The effect of food simulants on Corrosion of simulated fixed Orthodontic appliance

(An in vitro study)

A thesis submitted to the council of the College of Dentistry at the University of Baghdad, in Partial fulfillment of the requirements for the Degree of Master in science of Orthodontics.

by
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بسم الله الرحمن الرحيم

وَأَنزَلَ اللَّهُ عَلَيْكَ الْكِتَابَ
وَالْحِكْمَةَ وَعَلَّمَكَ مَالَمْ تَكُنْ تَعْلَمُ
وَكَانَ فَضْلُ اللَّهِ عَلَيْكَ عَظِيمًا

صدق الله العظيم

سورة النساء – الآية ١١٣

بسم الله الرحمن الرحيم

Dedication

To

My wonderful parents

Beloved sister and

Brothers

Certification of the Supervisor

I certify that this thesis entitled **"*The effect of food simulants on Corrosion of simulated fixed Orthodontic appliance*"** was prepared by **(Afaf Hameed Hussien)** under my supervision at the University of Baghdad in partial fulfillment of the requirements for the Degree of Master of Science in Orthodontics.

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Certification of the examining committee and the signature of the dean

We, the members of Examining Committee, certify that after reading the thesis and examining the student ***Afaf Hameed Hussien*** in its contents, we think it is adequate for the award of the Degree of Master of Science in Orthodontics.

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ABSTRACT

Fixed orthodontic appliances were considered to be a source of human exposure to different elements used in manufacturing the components of these appliances. Physical and chemical properties of food and liquids can affect the corrosion of these appliances. So this in vitro study was designed to determine the effect of dietary simulating liquids that representing the fatty and acidic products and artificial saliva, so the oral fluid respectively on the corrosion rate and the amount of ions released from fixed orthodontic appliances.

In this study, we used a seventy set of fixed orthodontic appliances (Ortho Technology, USA) each one simulated half maxillary arch. These sets of orthodontic appliances were divided in to seven groups, each group contain ten sets. Then these appliances immersed in each of the storage solutions which include: artificial saliva, distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, and 1% citric acid, the immersion done at 37° for 28 days, and the storage solutions were changed on each 7 days interval during the whole storage period to avoid saturation of solutions with ions.

At the end of each storage period, a sample of the testing solution was collected from each group for elemental analysis. The measurement of the released chromium, nickel, copper, and iron ions was done by using the Atomic Absorption Spectrophotometer and Spectrophotometer. Weighing the orthodontic sets was done before and after each immersion period. The types and distribution of the corroded areas were examined using high resolution optical microscope.

The results of immersion studies revealed that the higher amounts of chromium, nickel, copper and iron ions were released on the first 7 days of the study, the higher levels of ions were released in the 1% citric acid and 3% acetic acid solutions, and the least amount of the ions were released in corn oil and distilled water solutions.

The results of microscopical examination showed that pitting and crevice corrosion was the most common type of corrosion seen in different parts of the appliances, with more sever corrosion seen in appliances immersed in acidic solutions, and the least amount of corrosion was seen in appliances immersed in corn oil and distilled water.

The results of weighing the orthodontic sets revealed that there was a changes in the weight of the appliances during the study, and all the appliances has a higher weight at the end of the study.

The overall findings refer that the corrosion rate of orthodontic appliances increases with decreasing the PH of the solutions, and the presence of oil decreases the corrosion rate of these appliances. Corrosion could change the surface roughness that might result in higher friction during sliding of the bracket along the archwire. As organic acids facilitate the release of metal ions, so oral hygiene could be an important factor in reducing corrosive events.

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Lists of Abbreviation

%	*Percent*
&	*And*
AAS	*Atomic absorption spectrophotometry*
ANOVA	*Analysis of variance*
C°	*Degree celluloid*
Ca	*Calcium*
Cl	*Chloride*
Cr	*Chromium*
Cu	*Copper*
CuNiTi	*Nickel titanium cupper chromium*
d.f.	*Degree of freedom*
Dw	*Distilled water*
ed.	*Edition*
eg.	*Example*
EU	*European Union*
F	*Fissure exact test*
Fig	*Figure*
FDA	*Food and Drug Administration*
Fe	*Iron*
G	*Gram*
Gr	*Group*
H	*Hydrogen*
HS	*Highly significant*
i.e.	*That's to mean*
LSD	*Least significant test*
Max	*Maximum*
Min	*Minimum*
µg	*Microgram*
ml	*Milliliter*
mm	*Millimeter*
Mn	*Manganese*
Ni	*Nickel*
nm	*Nanometer*
NS	*Non significant*
O	*Oxygen*
P	*Probability value*

PH	***Minus logarithm the hydrogen ion***
ppm	***Part per million***
S	***Significant***
SD	***Standard deviation***
SPSS	***Statistical package for social science***
Ti	***Titanium***
TiNb	***Titanium niobium***
TMA	***Titanium molybdenum alloy***
UV	***Ultraviolet***
V	***Volume***
VHS	***Very highly significant***
W	***Weight***
WHO	***World Health Organization***

Introduction

Fixed orthodontic appliances usually include brackets, bands, and archwires. Most orthodontic bands, brackets, and archwires are made of stainless steel containing 8% to 12% nickel, 17% to 22% chromium, and various proportions of manganese, copper, titanium, and iron***(Gursoy et al, 2005; Kuhta et al, 2009; Sfondirini et al, 2009)***. The marked increase of orthodontic treatment has drawn attention to potential undesired effects ***(Staffolani et al, 1999)***.

In dentistry one of the most important factors affecting the choice of dental metals and alloys is their biocompatibility ***(Kedici et al, 1998)***. These alloys have to be fully biocompatible and must elicit an appropriate biological response within a host. Biocompatibility testing of dental material consists of three different phases. An in vitro laboratory tests and in vivo tests on animals have been done; however, clinical tests on patients, which are the most relevant, are very complex and could raise ethical concerns ***(O'Brien, 1997; Wataha, 2001; Kuhta et al, 2009)***.

In the oral environment, orthodontic appliances are exposed to potentially damaging physical and chemical agents which may cause metallic corrosion. Factors such as quantity and quality of saliva, salivary PH, plaque, the amount of protein in the saliva, physical and chemical properties of foods and liquids, and general and oral health conditions may influence corrosion in the oral cavity. Corrosion will occur continuously in the mouth, due the release of ions with abrasion by foods, liquids and tooth brush ***(Huang et al, 2001; Huang et al, 2004; Sfondirini et al, 2009)***. Brackets are subjected to corrosion in the oral cavity because they are immersed in the patient's saliva, that acting as an electrolyte. Additional factors influencing corrosion are varying oral temperatures, the presence of plaque and the daily dietary intake. Oxygen required for corrosion is present in abundance ***(Costa et al, 2007; luft et al, 2009)***.

The oral environment is very conducive to the formation of corrosion products. The mouth is moist and continually subjected to fluctuations in

temperature. Foods and drinks cause transitory, but important and wide, variations in the chemistry of the environment. The food and liquids ingested have wide ranges of PH. Acids are liberated during breakdown of foodstuffs. This food debris often adheres tenaciously to the metallic restoration providing a localized condition that is extremely conducive to an accelerated reaction between the oral media and the metal or alloy ***(Duffó and Quezada, 2004; Duffó and Farina, 2009)***.

Therefore, the present study has been established to evaluate the effect of food simulating factors on the corrosion resistance and the biocompatibility of orthodontic appliances. Chemical corrosion analysis and microscopical examination are shown to be valuable tools for providing data about the corrosion resistance and the biocompatibility of these appliances.

Aims of the study

This investigation was performed to study the effects of food simulants (aqueous food, alcoholic food, acidic food, and fatty food) in addition to distilled water and artificial saliva during different time intervals on the corrosion behavior of fixed orthodontic appliances by evaluating:

1. The concentration of chromium, nickel, copper, and iron ions released from orthodontic appliances.
2. Weight changes of the orthodontic appliances before and after immersion in each solution.
3. The corrosion types and distribution of the corroded regions on surfaces of different parts of orthodontic appliances.

Chapter One
Review of Literature

Chapter one

Review of literature

1.1 Fixed Orthodontic Appliance

Fixed appliances form a major part of orthodontic appliance system. A fixed appliance is an orthodontic device where attachments are fixed to the teeth and forces applied by archwire or auxiliaries through these attachments. These allow precise control over the nature and direction of the forces applied and the patients cannot remove it by himself or herself ***(Isaacson, 1984; Rani, 1995; Jones & Oliver, 2000)***.

1.1.1 Components of Fixed Appliances

The principle components of fixed orthodontic appliances are ***(Isaacson, 1984; Al-Mulla, 2009)***:

A. Attachments.

B. Archwires.

C. Auxiliaries.

1.1.1.1 Attachments

The orthodontic attachments are these components of the fixed appliance to which the force component can be applied **(*Reynolds, 1975; Demuth, 1981; Foster, 1985)***.

1.1.1.1.1 BRACKETS

It can be defined as precisely fabricated orthodontic attachment made of metal, plastic or ceramic material which can be bonded to a tooth or welded to a band ***(Daskalogiannakis, 2000)***. Brackets should have the correct hardness and strength. They should have a smooth archwire slot to reduce frictional resistance, and an otherwise smooth surface to reduce plaque deposition ***(Oh et al, 2005)***.

They can be classified according to the bracket material into:

A. Metal Brackets:

1. STAINLESS STEEL BRACKETS

The use of metal brackets with retentive bases was first reported by ***Mitchell in 1967***. Although not as esthetically pleasing as plastic, ceramic or fiber glass brackets, the stainless steel brackets were an esthetic improvement over previously used band ***(Graber and Swain, 1991; Proffit et al, 2007)***, Fig (1–1).

Most orthodontic brackets are made of stainless steel containing Nickel (Ni), Chromium (Cr) , and various proportion of Manganese (Mn), Copper (Cu) , Titanium (Ti) , and Iron (Fe) ***(Gürsoy et al, 2005)***.

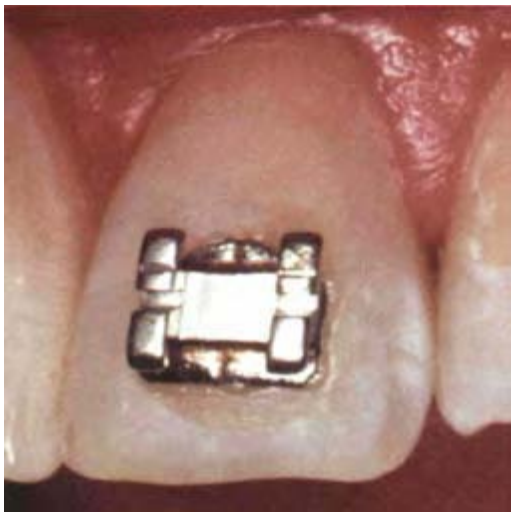

Fig (1–1) Stainless steel brackets *(Proffit et al, 2007)*

The color of the metal and its visibility may be objectionable to some adult patients. Manufacturers have tried to reduce the size of the bracket and hence its visibility by continuously redesigning the appliance for aesthetic reason, and for the mechanical advantage of increased interbracket distances ***(Hamula et al, 1996; Bishara, 2001)***. The base alloy is of a softer metal to facilitate easier debonding of the brackets, where as the tie-wing metal requires greater hardness in order to withstand the forces applied by the archwires ***(Eliades et al, 2003)***.

Metal orthodontic brackets are now made from five different American iron and steel institute types of stainless steel. Nominal compositions of the five different types of stainless steel are listed in Table (1-1). Smaller and more attractive and more

comfortable metal brackets can be made without compromising the force to deform if the metal brackets are made of stronger raw materials ***(Flores et al, 1994)***.

Table (1–1) Stainless steel alloy composition (*Flores et al, 1994*)

Raw materials	Cr	Ni	Mn	C	P	S	Si	Mo	Se
1. 310 SS	24.0 26.0	19.0 22.0	2.0	0.08	0.04	0.03	1.5	0.75	
2. 313 SE	17.0	8.0 19.0	2.0 10.0	0.15	0.20	0.06 0.17	1.0		0.15 0.35
3. 316 L	16.0 18.0	10.0 14.0	2.0	0.03	0.04	0.03	1.0	2.0 3.0	
4. 303 S	17.0 19.0	8.0 10.0	2.0	0.15	0.04	0.18 0.40			
5. 17-4 PH	15.5 17.5	3.0 5.0	1.0	0.07	0.04	0.03	1.0		

Stainless steel brackets are easy to manufacture, tough, cheap and can be recycled ***(Maijer and Smith, 1982; Foster, 1985)***. Moreover they can be produced either by casting, or from thin metal strip material that is stamped to shape. In an effort to prevent ion release, metal injection molding (MIM) has been introduced to construct a single uniform unit bracket to eliminate the possibility of galvanic corrosion that can occurred within the brackets components ***(Siargos et al, 2007)***. Nowadays, most of brackets are casted, because they are more effective to be used with straight wire techniques in contemporary orthodontics ***(Proffit et al, 2007)***.

