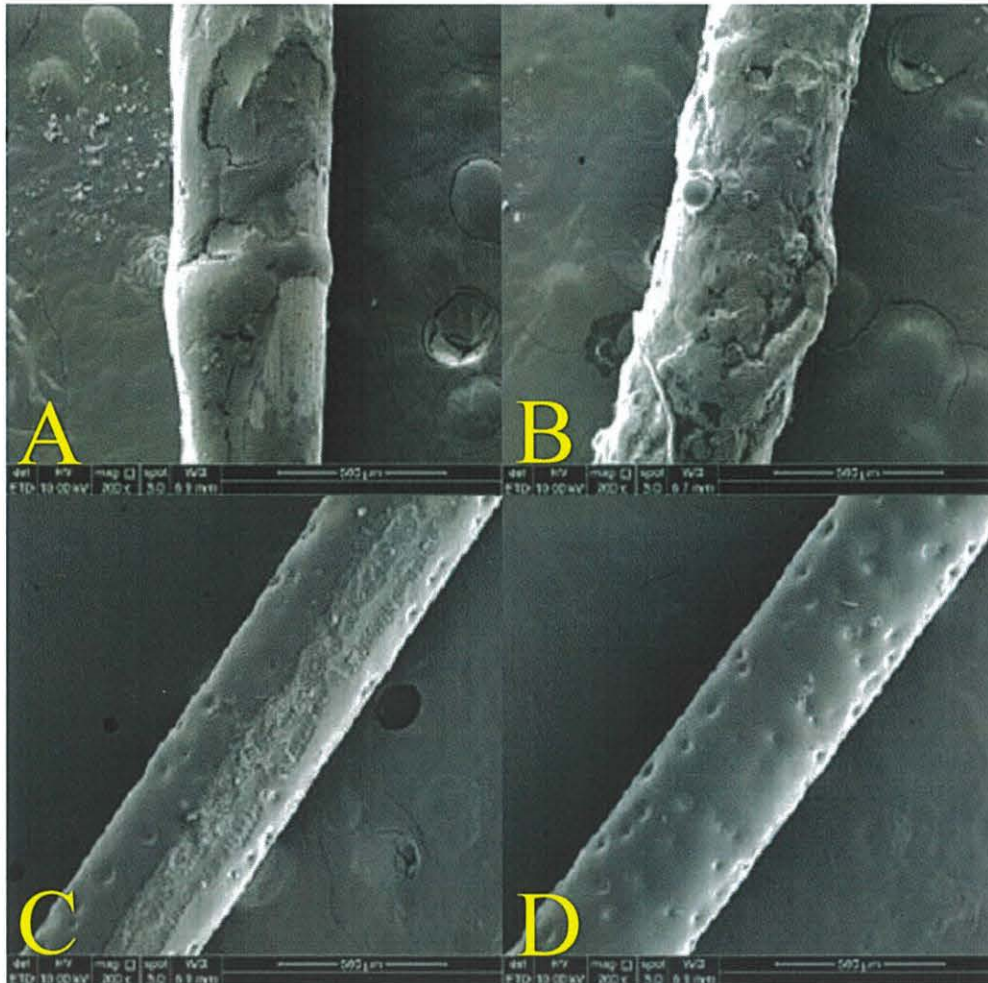


Figure 6: Epoxy resin coated archwires at magnification 200x. A, treated with pH 3.5 followed by mechanical stress. B, treated with pH 6.75 followed by mechanical stress. C, non-treated subjected mechanical stress only. D, as-received.

In PTFE treated groups, the most significant changes were observed in the pH 3.5 group (Figure 7A), although these changes were not visible by the naked eye, but under SEM, the changes are manifested as a decomposition on the coating layer. PTFE 6.75 (Figure 7B) group has a change somewhat similar to those in the control group (Figure 7C), where relative roughness in both groups can be observed if compared to as-received specimen (Figure 7D).