In spite of the use corrosion resistant stainless steel alloys, corrosion of these brackets occurs and green and black stains have all been noticed ***(Ceen and Gwinnet, 1980; Maijer and Smith, 1982; Brantley and Eliades, 2001)***, Fig (1–2).

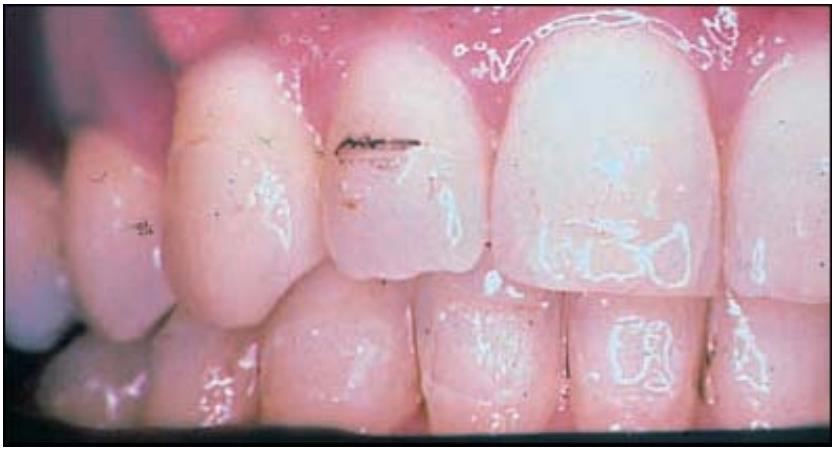

Figure (1–2) Corrosion attack of stainless steel alloy identified following debonding *(Brantley and Eliades, 2001)*

2. *NICKEL FREE AND LOW NICKEL ALLOY BRACKETS*

Most alloys used in orthodontics contain the potentially toxic nickel that might cause sensitivity and allergic reaction for the patient ***(Craig and Ward, 1997; Eliades and Athanasiou, 2002; Menezes et al, 2007)***. Nickel containing alloys can undergo corrosion with the release of metal ions, because they remain in the oral cavity and subjected to the oral environment's physical properties (chemical and microbiological properties), that stimulate the dissolution of metals ***(Setcos et al, 2006; Freitas et al 2008)***.

Non-nickel and low-nickel stainless steels were introduced in orthodontics as alternatives to conventional types. These steels contain substantially less nickel relative to conventional types and the same or even higher hardness and corrosion resistance relative to the types of steel used for bracket manufacturing ***(Eliades, 2007)***.

3. *TITANIUM BRACKETS*

The introduction of new, pure titanium brackets (Rematitan) that is competitive in cost to stainless steel bracket, has been solved the problems of corrosion and nickel sensitivity. It is one piece construction, requires no brazing layer, and thus it is solder and nickel free. Titanium brackets are made from pure, medical grade titanium that has greater strength, lower density and lower modulus of elasticity. Also, titanium has low thermal conductivity and it imparts none of the

metallic taste of stainless steel brackets ***(Hamula et al, 1996; Craig and Ward, 1997; Kapur et al, 1999)***.

Titanium and titanium-based alloys have the greatest corrosion resistance of any known metal (except pure gold and platinum) and have excellent compatibility with biological tissues ***(Schroeder et al, 1981; Craig and Ward, 1997; Gioka et al, 2004; Graber et al, 2005)***, Fig (1–3).

Figure (1– 3) Rematitan bracket (titanium bracket) ***(Abdulameer, 2008)***

B. Plastic Brackets

During the early 1970s, plastic brackets were marketed as the esthetic alternative to metal brackets ***(Russell, 2005; Chen et al, 2007)***. The main problems of plastic brackets is discoloration because of ultraviolet (UV) light and food dyes, decrease hardness and wear resistance, slot distortion caused by water absorption, and inability to withstand the torquing forces generated by rectangular wires ***(Reynolds, 1975; Bishara and Trulove, 1990; Faltermeier et al, 2007; Jena et al, 2007)***. Ceramic-reinforced, fiberglass-reinforced and polycarbonate brackets reinforced with metallic insert on the slot were subsequently introduced to provide rigidity to the bracket and enable the use of torquing forces **(*Graber and Swain, 1991; Feldner et al, 1994; Oh et al, 2005; Zinelis et al, 2005; Jena et al; 2007)***.

Steel slotted plastic bracket are useful as an esthetic alternative, but added bulk is required to provide adequate strength of the tie-wings ***(Graber et al, 2005;***

Liu et al, 2005). Despite these alterations, the clinical problems like distortion and discoloration persisted. Thus plastic bracket are used only when complex tooth movements are not required ***(Brantley and Eliades, 2001; Jena et al; 2007; Proffit et al, 2007)***, Fig (1–4).

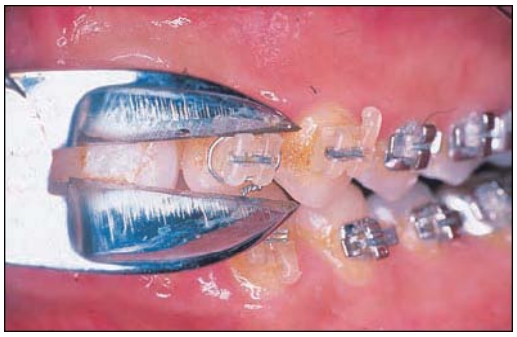

Figure (1–4) Plastic bracket exhibit bonding distortion during deboning *(Brantley and Eliades, 2001)*

C. Ceramic Brackets

In the mid 1980s, the ceramic brackets made from mono-crystalline and polycrystalline ceramic materials become widely available. The manufacturing process of mono-crystalline brackets results in a purer structure, a smoother surface, and a considerably harder substance than the fabrication of polycrystalline brackets. Ceramic brackets are designed to combine esthetic of plastic brackets and the reliability of metal brackets ***(Wang et al, 1997; Brantley and Eliades, 2001; Jena et al, 2007; Lee, 2008; Özcan et al, 2008)*** Fig (1–5).

Figure (1–5) Polycrystalline bracket (first, third, and fourth from the left), single crystalline bracket (second from the left) *(Brantley and Eliades, 2001)*

Ceramic brackets are more esthetic than metal brackets, and unlike plastic brackets, they resist staining and discoloration ***(Russell, 2005; Meguro et al, 2006; Chen et al, 2007; Jena et al, 2007; Reicheneder et al, 2007)***, Fig (1–6).They were

not only more transparent than plastic brackets but the color was also more stable. The ceramic brackets are also quite strong; these are more difficult to deform than plastic brackets and have higher or equal bond strength to that of stainless steel brackets ***(Gwinnett, 1988; Britton et al, 1990; Spiro et al, 1990; Viazis et al, 1990)***.

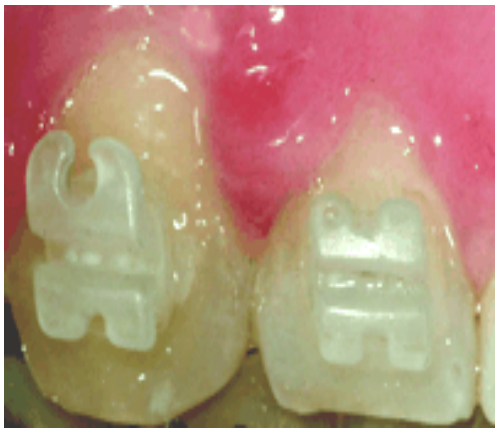

Figure (1–6) Polycrystalline ceramic bracket (***Russell, 2005)***

Nevertheless, the use of ceramic brackets may result in problems with excessive bond strength, damage to enamel during removal, high coefficient of friction between the bracket and the archwire, and bracket breakage because of brittleness ***(Gafari , 1992; Arici and Regan, 1997; Bishara et al, 2001; Johnsen et al, 2004; Neshio et al, 2004; Faltermeier et al, 2007)***. The newer designs of ceramic brackets offer excellent optical properties and the promise of additional esthetic appeal without significant functional compromises. Ceramic brackets are durable, allow adequate force control over long treatment periods, the risk for discoloration is minimal, and their debonding characteristics were improved, therefore, enamel damage risk during this stage is eliminated ***(Eliades, 2007; Gautam and Valiathan, 2007)***.

D. Composite Brackets

Composite bracket is manufactured from special thermoplastic polyurethane under advanced heat treatment system. The manufacture is based on the controlled addition of fillers to a synthetic resin matrix, which is then subjected to injection molding. It provides greater strength, sliding mechanism; and surface hardness than ceramic brackets. Its ability to withstand higher torquing force allows superior

control during treatment ***(Powers et al, 1997***; ***Birnie and Harradine, 2005; Lee, 2008)***.

1.1.1.1.2 BANDS

Until the 1980s, the only practical way to place a fixed attachment was to put it on a band that could be cemented to a tooth. The pioneer orthodontists of the early 1900s used clamp bands, which were tightened around molar teeth by screw attachments. Preformed steel bands came into widespread use during the 1960s, but are used now primarily for molar teeth ***(Gardiner et al, 1998; Benson and Douglas, 2007; Proffit et al, 2007)***, Fig (1–7).

The original edgewise appliances involved the banding of all teeth. These bands were customized for each patient. Hence bands were prepared by stretch molding the band material around the tooth, creating a joint, and then the brackets were welded to the bands. This involved a long appointment to fit the bands and cement the appliance in place. The introduction of preformed bands changed this procedure significantly. Preformed bands are available in a variety of sizes and follow the average anatomy for each tooth type. Minor tooth variations require band adaptation by the clinician to ensure a proper fit ***(Bishara, 2001)***.

Figure (1–7) Molar band *(Proffit et al, 2007)*

Orthodontic bands are made exclusively from stainless steel material. The buccal attachments on the molar bands vary according to the number of tubes and hooks incorporated in the design. These may be single, double, or triple tubes, depending on the practitioner's philosophy and requirements. Varieties of lingual

attachments are also available and can be preselected by the clinician. These attachments are generally casted separately and then welded to the preformed bands ***(Bishara, 2001)***. Fig (1–8)

Figure (1–8) A, Triple-tube molar attachment. B, lingual attachment *(Bishara, 2001)*

1.1.1.1.3 TUBES

Tubes which are usually fitted in the last molars in the arch may be round or rectangular in section Fig (1–8A); larger tuber is used to take extra-oral arches. These tubes usually pre-welded on molar band and can be cemented on molar teeth ***(Bishara, 2001; Al-Mulla, 2009)***, Fig (1–9).

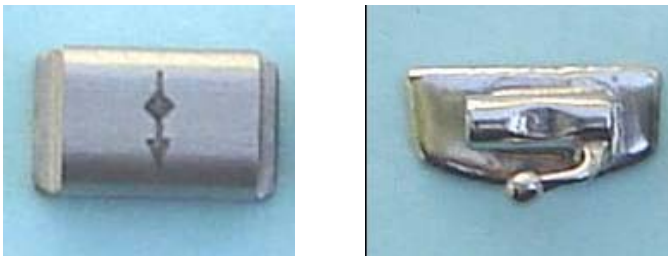

Figure (1–9) Molar tube*(Karam, 2006)*

1.1.1.1.4 BUTTONS AND CLEATS

Buttons and cleats can also be bonded to the teeth as an attachment of auxiliary forces ***(Al-Mulla, 2009)***. For impacted or un-erupted tooth a button or hook is better than standered bracket because it is smaller. It is possible to bond a button to a rotated tooth so that a rubber band can be used to rotate it ***(Proffit et al, 2007)***.

1.1.1.2 Archwires

The archwire is that part of a fixed appliance that spans the mesiodistal distances between teeth crowns and is possibly the principal component of active,

fixed-appliance therapy ***(Nikolai, 1997)***. The orthodontic archwire is a vital component of the fixed orthodontic appliance. An ideal archwire should be able to move teeth with a light, continuous force. This force should be designed to minimize patient discomfort, tissue hyalinization, and root resorption. When a force is applied, the archwire should behave elastically over a period of weeks to months ***(Gurgel et al, 2001)***.

1.1.1.2.1 Criteria for an Ideal Archwire

The ideal orthodontic archwire properties can be described in large terms of the following criteria, but in contemporary practice, no one archwire meets all these requirements, and the best result are obtained by using a specific archwire for specific purpose, the criteria are ***(Kusy, 1997; Laura and Nigel, 2000)***:

Strong, resilience, good range, stiffness, toughness, esthetic, springiness, formable, good spring back, weldable, solderable, biocompatibility, poor bio-host, low friction, low cost and high cross–sectional accuracy.

1.1.1.2.2 Types of Archwire

Orthodontic wires can be classified according to chemical composition, microstructure, or mechanical properties. The first two factors determine the third.

It is important to remember that composition alone does not predetermine the properties, since the micro-structural arrangement of the various components has a significant secondary influence ***(Burstone and Goldberg, 1980)***.

Also archwires may be classified by ***(Birnie, 2005)***:

- ***MATERIAL*** (Fig. 1-10)**:** into stainless steel, elgiloy, titanium alloys, glass, polymers.
- ***COATED OR NON-COATED:*** into ion implantation, spray coating, sleeving.
- ***MORPHOLOGY:*** into round, rectangular, single, multistrand or braided.
- ***COMPOSITE:*** into
  - ❖ Made of more than one morphology (e.g.: wonder wire).
  - ❖ Made of one or more materials with a mechanical join or material.

- Having different properties in different sections of the archwire (e.g.: GAC Bioforce, and Forestadent TripleForce).

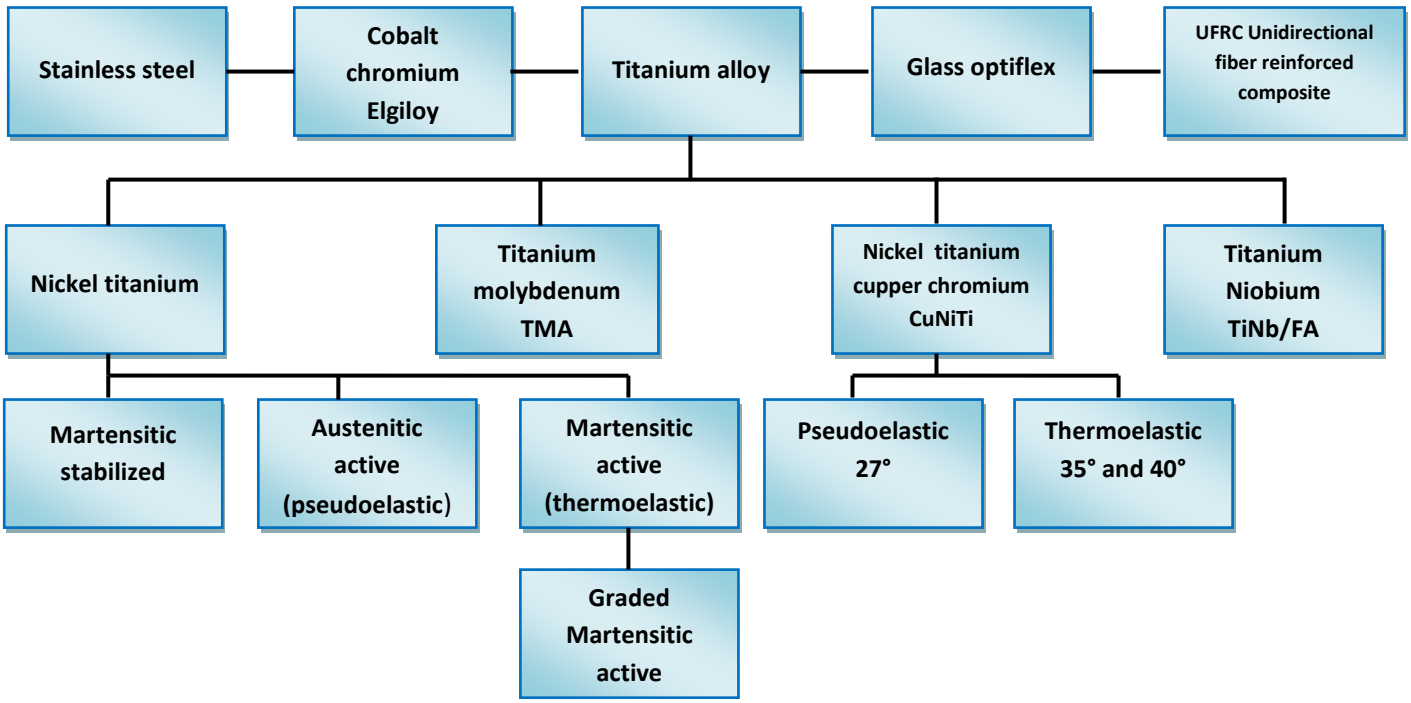

Figure (1- 10) Orthodontic archwires classified by material *(Birnie, 2005)*

1.1.1.2.3 Orthodontic Archwire Materials

1.1.1.2.3.1 Precious Metal Alloys

In the first half of the 20th century, precious metal alloys were used routinely for orthodontic purposes, primarily because nothing else would tolerate intraoral conditions. Gold itself is too soft for nearly all dental purposes, but alloys, which often included platinum and palladium along with gold and copper, could be useful orthodontically. These alloys could be heat treated to harden them and then provide a light gentle pressure, however it is prone to distortion and recently their cost has made its use unreasonable ***(Mills, 1987; Proffit et al, 2007)***.

1.1.1.2.3.2 Stainless Steel Alloys

It is an iron–carbon alloy to which 12% - 30% chromium is added. Stainless steel resists tarnish and corrosion because of the passivating effect of chromium oxide (Cr_2O_3) layer on the surface. With the advent of stainless steels in World War I and the refinement of drawing processes to form wires in the late 1930s, gold

archwires gradually lost favor to the smaller cross-sectional areas that stainless steel archwires could provide ***(Kusy, 1997)***. These properties of steel wires can be controlled over a reasonably wide range by varying the amount of cold working and annealing during manufactureing. Steel is softened by annealing and hardened by cold working. ***(Proffit et al, 2007)***.

Types of stainless steel:

1) MARTENSITIC STAINLESS STEELS:

They can be hardened by heat treatment. Strength increases and ductility decreases with increasing hardness. Because of their high strength and hardness, martensitic stainless steels are used for surgical and cutting instruments ***(Shrier, 1979)***. The composition of this type of stainless steel includes chromium (11.5–17%), nickel (0–2.5 %), and carbon (0.15–1.20 %) ***(Craig and Powers, 2002)***.

2) ***FERRITE NON–HARDENABLE STAINLESS STEELS*****:**

They cannot be hardened by heat treatment; it has low strength, good corrosion resistance and low cost. The composition of this type include, chromium (11.5–27%), nickel (0%), and carbon (0-2%). This type of alloy finds little application in dentistry. At room temperature it has a body-centered cubic structure ***(Craig and Powers, 2002)***.

3) AUSTENITIC STAINLESS STEELS:

Are essentially non magnetic and cannot be hardened by heat treatment, like the ferritic steels. They are hardenable by cold working only. The composition of this alloy basically include, chromium (16 – 26%), nickel (7 –22%), carbon (0.25% max), and iron (72%). At temperature between 912 and 1394 C°, the stable form of the alloy is face centered cubic, when austenite is cooled slowly from high temperature, the excess carbon that is not soluble in ferrite forms iron carbide (Fe_3C), which is hard and brittle phase and adds strength to the relatively soft and ductile ferritic and austenitic forms of iron based alloy; however, this transformation requires diffusion and defined period of time ***(Craig and Powers, 2002)***.

1.1.1.2.3.3 COBALT-CHROMIUM ALLOYS

These alloys are originally developed for use as watch spring, but their properties are also excellent for orthodontic purposes. It is composed of 40% cobalt, 20% chrome, 15% nickel, 15% iron with small quantity of molybdenum, manganese, beryllium and carbon, their resistance to tarnish and corrosion in the mouth is excellent, and they can be soldered and welded like the stainless wires ***(Anusavice, 1996)***.

Elgiloy, the cobalt chromium alloy, has the advantage that it can be supplied in a softer & therefore more formable state, and then can be hardened by heat treatment after being shaped. The heat treatment increases strength significantly. After heat treatment, the softest elgiloy becomes equivalent to regular stainless steel, while harder initial grades are equivalent to the "super" steels ***(Proffit et al, 2007)***.

1.1.1.2.3.4 TITANIUM ALLOYS

Titanium alloys in orthodontic archwires were introduced in the early 1970s ***(Kocadereli et al, 2000; Aderico et al, 2005)***. At first glance, titanium alloys archwires would appear to be preferable to the stainless steel archwires, mainly because nickel titanium archwires have superior memory and spring back (superelasticity) properties and express relatively low force level and wide range of activation, so it can be used in different orthodontic treatment stages to correct numerous clinical conditions (***Sang and Quick, 1993; Schiff et al, 2004)***. It is the aim of all clinicians to accomplish biological tooth movement which implies the use of low, continuous force and requires archwire with low stiffness, with a plateau during loading and unloading in load deflection curve ***(Iijima et al, 2002; Garrec and Jordan, 2004)***.

They are used in dentistry in cast and commercially pure wrought form. Titanium is used for dental implants, surface coatings, and more recently, for crowns, partial and complete dentures, and orthodontic wires. Wrought alloys of titanium and nickel and of titanium and molybdenum are used for orthodontic wires; the term

titanium is often used to include all types of pure and alloyed titanium ***(Craig and Power, 2002; Chaturvedi, 2009)***.

The following titanium alloys are used in orthodontics:

A. *NICKEL-TITANIUM*

Nickel titanium wires are commonly used during the aligning phase of orthodontic treatment, because they possessed a much lower elastic modules and higher elastic range than stainless steel wires, there by better able to provide the light, continuous force desired for orthodontic treatment (***Hazel et al, 1984; Jia et al, 1999; Pun and Berzins, 2008)***.

Nickel has been used since the 1920s in different forms and for different applications and nickel-titanium (Ni-Ti) wires have become an integral part of orthodontic treatment in the past 30 years. Martensitic Nickel Titanium is also offered in round, square, and rectangular cross sections. The martensitic characteristic that are referred to as the 'soft' state of the Nickel Titanium material, offering a soft easy manipulating wire at room temperature and a fully resilient wire after insertion into the mouth. The austenitic finish temperature for these wires is at approximately 30C°; this will ensure the wire is fully transformed at body temperature and offer superior treatment of severe malocclusion ***(Birnie and Harradine, 2005; Wichelhaus et al, 2005; Zinelis et al, 2007)***.

B. *NICKEL TITANIUM COPPER CHROMIUM ALLOYS (CuNiTi)*

The addition of copper to nickel titanium alloys increases strength, reduces hysteresis (the energy lost between activation and deactivation) and allows greater precision in the setting of the austenitic transformation temperature. The addition of copper, however, increases the transformation temperature to above that of the oral cavity and to compensate for this, 0.2 to 0.5% chromium is added to reduce the transformation temperature. CuNiTi was originally produced with four different austenitic transformation temperatures covering both pseudoelastic and thermoelastic archwires ***(Birnie, 2005)***.

C .TITANIUM MOLYBDENUM ALLOYS (TMA)

Stabilized beta-titanium alloys (TMA) contain 80% titanium as well as 10% molybdenum, 6% zirconium and 4% tin. TMA is twice as stiff as martensitic stabilized nickel titanium alloys and a third as stiff as stainless steel. These archwires are also useful in the final stages of treatment involving the use of steel and ceramic brackets. TMA archwires are good at delivering the range of their activation ***(Birnie, 2005)***. It is formable and weldable, but they have a tendency to fracture and possess a high coefficient of friction ***(Gurgel et al, 2001)***.

D .TITANIUM NIOBIUM FINISHING ARCHES (TiNb/Fa)

Titanium niobium archwires have become available relatively. These wires have 60% of the stiffness of TMA also, even though their resilience is similar to that of stainless steel, Ti-Nb wires have only one quarter of the stiffness of stainless steel. Ti-Nb wires are very easy to bend. They are recommended by the manufacturer for use as finishing archwires ***(Birnie, 2005; Graber et al, 2005)***.