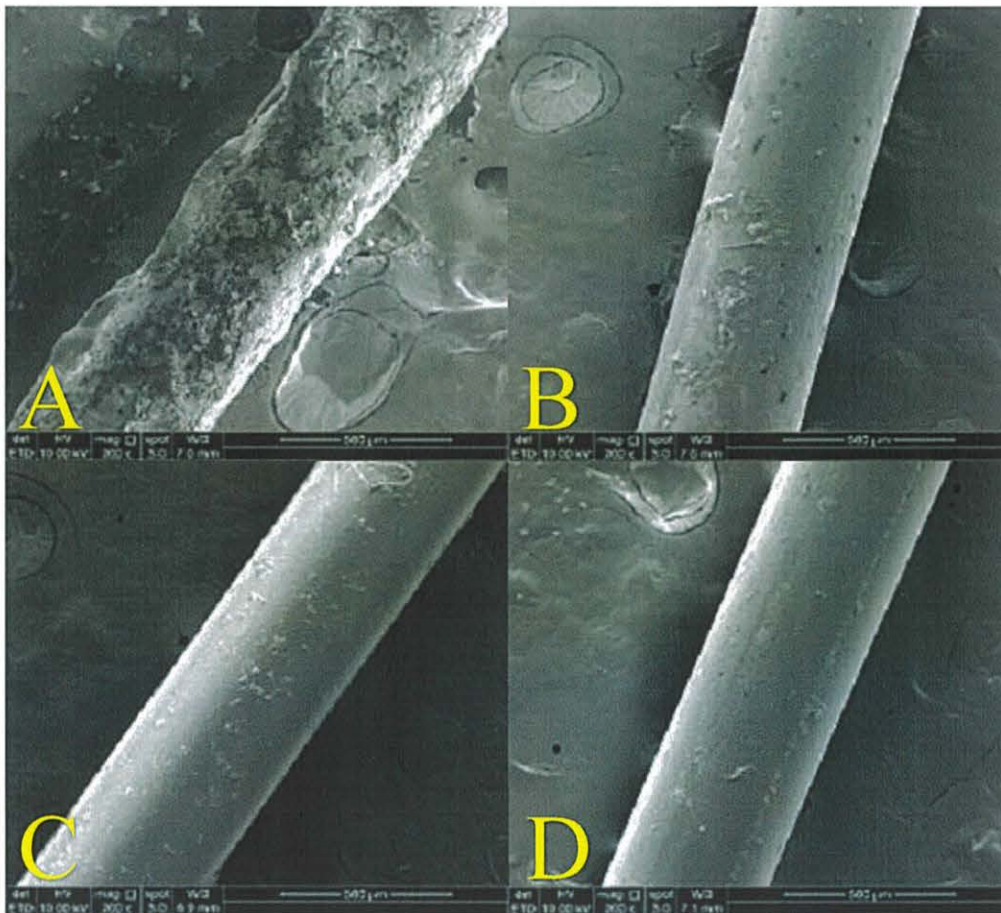


Figure 7: PTFE coated archwires at magnification200x. A, treated with pH 3.5 followed by mechanical stress. B, treated with pH 6.75 followed by mechanical stress. C, untreated subjected to mechanical stress only. D, as received.

4.5.2 Inter-group comparison

SEM images of coated archwires were presented below in **Figures 8, 9, 10, 11, 12** and **13**. There was an alteration on the surface topography of both epoxy resin and PTFE groups which were treated in the acidic medium of pH 3.5 followed by mechanical stress test. This surface alteration was manifested as fractures and ruptures on the coating layer as well as localised coating loss exclusively found on the epoxy resin coatings (**Figure 8**).