1.1.1.2.3.5 COMPOSITE PLASTICS

The wires can be composed from ceramic fibers that are embedded in a linear or cross-linked polymeric matrix. It is interesting that one non-metallic “wire” already has been offered for clinical use. Optiflex (Ormco/Sybron) is a composite structure formed by top coating optical glass fibers (which are pure silicon dioxide) with a hot melt adhesive and a nylon skin. Its advantages are light forces for initial alignment and excellent esthetics ***(Kusy, 1997)***. Fiber-reinforced thermoplastic wires have also been studied. Candidate fibers include fiberglass and aramid. Candidate resins include polycarbonate and polyethylene terephthalate glycol ***(Craig and Marcus, 1996; Kusy, 1997)***.

1.1.1.3 Auxiliaries

There are different accessories that are used in conjunction with base archwire to produce tooth movement; the accessories are elastics, whips, uprightening spring and coil spring ***(Isaacson, 1984)***.

A. Elastics

They are available in several forms for orthodontic use, and they are available in different sizes and coloures. Latex elastic loops are used for applying inter-or intra maxillary traction. Commercially produced elastic loops, chains and thread are manufactured from a synthetic elastic material. Small single loops (bracket elastics) are available for maintaining the engagement of an archwire in the brackets. The chain elastics may be used for mesiodistal tooth movement along the archwire and rotation of a tooth ***(Isaacson, 1984)***, Fig (1–11).

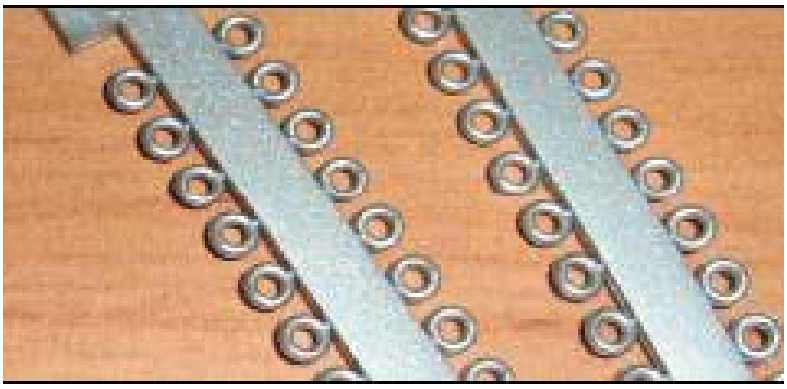

Fig (1–11) Elastic ligature*(Al- Khafaji, 2006)*

B. Whips and Uprightening Springs

These are accessory springs which are used to align individual teeth; the uprightening springs produce mesiodistal tipping, and whip springs, or rotation springs, produce rotation of a tooth about its long axis ***(Isaacson, 1984)***.

C. Coil Springs

Coil springs have provided an alternative to the use of the elastic product for orthodontic tooth movement ***(Nanda, 1997)***. Open coil spring can be used for mesial or distal movement of teeth by compressing the coil against the bracket of the tooth to be moved ***(Isaacson, 1984)***. Closed coil springs are used for space closure, individual tooth retraction or protraction, distal movement of teeth, and traction on impacted teeth, while the opening coil springs are mainly used for opening spaces to unravel the teeth or for the distalization of the molars ***(Manhartsberger and Seidenbusch, 1996; Samuel and Peak, 1998; Maganzini et al, 2010)***.

1.2 Corrosion

1.2.1 Definition

Corrosion is a chemical or electrochemical oxidation process, in which the metal transfers electrons to the environment and undergoes a valence change from zero to a positive value ***(Nestor, 2004)***.

They imply two or more electrode reactions: the oxidation of a metal (anodic partial reaction) and the reduction of an oxidizing agent (cathodic partial reaction) ***(Marcus, 2002)***. These reactions are totally independent but interrelated through electric neutrality of the metal-electrolyte solution system ***(Goldade et al, 2005)***. The most notable effects of corrosion are the loss of metal weight and decreasing the strength of metallic appliance ***(Matasa, 1995; Kerosuo et al, 1997)***.

The environment may be a liquid, gas or hybrid soil-liquid. These environments are called electrolytes since they have their own conductivity for electron transfer ***(Nestor, 2004)***.

An electrolyte is analogous to a conductive solution, which contains positively and negatively charged ions called cations and anions, respectively, an ion is an atom that has lost or gained one or more outer electron(s) and carries an electrical charge. Thus, the corrosion process which can be chemical in nature or electrochemical due to a current flow requires at least two reactions that must occur in a particular corrosive environment. These reactions are classified as anodic and cathodic reactions. For example: when a metal *M* immersed in sulfuric acid solution, a metal oxidation occurs through an anodic reaction and reduction is through a cathodic reaction as shown in the following equations ***(Nestor, 2004)*** :

$M \longrightarrow M^{+Z} + ze^{-}$ (Anodic = Oxidation)

$zH^{+} + zSO_4 + ze^{-} \longrightarrow 2/2\ H_2SO_4$ (Cathodic = Reduction)

$M + zH + zSO_4^{-} \longrightarrow M^{+Z} + 2/2\ H_2SO_4$ (Overall = Rerdox)

Where M= Metal M^{+Z} = Metal cation H^{+} = Hydrogen cation

SO_4^{-} = Sulfate anion Z = Valence or oxidation state

1.2.2 Classification of Corrosion

Generally, corrosion classified into two types:

1.2.2.1 Chemical Corrosion (Dry corrosion)

In which, there is direct combination of metallic and non – metallic elements, this type is exemplified by oxidation , halogenation, or sulfurization reactions, such type called dry corrosion, because it occurs in the absence of water and other fluid electrolytes ***(Fontana and Greene, 1982; Chaturvedi, 2009)***.

1.2.2.2 Electrochemical Corrosion (Wet corrosion)

It requires the presence of water or other fluid electrolyte, it also requires a pathway for transport of electrons (an electrical current), this type also referred as a wet corrosion, because the oral cavity is a wet environment. This type is the principle type of corrosion occurs in the mouth ***(Anusavice, 1996; Craig and Powers, 2002; Chaturvedi, 2009)***.

1.2.3 Electrode Potential

It indicates the metal tendency to produce ions in an electrolyte. Within the electrolyte, the current flows from the metal with a more negative potential to the one with a more positive potential. The first metal (anode) dissolves, where as the second, more cathodic metal, remains intact ***(Anderson, 1972)***.

At the anode, films which tend to decrease the ease of emergence of metal cations may form and may slow down the corrosion rate and this is known as anodic polarization. Similarly, if incoming electrons from the anode can’t be released easily and tend to accumulate at the cathode, the current density in the cell is reduced and hence corrosion of the anode is reduced. In fact, it can be seen that a corrosion process can be controlled by controlling the situation at either the anode or the cathode ***(Miller, 1981)***.

1.2.4 Forms of Corrosion

Corrosion can be divided based on the appearance of the corrosion damage or the mechanism of attack into the following categories ***(Jones, 1996; Davis et al, 2001)*** (Fig. 1-12***)***:

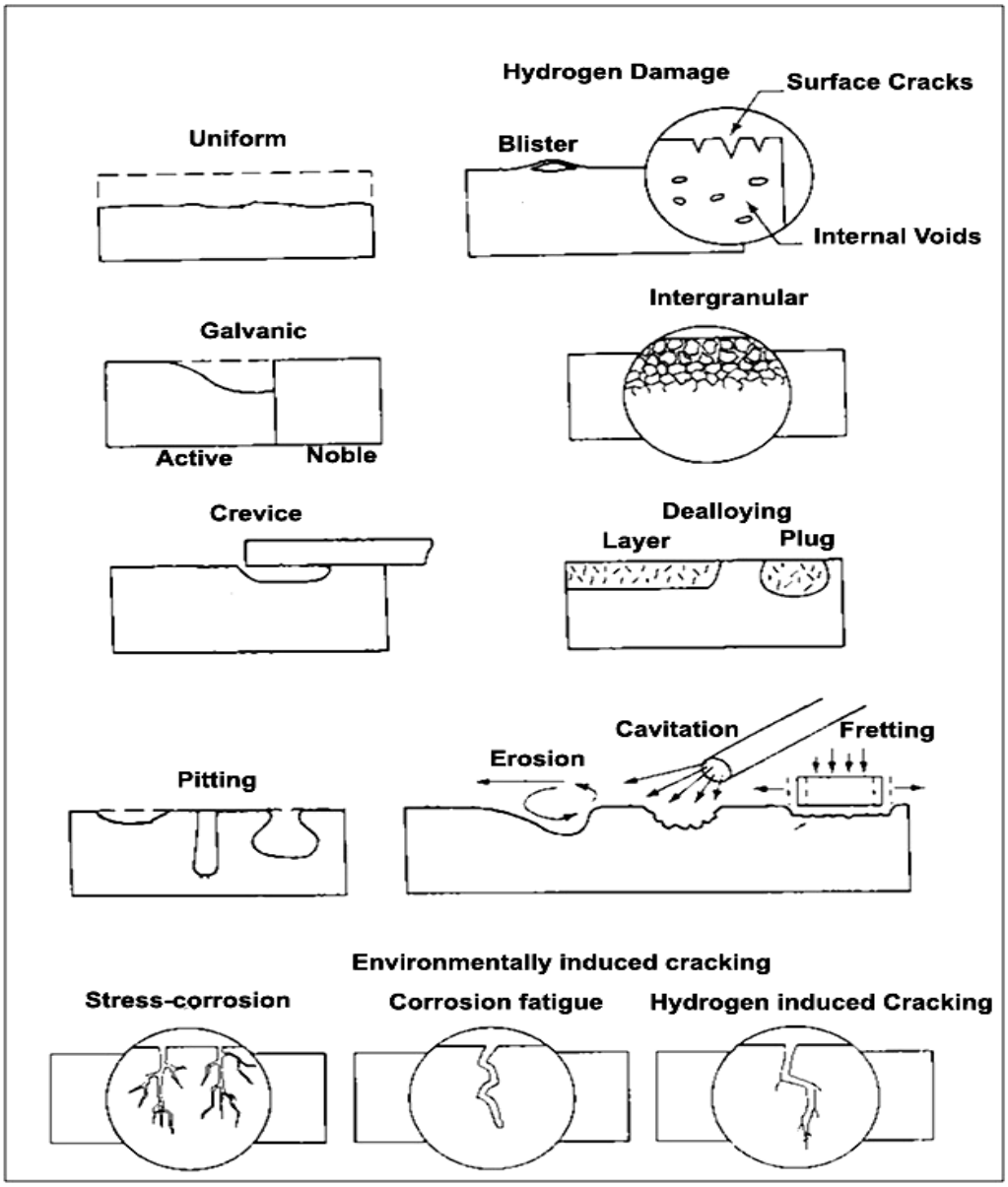

Fig (1-12) Schematic representations of corrosion forms *(Jones, 1996)*

1.2.4.1 Uniform Corrosion

Uniform attack is the most common type of corrosion, occurring with all metals at different rates. The process arises from the interaction of metals with the environment and the subsequent formation of hydroxides or organometallic

compounds. Uniform attack may not be detectable before large amounts of metal are dissolved ***(Eliades and Athanasiou, 2002)***.

For uniform corrosion, the corrosive environment must have the same access to all parts of the metal surface, and the metal itself must be metallurgically and compositionally uniform. Atmospheric corrosion is probably the most prevalent example of uniform corrosion at a visually apparent rate. The other frequently cited example is uniform corrosion of steel in an acid solution. A properly specified alloy must corrode uniformly at a low rate in service ***(Jones, 1996)***.

1.2.4.2 Galvanic Corrosion

It is either a chemical or an electrochemical corrosion. The latter is due to a potential difference between two different metals connected through a circuit for current flow to occur from more active metal (more negative potential) to the more noble metal which is more positive potential (***Nestor, 2004)***.

When different metals such as in orthodontic brackets and archwire or even the same alloy, subjected to different treatments are joined, a combined process of oxidation and dissolution takes place. The less noble metal is oxidized and becomes anodic and, as some of its atoms release electrons, the resulting ions dissolve and become soluble ions ***(Merritt and Brown, 1995; Eliades and Athanasiou, 2002; Iijima et al, 2006*)**. Any alloy will be preferentially corroded when coupled to another alloy with a more positive or noble potential in the galvanic series. At the same time, the more noble alloy is protected from corrosion. The negative, preferentially corroded alloy in the couple (anodic) is said to be active in the galvanic series ***(Jones, 1996; Lemons and Dietsh-Misch, 1999)***.

Stainless steel is characterized by a passive-active behavior depending on the environmental conditions in which the protective chromium oxide layer may be eliminated (active form) or regenerated (passive form). Thus, galvanic corrosion may take place depending on the status of the stainless steel. Nonetheless, this type of corrosion is more common in the broader dental applications of materials ***(Lemons and Dietsh-Misch, 1999; Eliades and Athanasiou, 2002)***.

1.2.4.3 Crevice Corrosion

It is a form of localized attack that occurs at narrow openings or spaces (gaps) between metal-to-metal or nonmetal-to-metal components ***(Davis et al, 2001)***. Crevice corrosion is intense localized corrosion frequently occurs within crevices and other shielded areas on metal surfaces exposed to corrosives ***(Fontana and Greene, 1982)***. The electrolyte solution contacting open areas of a metal structure gains limited mass exchange with the solution trapped in the voids and clearances. The main reason for crevice corrosion in atmospheric conditions is moisture condensation on the surface. In liquid media there is a hampered access of O_2 and removal of corrosion products from the gaps, where the latter leads to violation of metal passivity ***(Jones, 1996)***. The process arises from differences in metal ion or oxygen concentration between the crevice and its surrounding area ***(Eliades and Athanasiou, 2002)***.

This form of corrosion usually exists when there are variations in the electrolyte or in the composition of the given electrolyte within the system. For example, there are often accumulations of food debris in the interproximal area of the mouth and this debris then produces one type of electrolyte that differs than that at the occlusal surface, therefore electrochemical corrosion can occur with preferential attack of the metal surface occurring underneath the layer of food debris ***(Anusavice, 1996)***.

Deposit corrosion and gasket corrosion are terms sometimes used when a nonmetallic material forms a crevice on the metal surface. If the crevice is made up of differing alloys or if the deposit is conductive (e.g., magnetite or graphite), crevice corrosion may be compounded by galvanic effects. Corrosion within a crevice may be caused in atmospheric exposures by retention of water, while the outer surfaces can drain and dry ***(Jones, 1996)***.

Orthodontically, this form of corrosion occurs when the attachment is in contact with plastic materials, an adhesive, an acrylic prosthesis, or elastic ligature ***(Matasa, 1995)***. In clinically derived material, the depth of the crevice can reach 2–5

mm, perforating the base in one piece brackets, and the amount of metal dissolved can reach high levels. The attack may be attributed to the lack of oxygen associated with plaque formation and the byproducts of microbial flora, which reduce the oxygen disturbing the regeneration of the passive layer of chromium oxides ***(Eliades and Athanasiou, 2002)***. Discoloration on the underlying tooth surface during orthodontic treatment has been regarded as the consequence of crevice corrosion of the bracket bases ***(Gwinnett, 1982; Maijer and Smith, 1982; Hodges et al, 2000)***, Fig (1-13).

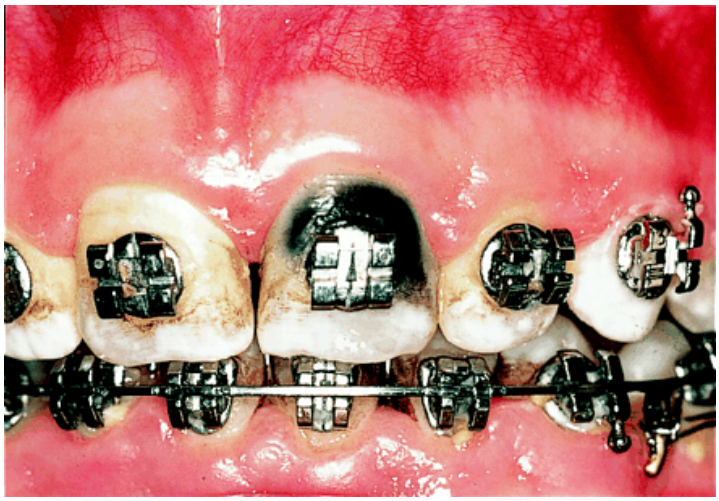

Fig (1-13) Green-black staining evident around bracket base *(Hodges et al, 2000)*

1.2.4.4 Pitting Corrosion

This form of corrosion is extremely localized and it manifests itself as holes on a metal surface. The initial formation of pits is difficult to detect due to the small size, the pits may be deep, shallow or undercut ***(Jones, 1996; Nestor, 2004)***.

Pits are sometimes isolated or so close together that they look like a rough surface. It is one of the most destructive forms of corrosion. Pits begin by breakdown of passivity at the favored nuclei on the metal surface. The breakdown is followed by the formation of electrolytic cell. The anode of which is a minute area of active metal and the cathode of which is a considerable area of passive metal ***(Fontana and Greene, 1982; Juraga et al, 2007)***. From a practical stand point, chloride and chlorine containing solutions cause most pitting failure. The locally high concentration of chloride and hydrogen ion may be swept away by stray convection

currents in the solution since protective pit cavity does not exist ***(Uhlig, 1980; Anusavice, 1996)***.

The stainless steels and nickel alloys with chromium depend on a passive film for corrosion resistance and are especially; susceptible to pitting by local breakdown of the film at isolated sites. The pit is a self-serving crevice that restricts transport between the bulk solution and the pit anode ***(Jones, 1996)***.

It is the most common form of corrosion in orthodontic attachments, effects the mechanical properties or aspect much more than could be inferred from the weight loss. It happens when the attachment is made of several parts, improperly treated, or contains impurities ***(Matasa, 1995)***.

Interestingly, initiation of the process may take place before intraoral placement since excessively porous surfaces have been found on as-received products, so these pores may give rise to attack since they represent sites susceptible to corrosion ***(Eliades and Athanasiou, 2002)***.

1.2.4.5 Hydrogen Damage

Hydrogen damage is a general term which refers to mechanical damage of a metal caused by the presence of, or interaction with, hydrogen. Hydrogen damage is classified into four distinct types ***(Fontana and Greene, 1982)***:

1. Hydrogen attack.
2. Decarburization.
3. Hydrogen blistering.
4. Hydrogen embrittlement.

Hydrogen induced cracking (HIC) and the impairment of ductility by relatively low levels of hydrogen are reversible to some extent if the hydrogen is permitted to escape by baking at elevated temperature. Other effects at higher hydrogen levels are usually irreversible. Hydrogen attack is the reaction of hydrogen with carbides in steel to form methane, resulting in decarburization, voids, and surface blisters. Hydrogen blisters or smaller hydrogen cracks become evident when internal hydrogen-filled voids erupt at the surface. Voids are formed when atomic

hydrogen migrates from the surface to internal defects and inclusions, where molecular hydrogen gas can nucleate, generating sufficient internal pressure to deform and rupture the metal locally ***(Jones, 1996; Chaturvedi, 2009*)**.

1.2.4.6 Inter-granular Corrosion

It is defined as the selective dissolution of grain boundaries, or closely adjacent regions, without appreciable attack of the grains themselves ***(Davis et al, 2001)***. Reactive impurities may segregate, or passivating elements such as chromium may be depleted at the grain boundaries. As a result, the grain boundary or adjacent regions are often less corrosion resistant and preferential corrosion at the grain boundary may be severe enough to drop grains out of the surface ***(Jones, 1996)***.

There are many reasons for such type of attack, as the grain boundaries represent the region of transition between the differently oriented crystals and their structure is normally more non–crystalline particularly towards the central region of grain boundary. This region therefore must be considered to be of higher energy than the interior of the grain, additionally impurities which when present in the alloy enhance corrosion in the metal may be found in greater concentration at the grain boundaries than in the grain proper. Also, the region is more readily attacked by chemicals ***(Huston, 1984)***. In alloys, usually solid solution; of the alloy that has homogenized structure are susceptible to corrosion because the difference in structure between the grains and their boundaries. For the reasons discussed above, the grain boundaries may act as the anode, and the interior of the grains as the cathode ***(Uhlig, 1980)***.

There are many factors that affect the severity of this corrosion including carbon content, time and temperature , excessive temperature with or without prolonged time can increase the inter– granular corrosion ***(Shrier, 1979)***.

The metal weight remains approximately constant, but the loss of mechanical properties (sensitization) can result in poor performance or even collapse. This insidious type of attack can go so far as to reduce the metal to grains ***(Matasa, 1995)***.

1.2.4.7 Selective Leaching or De-alloying

Is the removal of one element from a solid alloy by corrosion process. The most common example is the selective removal of zinc in brass (dezincification); it is uncommon in dental alloys ***(Craig and Powers, 2002)***.

Selective leaching and parting are alternative terms used occasionally for the same phenomenon. Zinc is strongly active to copper and readily leaches out of brass, leaving behind relatively pure porous copper with poor mechanical properties. Dezincification often occurs under deposits and in other non-visible locations, and failures may be sudden and unpredictable. Copper is the most noble of the major constructional metals, and selective leaching of other more active alloying elements, such as nickel, silicon, and aluminum has also been reported ***(Jones, 1996)***.

1.2.4.8 Erosion Corrosion

It is the acceleration or increase in rate of deterioration or attack on a metal because of relative movement between a corrosive fluid and the metal surface. Generally this movement is quite rapid, and mechanical wear effects or abrasion are involved ***(Fontana and Greene, 1982)***.

The combination of a corrosive fluid and high flow velocity results in erosion-corrosion. The same stagnant or slow-flowing fluid will cause a low or modest corrosion rate, but rapid movement of the corrosive fluid physically erodes and removes the protective corrosion product film, exposes the reactive alloy beneath, and accelerates corrosion. Sand or suspended slurries enhance erosion and accelerate erosion-corrosion attack. Low-strength alloys that depend on a surface corrosion product layer for corrosion resistance are most susceptible. The attack generally follows the directions of localized flow and turbulence around surface irregularities ***(Jones, 1996)***.

Erosion corrosion can be classified into ***(Davis et al, 2001)***:

A. Cavitation

Cavitation is a special case of erosion-corrosion. It occurs where velocity is so high that pressure reductions in the flow are sufficient to nucleate water vapor

bubbles, which then implode (collapse) on the surface ***(Fontana and Greene, 1982; Jones, 1996)***.

The appearance of cavitation is similar to pitting except that surfaces in the pits are usually much rougher. The affected region is free of deposits and accumulated corrosion products if cavitation has been recent ***(Davis et al, 2001)***.

B. Fretting Corrosion

Fretting corrosion describes corrosion occurring at contact areas between materials under load subjected to vibration and slip. The basic requirements for the occurrence of fretting corrosion are ***(Fontana and Greene, 1982)***:

- The interface must be under load.
- Vibration or repeated relative motion between the two surfaces must occur.
- The load and the relative motion at the interface must be sufficient to produce slip or deformation on the surface.

The erosion is provided by repeated small movement, often from vibration, between the corroding metal and another contacting solid under load. The motion abrades surface oxide films of the metal surface, again exposing the reactive metal to increased oxide formation. The effect is compounded by the oxide debris, which acts as an additional abrasive between the contacting surfaces ***(Jones, 1996)***. It finds its analogue in the bracket's slot-archwire interface; the individual components of orthodontic appliances when are used in the oral cavity, they are mechanically activated to enable movement of teeth. This is a dynamic situation, so the relative movements of wires and friction in the brackets may lead to fritting corrosion ***(Grimsdottir et al, 1992; Eliades and Athanasiou, 2002)***.

1.2.4.9 Environmentally Induced Cracking

Brittle fracture of a normally ductile alloy in the presence of an environment that causes minimal uniform corrosion is defined as environmentally induced cracking (EIC). Three related but distinct types of failure are included in EIC: stress corrosion cracking (SCC), corrosion fatigue cracking (CFC), and hydrogen-induced

cracking (HIC). Alternative terms for HIC are hydrogen embrittlement, hydrogen-assisted cracking, and hydrogen stress cracking ***(Jones, 1996)***.

A. Stress Corrosion

This type of corrosion may arise between two different locations on the same material because of imbalance in electrode potential between these different surface locations ***(Nikolai, 1985)***. Any cold working or bending (stress) in some parts of the structure, a couple is composed of the stressed metal, saliva and the unstressed metal is thus formed. The stressed area is then more anodic and readily dissolved by the electrolyte in comparison to the passively straight portion of the wire ***(Nikolai, 1985; Anusavice, 1996)***.