In **Figure 9**, both epoxy resin and PTFE coated archwires which were treated with pH 6.75 followed by mechanical stress test were compared to one another. The comparison revealed there was destruction on the surface of the epoxy resin coating layer and negligible changes on the surface characteristics of the PTFE layer. In contrast, the non-treated control groups that have been subjected to only mechanical stress test showed localised damage along the wire on the epoxy resin layer while some minor roughness was observed on the PTFE layer (**Figure 12**). When both types of coated archwires were treated solely in the two different acidic medium, there were coating loss on the epoxy resin coating layer treated with acidic pH whereas the PTFE layer showed relative stability with no significant changes to its surface topography in both different pH values (**Figure 10** and **11**). As a result, epoxy resin coated archwires were affected and showed considerable alterations in all treatment groups. However, the PTFE coated archwires showed considerable changes only in the group that was treated with the acidic medium (pH 3.5) followed by mechanical stress using the toothbrush.

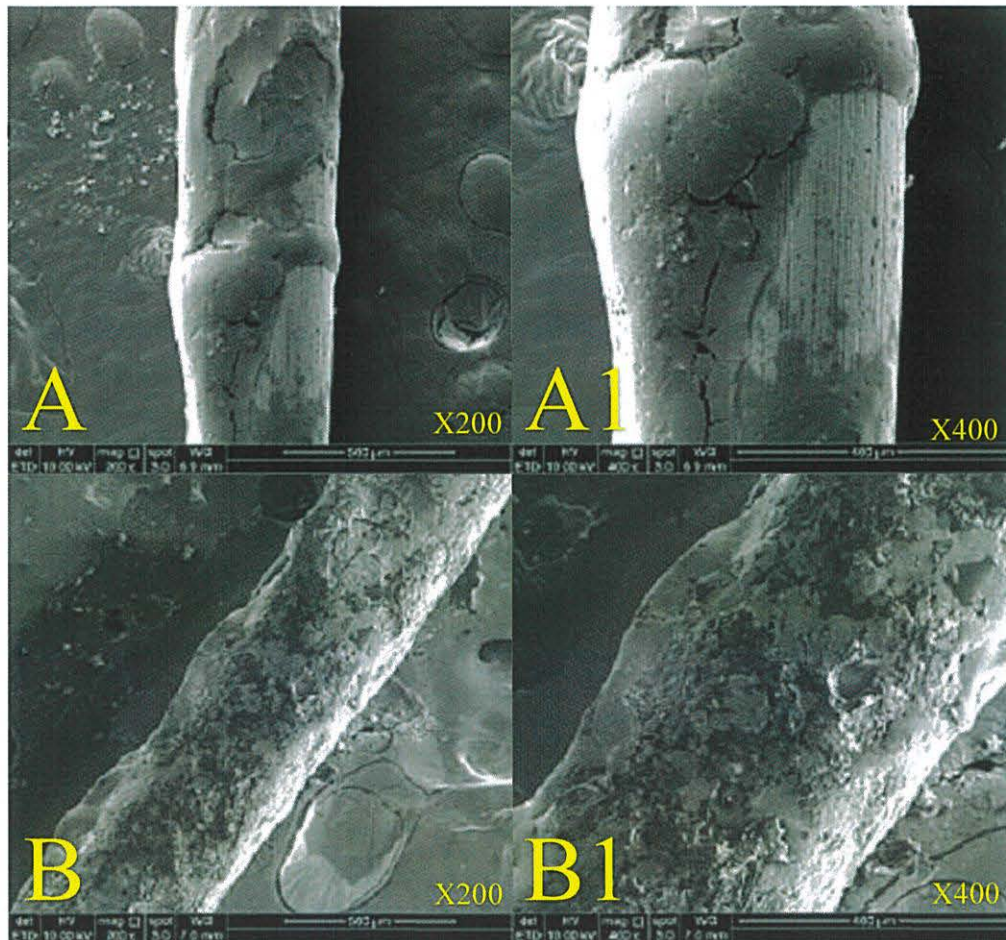


Figure 8: Treated groups. (A, A1) Epoxy coated archwire treated with pH 3.5 followed by mechanical stress. (B, B1) PTFE coated archwire treated by pH 3.5 followed by mechanical stress.