Oxygen and the presence of fluid are the most predisposing factor for initiation of corrosion, while the electrochemical factor is responsible for progression and hence the severity of corrosion ***(Corso et al, 1985)***. Stress corrosion involves lattice distortions at the point of stress in metals, producing an area of preferential electrochemical attack that may lead to breakage and corrosion ***(Kirchhoff et al, 1987)***.

When archwires are engaged to brackets bonded to crowded teeth, the reactivity status of the alloy increases. The increased reactivity results from the generation of tensile and compressive stresses developed locally because of the multiaxial, three-dimensional loading of the wire. Thus, an electrochemical potential difference occurs with specific sites acting as anodes and other surfaces acting as cathodes ***(Eliades and Athanasiou, 2002)***.

B. Corrosion Fatigue

A highly important process for the aging of orthodontic alloys is the tendency of a metal to fracture under repeated cyclic stressing. This process (fatigue) is accelerated by the reduction in fatigue resistance induced by exposure to a corrosive medium such as saliva (corrosion fatigue). The process occurs frequently in wires left in the intraoral environment for extended periods of time under load, and in

general, it is characterized by the smoothness of the fractured areas, which also include a site of increased roughness and crystalline appearance.

Also, fracture incident of headgear face bow wires, especially in the inner arch located at the wire segment entering the buccal tube, may be due to corrosion fatigue. Unfortunately, fatigue tests are only scarcely present in the relevant orthodontic materials literature ***(Eliades and Athanasiou, 2002)***.

C. Hydrogen Induced Cracking

It is caused by hydrogen diffusing into the alloy lattice. When the hydrogen evolution reaction occurs:

$$2H^{+1} + 2e^{-1} \longrightarrow H_2 \quad (4)$$

Although stress corrosion cracks often show more branching, cracks have a very similar appearance otherwise, however, hydrogen induced cracking is accelerated by cathodic polarization, whereas stress corrosion cracking and corrosion fatigue cracking are suppressed ***(Jones, 1996)***.

1.2.5 Factors Influence Corrosion

A. *Temperature Effect:* Increasing the temperature of a corrosive system will normally have the effect of increasing the corrosion rate. The kinetic rate of motion or reaction is said to be increased. Even in water solution at room temperature, so the part of a piece of material, which has a higher temperature than another part, may be anodic to the other ***(Uhlig, 1980)***.

B. *Potential Difference:* When there is difference in the potential between metals exposed to the same environment, the metal with higher electrode potential is cathodic and the lower potential is anodic, the higher the ratio of cathode/anode surface area, the higher the corrosion rate ***(Evans, 1977)***.

C. *Heat Treatment:* The corrosion behavior of many metals and alloys can be influenced by heat treatment effect, which is related to annealing, excessive heat treatment reduces corrosion resistance ***(Anderson, 1972; Anusavice, 1996; Craig and Powers, 2002)***.

D*. Surface Condition:* Morphology of the surface, surface roughness, cleanliness of the surface, surface film and the presence of foreign matter can exert a very strong influence on the initiation and rate of corrosion ***(Fontana andGreene, 1982)***.

E*. Effect of Erosion*: Erosion itself is not corrosion; however, even mildly abrasive condition may remove a protective film from the surface, thus exposing a fresh metal to corrodes and there by accelerating damage ***(Uhlig, 1980)***.

F*. Radiation:* Few known tests reveal only a slight additional increase in corrosion when there is exposure to ionized particles ***(Huston, 1984)***.

G*. Effect of Stress*: Under the proper corrosive condition, a material will show a somewhat higher overall corrosion rate when exposed to tensile stress; however, the major concern is propagated cracking of a metal or plastic under the combined effect of tensile stress and corrosion to produce a brittle failure of the material ***(Huston, 1984)***.

H*. Concentration Difference:* When there are differences in concentration or pH of corrosives in a liquid in contact with a metal surface, a corrosion cell usually will develop between the zones exposed to the differing solutions ***(Huston, 1984)***.

I*. Differential Aeration:* A surface, one part of which is exposed to an aerated liquid, while another part is exposed to liquid with less aeration, will corrode if there is an electrical path through the liquid ***(Fontana and Greene, 1982)***.

J*. Biological Effect:* Macro and microscopic organisms influence corrosion by creating mats or obstructions on the surface which produce differential aeration cells, or by absorbing hydrogen from the surface of steel and thus removing the hydrogen as a resistance factor in the corrosion cell ***(Miller, 1981)***.

1.2.6 Corrosion of Biomaterial in Dentistry

The oral cavity is inherently corrosive because the oral fluids are strong potential reactants toward oxidation of metals. The warm and moist condition in the mouth and its microbiologic and enzymatic properties offers an ideal environment for the biodegradation of metals. Saliva is known to contain salts and acids which are often liberated during mastication. Ingested food and drinks vary widely in their

levels of acidity or alkalinity, so some corrosion is almost inevitable in the oral environment ***(Bell et al, 1979; Kerosuo et al, 1995; Faccioni et al, 2003; Fors and Persson, 2006; Amini et al, 2008)***.

In the oral environment, biodegradation of metals occurs, usually by electrochemical breakdown and the association of different metals in the oral environment, where saliva as well as soft and hard tissue has the role of electrolyte, so it may produce electro-galvanic currents that produce a discharge of ions and metallic compounds when combined with the chemically corroded metal. Masticatory forces may also produce a discharge of these ions, as a result of wearing restorations ***(Maijer and Smith, 1982; Janson et al, 1998; Guberina et al, 2002)***.

As orthodontic appliances hamper oral hygiene, dental biofilm accumulates with greater facility on tooth surfaces as well as on the appliance itself in most patients. When this biofilm is thicker and has been present for a longer amount of time, there is an increase in the anaerobic status, thereby further favoring the corrosion of the metals in the appliance ***(Pazzini et al, 2009)***.

Bacteria and their waste products, selective interaction with gases such as oxygen and carbon dioxide, amount of protein in the saliva, physical and chemical properties of food and liquids, may all contribute to the breakdown of dental materials placed in the mouth; pH of the environment has significant effect on the rate of corrosion plaque may or may not help this breakdown ***(Maijer and Smith, 1986; Sfondrini et al, 2009)***.

In the past years, there were increased evidences concerning the degradation of stainless steel and titanium alloys. These alloys are very susceptible to galvanic pitting, intergranular, crevice and erosion corrosion ***(Shrier, 1979; Anusavice, 1996; Craig and Powers, 2002)***. Crevice corrosion has been implicated as the mechanism involved in the corrosion of orthodontic bracket. Discoloration on the underlying tooth surface during orthodontic treatment has been regarded as the consequence of crevice corrosion of the bracket bases ***(Park and Shearer, 1983; Kerosuo et al, 1997)***.

Mechanical distortion and excessive cold working promote corrosion by making the distorted portion of the wire or the band more anodic, the alloy then behave electrochemically as if two alloys were present, the presence of dissimilar metals or alloys such as silver solder, amalgams, or gold, may lead to galvanic corrosion ***(Craig and Powers, 2002)***.

1.2.6.1 Corrosion Resistance of Stainless Steel

Stainless steel owes its corrosion resistance property to chromium, a highly reactive base metal. The alloy's corrosion resistance depends on its passive film, the thickness of this passive layer is in range 1 to 10 nm; being very thin layer can be easily damaged, which spontaneously forms (passivation) and reforms (repassivation) in air and under most tissue fluid conditions. Oxygen is necessary to form and maintain the film, whereas acidity and chloride ions can be particularly detrimental to it ***(Eliades and Athanasiou, 2002; Juraga et al, 2007)***. The filmed formed by chromium complex also contains iron, nickel and molybdenum. In an aqueous environment, this film consists of an inner oxide layer and an outer hydroxide layer. Important factor in stainless steel is that the protective oxide is self healing, so that this material can be worked without losing its oxidation resistance ***(Scully, 1978; Eliades and Athanasiou, 2002)***.

The amount of carbon must also be tightly controlled; otherwise the carbon will react with chromium forming chromium carbides at the grain boundaries and decrease the corrosion resistance. This is known as sensitization. The presence of molybdenum increases the resistance to pitting corrosion ***(Craig and O'brien, 1995; Eliades and Athanasiou, 2002)***. Additionally, the surface should be clean, smooth and polished; irregularities appear to promote electrochemical action of the surface of the alloy ***(Shrier, 1979; Craig and Powers, 2002)***.

The 18 –8 stainless steel might lose its resistance to corrosion, if it is heated between (400 – 900) degree centigrade, the reason for decrease in corrosion resistance is the precipitation of chromium carbides at the grain boundaries at high temperature. When chromium combines with the carbon in this manner, its

passivating qualities are lost and as a consequence, the corrosion resistance of the stainless steel is reduced because the portion of the grain adjacent to the grain boundary is generally depleted from chromium to produce chromium carbide (Cr_3C), as a result, an intergranular corrosion occurs ***(Ceen and Gwinnett, 1980)***.

If the stainless steel is severely cold worked the carbides precipitate along slip planes. As a result, the distribution of areas deficient in chromium is less localized, or in other words, the carbides are more uniformly distributed so that the resistance to corrosion is greater than when only the grain boundaries are involved ***(Uhlig, 1980; Corso et al, 1985)***. Some elements are present in small amount tend to prevent the formation of carbides between the carbon presents in the alloy and the iron or chromium. They are described as stabilizing elements that include titanium, niobium, or tantalium, so that the carbides that do form are titanium carbides rather than chromium carbides ***(Craig and O'brien, 1995)***.

1.2.6.2 Corrosion Resistance of Titanium Alloys

The high corrosion resistance and biocompatibility of titanium and its alloys are attributed to a thin passive film that consists essentially of titanium bioxide (TiO_2), and titanium monoxide (TiO) and it re-passivates in a time on the order of nanoseconds ***(Pan and Theirry, 1997; Wang and Fung, 1997; Craig and Powers, 2002; Huang, 2002)***, however, even in its passive condition, titanium is not inert and titanium ion release that does occur is a result of chemical dissolution of titanium oxide ***(Ishizaw and Ogino, 1995; Craig and Powers, 2002)***.

The addition of nickel to the alloy, in addition of increasing hardness and strength, and also increases the resistance to galvanic action and hence enhances corrosion resistance ***(Chern, 1995; Craig and Powers, 2002)***.

1.2.7 Techniques Employed to Evaluate Corrosion Behavior

A wide variety of techniques are used to evaluate corrosion behavior of dental alloys, these are:

1.2.7.1 Pontentiostatic and Potentiodynamic Polarization Techniques

These are the most famous techniques used in many experiments; these techniques use potentiostatic and / or potentiodynamic polarization methods (anodic and / or cathodic). In most of these experiments, the electrochemical cell consists of a glass flask with orifices oriented for different electrodes, the electrolyte media in these studies are selected according to the demand of the study. Such techniques were used by many investigators in their studies ***(Corso et al, 1985; Sutow and Milne, 1985; Gerstofer and Weber, 1987; Chern et al, 1996; Kim and Johnson, 1999; Poduch et al, 1999; Al- Joboury, 2001; Chotiros et al, 2001; Rondelli and Vincentini, 2002; Yonekura et al, 2004)***.

1.2.7.2 Microscopical Analysis

Including refractory optical microscope used to study the corrosion type and site in many corrosion studies ***(Malhotra and Asgar, 1981; Palaphias, 1985; Taira et al, 1989; Al- Joboury, 2001; Hassoon AA, 2008)***.

Scanning electorn microscope for the surface of the metal is a common technique used in some studies ***(Grimsdottir and Hensten, 1997; Ikuya and Etsuko, 2003; Eliades et al, 2004a; Huang, 2005; Schiff et al, 2005)***.

Stereo–optical microscope was another technique that used in corrosion studies ***(Sutow and Jones, 1979; Wright et al, 1981; Platt et al, 1997)***.

1.2.7.3 Energy Dispersive Detector

Is a chemical microanalysis technique used in conjunction with scanning electron microscopy. This was used in studying the corrosion process in phosphate containing solution ***(Moberg and Johansson, 1991; Larry and Hanke, 2006)***.

1.2.7.4 Laser Specular Reflectance and Profilometry

These techniques are used to detect the surface corrosion and surface roughness in which a fine stylus is used to scan the topography in a single line of a preselected area ***(Bourauel et al, 1998)***.

1.2.7.5 X–Ray Techniques

Which involve photo electron spectroscopies, and fluorescence techniques. X–ray photo electron spectroscopy (XPS) was used in many studies ***(Dickinson and John, 1980; Malhotra and Asgar, 1981; Iijima et al, 2001; Iijima et al, 2002; Shin et al, 2003; Huang, 2005; Michiardi et al, 2006; Hassoon, 2008)***.

Most surface spectroscopies are based on the principle that the surface to be investigated is irradiated by beam of particles like (photons, electrons, or ions). The primary particles induce excitations in the surface, which respond by emitting secondary particles, and information about the surface may be derived, also electron spectroscopy for chemical analysis (ESCA), Auger electron spectroscopy (AES), and secondary ion mass spectroscopy (SIMS) can be used ***(Al-Said, 1994; Huang, 2005)***.

1.2.7.6 Chemical Analysis by Atomic Absorption Spectrophotometeries (AAS):

Atomic absorption spectrophotometry is a technique used for determining the concentration of a particular metal element in a sample, in both trace and major concentration. The technique can be used to analyze the concentration of over 70 different metals in a solution ***(Haswell, 1991; L'vov, 2005)***.

The technique makes use of absorption spectrometry to assess the concentration of an analyte in a sample. The electrons of the atoms in the atomizer can be promoted to higher orbitals for a short amount of time by absorbing a set quantity of energy (i.e. light of a given wavelength). This amount of energy (or wavelength) is specific to a particular electron transition in a particular element, and in general, each wavelength corresponds to only one element ***(Alkemade et al, 1982)***.

The practical advantages of AAS such as speed, simplicity, precision, cost and ease of operation are ideal and necessary for the many situations where large numbers of accurate results are required for diagnosis and management of patient's clinical conditions ***(Haswell, 1991)***.

These involve flame and flameless methods, which are used mainly to detect the presence of the elements in an aqueous solution ***(Barrett et al, 1993; Al- Joboury, 2001; Yonekura et al, 2004; Yokoyama et al, 2005; Hassoon AA, 2008)***.

1.2.7.7 Weighing of the Appliance

Blind method; no information on what is actually released; no information on mechanism; lacks clinical relevance (oral flora, plaque, and calcification processes are not integrated in the model), used for corrosion studies ***(Eliades and Athanasiou, 2002; Hassoon AA, 2008)***.

1.2.8 Protection against Corrosion

1. *A coating of a noble alloy* may be applied to the surface of a second metal, for instance, a base metal; however, the noble metal is soft and when its surface becomes scratched or pitted to such a depth that the base metal is exposed to the environment, the base metal will be corroded at a rapid rate. This occurs for three reasons ***(Anusavice, 1996)***:

a. A surface defect is created that could set up a concentration cell.

b. Two dissimilar metals are in a direct contact, thus producing a galvanic cell.

c. There is an unfavorable anode/cathode surface area ratio. In general, when the anode surface is small (base metal at the bottom of the scratch) and the cathode surface is large (the noble metal coating covering the entire casting), rapid corrosion is expected ***(Anusavice, 1996)***.

2. *Polishing* is significantly reduced the corrosion rate of nickel titanium orthodontic archwires ***(Hunt et al, 1999)***.

3. *Paints or other types of inorganic or organic coatings* are used for protection, any pit or scratch in the protective layer may lead to rapid corrosion of the base metal. In case of dissimilar metals, paint or other non – conductive film can be used to advantage, if it is applied to the more noble of the two metals, the corrosion rate of the more active metal is reduced because the surface area available for the reduction reaction has been decreased and scratch in this type of coating does not lead to rapid attack on the active metal ***(Anusavice, 1996)***. An epoxy coating can be used to decrease the corrosion of nickel titanium archwires and makes them least corrosion potential ***(Kim and Johnson, 1999)***.

4. *Certain metals develop a thin, highly protective film* by reaction with the environment, such a metal is said to be passive. Chromium is a good example of passivating metal. This important metal form a film of corrosion product, which consists of closely packed oxide layer. Another example is titanium, which also form strongly adherent oxide layer. Passivating metals are without drawbacks, tensile stresses and certain ions, such as chloride, can disrupt the protective film, and rapid corrosion may ensue ***(Uhlig, 1980; Huston, 1984; Anusavice, 1996)***.

5. *Oxidation treatment* in a low–oxygen pressure atmosphere leads to high surface titanium / nickel ratio, a very low nickel surface concentration and a thick oxide layer, and this give a great interest for biomedical applications, as it could minimize sensitization and allergies and improve biocompatibility and corrosion resistance of nickel titanium shape memory alloys ***(Michiardi et al, 2006)***.

1.3 Biocompatibility

It can be defined as the compatibility of any foreign material with the living organism. Defining the term closely, biocompatibility refers to the ability of a material to perform its desired function with respect to a medical therapy, without eliciting any undesirable local or systemic effects in the recipient or beneficiary of that therapy, so the interaction between the material and vital tissue is so minimal that the material is not detrimentally affected by the tissue or the tissue by the material, factors influencing biocompatibility include chemical, mechanical, electrical and surface specific properties ***(Schroeder and Kerkeler, 1991; Williams, 2008; Elshahawy et al, 2009b)***.

Dental alloy are in direct contact with oral tissues and because of that they must be completely biocompatible: biologically tolerant (without causing antigen-antibody reaction), biochemically indifferent (remain unchanged in the body without causing any effect on the organism), electrically and magnetically inert (without causing a galvanic current or magnetic field) ***(Rinĉić et al, 2003)***. So among the aspects which affecting the biocompatibility are the amount and forms of released corrosion products, and their deposition in the body after release ***(Bundy, 1994)***.

Biocompatibility is measured using three types of biological tests ***(Brantley and Eliades, 2001)***:

A. *IN VITRO BIOCOMPATIBILITY TEST*: can be performed in test tube, cell-culture dish or otherwise outside of a living organism. This test is controllable experimentally, repeatable, fast, relatively inexpensive, relatively simple and avoid ethical issue, but its result may be misleading as to ultimate biological response to a material.

B. *ANIMAL TEST*: the material is placed into an animal usually a mammal. The biological response is more comprehensive and relevant, but it's difficult to control variables in these tests. Moreover, these tests are time consuming and expensive.

C. *USAGE TEST*: The most relevant biocompatibility test and all other test must be measured against it for relevance; however, it has many complications and problems. They are expensive, time-consuming, difficult to control, difficult to interpret and may be legally and ethically complex.

There is increasing concern with the biocompatibility of dental materials; this might be due to a real increase in the frequency of allergic reactions to the materials or to an increase in awareness of adverse effects from these materials ***(Stenman and Bergman, 1989; Menezes et al, 2007)***. The biocompatibility of dental alloys has been investigated over the past years. However, studies on this issue have given rise to questions without answers, confirming the need to learn more about the biocompatibility of these materials. Since this process has not been fully explained, orthodontists may be confused in the selection of biologically safe appliances for their patients ***(Wataha, 2000; Souza and Menezes, 2008)***.

The release of metal ions from dental alloys is a phenomenon that cannot be avoided; it is difficult to find a material that will be fully stable within an organism and will show no signs of biodegradation. A growing number of recent studies are investigating the problem of biocompatibility with the goals of determining the upper limit of biological tolerance and finding means through which the release of ions will be kept within these limits ***(Kuhta et al, 2009)***.

1.4 Hypersensitivity and Allergic Reaction

1.4.1 Definition

Hypersensitivity is an adverse reaction following contact with sub-toxic doses of foreign substances. Hypersensitivity reactions comprise three different mechanisms ***(Brantley and Eliades, 2001)***:

I. *INTOLERANCE REACTIONS* are mostly related to foodstuffs and inborn errors of metabolism.

II. *HYPER REACTIVITY* responses are often related to volatile irritants.

III. *AN ALLERGY* is an acquired condition resulting in an overreaction upon contact with a foreign substance, and arises from a genetic predisposition and previous sensitization from exposure to the substance. Allergic reaction most often categorize into four groups, type I to IV, of which the immediate reaction (type I) and the delayed reaction (type IV) have relevance to orthodontic biomaterial.

1.4.2 Allergic Reaction to Orthodontic Appliance

In orthodontic practice, a variety of metallic alloys, such as stainless steel, cobalt-chromium, nickel-titanium and beta-titanium are used, and the majority of these contain nickel. The percentage of nickel in the appliances, auxiliaries and utilities used in orthodontics range from 8% (as in stainless steel) to more than 50% (as in the nickel-titanium alloys). Leaching of these metallic components may be a potential trigger to an allergic reaction ***(Leite and Bell, 2004; Kolokitha and Chatzistavrou, 2009)***.

Researchers have focused on the release of metal ions from stainless steel brackets, mainly involving iron, chromium, and nickel, which are the major corrosion products of stainless steel. Although all the elements can have adverse effects, nickel has received the most attention because of its reported potential for hazardous effects **(*Eliades et al, 2004b)***. Nickel is one of the most common causes of allergic contact dermatitis, and the incidence of such contact dermatitis is as high as approximately

20– 30% ***(Romaguera et al, 1989; Bass et al, 1993; Rahilly and price, 2003; Ramadan, 2004; Kao et al, 2007; Kolokitha et al, 2008)***.

Chromium is known to be an essential element for human beings and animals. While nickel is essential for some animals, a similar role in human beings has not been conclusively identified ***(Fishbein, 1984; Bishara et al, 1993; Sfondrini et al, 2009)***. In general, the most significant human exposure to nickel, chromium, and titanium occurs through the diet, atmosphere, drinking water, clothing fasteners, and jewelry. The major dietary sources for these three metals are vegetables, grains, and cereals ***(Grandjean, 1984; Kocadereli et al, 2000; Ağaoğlu et al, 2001; Sfondrini et al, 2009)***. On the other hand, iatrogenic exposures to nickel, chromium, and titanium can occur from joint prostheses, dental implants, orthopedic plates and screw, surgical clips and steel sutures pacemaker leads, prosthetic heart valves, dental alloys and orthodontic appliances ***(Edie et al, 1981)***.

Characteristic lesions of contact stomatitis vary from barely visible, mild erythema to a fiery red color with or without edema. Symptoms may include loss of taste, numbness, burning sensation and soreness of the involved area, often accompanied by angular cheilitis, severely inflamed hyperplastic gingival tissue, labial desquamation, alveolar bone loss, and edema of the throat, palate and gums. In addition to osteomyelitis, itching is not a frequent symptom. Although it is more difficult to provoke contact stomatitis than contact dermatitis, and severe gingivitis associated with orthodontic therapy ***(Park and Shearer, 1983; Lamster et al, 1987; Genelhu et al, 2005; Pantuzo et al, 2007)***. The manifestations of nickel allergy (dermatitis and urticaria) can be found distant from the nickel source, and is one of the reasons why nickel hypersensitivity has been of growing concern among dentists. Adverse reaction to orthodontic material were six times more common with extraoral than with intraoral ones, Fig (1-14). Most extraoral reactions were seen where the metal parts came in contact with the skin ***(Grimsdottir et al, 1992)***. Alteration in blood pressure, pulse, or temperature in

patients with nickel hypersensitivity was also found ***(Menezes et al, 2004)***.

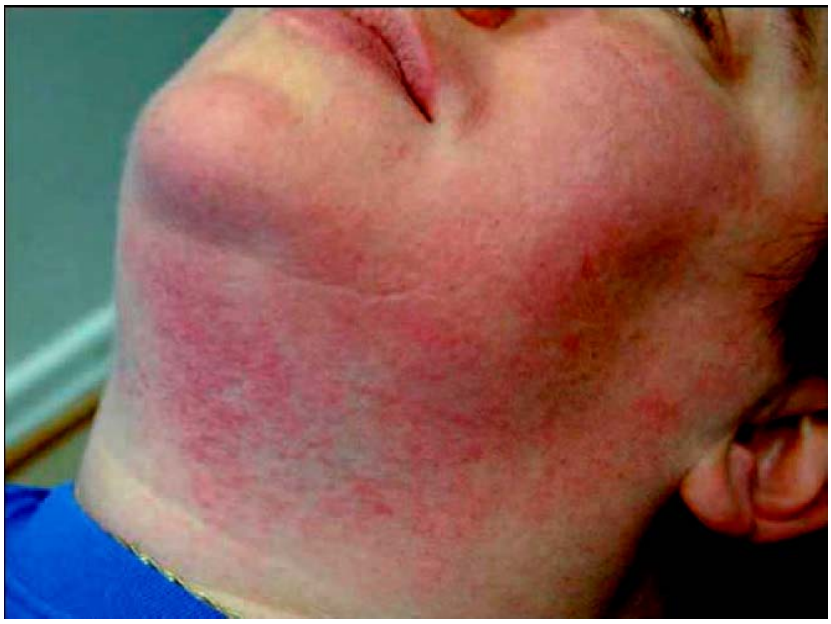

Fig (1-14) Allergic reaction on the skin caused by wearing intraoral orthodontic appliance *(Ehrnrooth and Kerosuo, 2009)*

The hypersensitivity reaction to nickel is due to a direct relationship with the presence of this metal in the environment, and may be caused by ingestion or direct contact with the skin and/or mucosa ***(Blannco et al, 1984; Sağlam, 2004; Kolokitha and Chatzistavrou, 2009)***. Factors that have been documented to influence the development of sensitization include mechanical irritation, skin maceration, increased environmental temperatures, and increased intensity and duration of exposure. Genetic factors also have been reported to play a role in the acquisition and the regulation of the immune response although studies on the mode of inheritance have been inconclusive ***(Raitt et al, 1985; Fisher, 1986)***. The diagnosis of allergy has usually been based on patient history, clinical findings and the result of patch testing. Patch test reactions properly interpreted are acceptable as evidence of sensitivity to a particular allergen ***(Fisher, 2001)***.

It is estimated that 4.5% to 28.5% of population have hypersensitivity to nickel, with a higher prevalence in females. Epidemiological data indicates that the number of nickel sensitive females has increased, especially among younger

age groups. The frequency of nickel hypersensitivity in young women has been reported to be 10 times higher than that reported for young men ***(Nielsen and Menne, 1993; Kerosuo et al, 1996; Kolokitha and Chatzistavrou, 2009)***.

Hypersensitivity to nickel in females is thought to be related to environmental exposure as a result of contact with detergents, jewelry, earring and other metallic objects. In males, the hypersensitivity is usually related to occupational exposure, especially in industries that use nickel as raw material. Environmental exposure may also result from contact with wrist watches, metal buttons, metallic frames of glasses, buckles and other metallic objects **(*Wilkinson and Rycroft, 1992; Guilherme et al, 1998; Sağlam, 2004)***.

1.4.3 Nickel and Chromium Carcinogenicity

The introduction of metal ions into the human body is an additional risk to health since these ions may be released in different places and at different levels, depending on the characteristics and solubility of the products containing them. Consequently, biological functions are affected, which may lead to systemic and local effects. In addition to the allergic issue, carcinogenic, mutagenic and cytotoxic effects have been assigned to nickel and to a lesser extent, chromium ***(Savarino et al, 2002; Kusy, 2004; Souza and Menezes, 2008)***.

The fact that nickel, chromium, and their compounds present a known cancer risk has been well documented in the literature. Nearly all the reported cases of nickel and chromium induced carcinoma have occurred from occupational exposures to inhaled metal compounds. The primary tumor locations are the lung and the nasal mucosa ***(Psaila, 1982; Langard, 1988)***.

Not all nickel and chromium compounds have carcinogenic potential. For nickel compounds, risk is inversely related to its solubility in an aqueous media ***(Leonardo et al, 1981)***. For chromium compounds, carcinogenic risk has only been identified with compounds in which the chromium is in a hexavalent oxidation state. There is no experimental evidence that nickel or chromium compounds are carcinogenic when administered by oral or cutaneous routes

(Bishara et al, 1993). The average latency period from the time of exposure to these metal compounds to the development of cancer has been reported to be between 20 and 25 years ***(Norseth, 1981; Newman, 1986)***.

1.5 U.S. FDA'S Food Classifications

United State-Food and Drug Administration classification system was a universal system in which restrictions often were placed by a Food Additive Regulation or Food Contact Notification (FCN) on the foods with which the substance comes into contact. FDA had developed several different classification schemes for food types. These classification schemes vary from situation to situation, case by case usually were needed when interpreting regulatory clearances with regard to food types. FDA identified four types of food: aqueous, acidic, alcoholic and fatty food. At that time, FDA didn't include dry food because since it is without free fat or oil on the surface, so had traditionally little or no propensity to extract components of food-contact articles. However, the agency had recognized that it would be impractical to impose limits on specific foods and instead released on general groupings of food that possess common characteristic ***(Borodinsky et al , 2005)***.

1.5.1 Types of Food (United States Food and Drug Administration Guideline, 2006):

According to the United States Food and Drug Administration Guideline, 2006 food was classified into:

I. Nonacid, aqueous products; they may contain salt or sugar or both (pH above 5.0) eg. Jelly, canned corn.

II. Acid, aqueous products; they may contain salt or sugar or both, in addition may include oil-in-water emulsions of low- or high-fat content eg. vinegar.

III. Aqueous, acid or nonacid products containing free oil or fat; they may contain salt, in addition may include water-in-oil emulsions of low- or high-fat content eg. Sea foods.

IV. Dairy products and modifications:

A. Water-in-oil emulsions, high- or low-fat eg. Butter.

B. Oil-in-water emulsions, high- or low-fat eg. Milk.

V. Low-moisture fats and oil eg. Lard, vegetable oil.

VI. Beverages:

A. Containing up to 8 % of alcohol (beers) ***(Scottish Statutory Instruments, 2006)***.

B. Nonalcoholic. Water, ciders, fruit or vegetable juices of normal strength or concentrated, musts, fruit nectars, lemonades and mineral waters, syrups, bitters, infusions, coffee, tea, liquid chocolate and other ***(Scottish Statutory Instruments, 2006)***.

C. Containing more than 8 % alcohol. Wines, spirits and liqueurs ***(Scottish Statutory Instruments, 2006)***.

VII. Bakery products other than those included under Types VIII or IX of this table:

A. Moist bakery products with surface containing free fat or oil eg. Meat pies, fruit pies.

B. Moist bakery products with surface containing no free fat or oil eg. bread, cake.

VIII. Dry solids with the surface containing no free fat or oil (no end test required) eg. macaroni, corn meal.

IX. Dry solids with the surface containing free fat or oil eg. Pizza.

1.5.2 Food Simulating Liquids

These are test media simulating foods in their behavior of extracting substances from food contacting materials, each one type of these media simulated certain type of food in its behavior for extraction of substances from food contacting material ***(European Commission Health and Consumer Protection Directorate-General, 2003; Borodinsky et al, 2005)***; Table (1-2).

Table (1-2) Recommended food simulants *(United States Food and Drug Administration Guideline, 2006; Guidance for Industry Preparation of Premarket Notifications for Food Contact Substances, 2007).*

Food type	Recommended food-simulating solvents
Aqueous and acidic foods	**10% Ethanol**
Low and high-alcoholic foods	**10 Or 50% Ethanol**
Fatty foods	**Food oil (eg. Corn oil, sun-flower oil, olive oil)&synthetic oil(HB307 or Miglyol 812^{TM})**

Previous test protocols (prior 1988) recommended the use of water and 3% acetic acid as food simulants for aqueous and acidic foods. However, water and 3% acetic acid had been shown, in a number of instances –to under estimate migration into aqueous foods– therefore, 10% ethanol was now recommended as an aqueous food stimulant ***(Recommendation for chemistry data for indirect food additive petitions, 1995)***. On other hand 3% acetic acid was recommended by FDA as acidic food simulant in the following conditions:

1. When food acidity was expected to lead to significantly higher levels of migration than with10% ethanol.
2. If the polymer or adjuvant was acid sensitive.
3. If trans–esterification occurred in ethanol solution.

Previous protocol (prior to 1988) recommended the use of heptanes as a fatty food simulants. But due to the exaggerated effect of heptanes relative to food oil, it was no longer recommended as a fatty food-stimulant ***(National Institute of Standards and Technology, 2002)***.

1.5.3 European Union Food Classification System (Scottish Statutory Instrument, 2006)

The EU system was one in which a given food-contact substance might be used as cleared or as an additive, might be used in any manner, including in contact

with any food stuff. European Council Directive serves as a form of food type classification system which contains eight categories of food stuff as below:

1. Beverages:
    a) Alcoholic beverages.
    b) Non-alcoholic beverages.
    c) Un-denaturated ethyl alcohol.
2. Cereals, cereal products, pastry, biscuits, cakes and other baker's wares.
3. Chocolate.
4. Fruit, vegetables and products thereof.
5. Fats and oils.
6. Animal products.
7. Milk products.
8. Miscellaneous products like coca, liquid coffee extracts, vinegar.

In addition EU system laying down the list of simulants to be used for testing migration of constituents of plastic materials and articles intended to come into contact with food stuffs ***(Borodinsky et al, 2005)*** table (1-3).

EU system also set forth the simulants to be used for testing migration of components of plastic food- contact articles in special cases table (1-4).

Table (1-3) Food simulants to be used in migration tests (*Scottish Statutory Instrument, 2006)*

Abbreviation	Food-simulants
Simulant A	**Distilled water or water of equivalent quality**
Simulant B	**3% acetic acid(w/v) in aqueous solution**
Simulant C	**10% ethanol (v/v) in aqueous solution save that the concentration should be adjusted to the actual alcoholic strength**
Simulant D	**Rectified olive oil, if for technical reasons connected with the method of analysis, sunflower oil or synthetic triglycerides must be used**

Table (1-4) Simulants to be selected for testing food contact materials in special cases *(Scottish statutory instrument, 2006)*

Contact-food	Food simulants
All aqueous and acidic foods	Simulant B
All alcoholic and aqueous foods	Simulant C
All alcoholic and acidic foods	Simulant C&B
All fatty and aqueous foods	Simulant D&A
All fatty and acidic foods	Simulant D&B
All fatty and alcoholic acid	Simulant D&C
All fatty, alcoholic and acidic foods	Simulant D,C&B

1.6 Chemical Models

Numerous different chemical models were developed in order to evaluate the effect of the oral environment and its ingredients (saliva, bacteria, and microorganisms) on the stability of dental materials. Each one of them was proposed to simulate different factors of the oral environment. Most of those models comprised a solution which was designed:

a) To simulate as close as possible the conditions in the oral cavity or

b) To accelerate the degradation in order to meet a time effective answer whether the tested materials are resistant to degradation in vitro or not.

Some of them intended to simulate the effect of the chemical conditions (different pH levels) found in the oral cavity, some others the impact of thermal fluctuations occurring in the oral environment and some others the solubility action of food elements. It was very often attempted to incorporate all these factors in a solution, which could simulate saliva. Therefore numerous artificial saliva models were developed. A newer more reasonable approach includes solutions, which comprise active ingredients of saliva (such as proteins) ***(Kournetas, 2005)***.

1.6.1 Artificial Saliva

It is a solution, which simulates all chemical, thermal and biochemical elements, and so would be ideal to reproduce the oral conditions and eventually predict certain characteristics of dental materials behavior in vivo. The concept of the so called artificial saliva solution had begun as early as 1931 ***(Leung and Darvell, 1997)***. It is also clear that exact duplication of the properties of human saliva is impossible due to the inconsistent and unstable nature of natural saliva ***(Mandel, 1974)***. This instability also makes natural saliva itself inappropriate for use in standardized in vitro studies. Large assortment of many different artificial saliva compounds is available. Very few of them were found to contain all major ionic components with concentrations in the physiological range. A main problem of many artificial compounds is their too high Ca^{++} content which is not bound by proteins as it happens with human saliva ***(Leung and Darvell, 1997)***.

1.6.2 Ethanol (ethyl alcohol)

Ethanol is a straight-chain alcohol; its molecular formula is C_2H_5OH, and its empirical formula is C_2H_6O ***(Myers et al, 2007)***.

Ethanol or ethyl alcohol is the active ingredient in alcoholic beverages such as beer, wine and whiskey. It is produced by the fermentation of carbohydrates (sugars and starches) in fruits and other plant sources (such as corn, barley, grapes and so on), using the process that was discovered thousands of years ago. During fermentation, microorganism, such as yeast, convert sugar to alcohol and carbon dioxide ***(Zumdahl, 1998; Buel and Girard, 2002)***:

$$C_6H_{12}O_6 \longrightarrow 2CH_3CH_2OH + 2\,CO_2$$

The properties of pure 100% absolute alcohol (dehydrated) are: colorless limpid volatile liquid; boiling point 78°C; freezing point 117.3°C; vinous odor; and pungent taste. It is miscible with water, methyl alcohol, ether, chloroform and acetone ***(Hawley, 1971)***. Ethanol is very soluble in water because it contains a polar O-H bond like those in water which makes it very compatible with water ***(Zumdahl,***

1998). It was sterilized by autoclave in sealed ampoules or by filtration ***(Budavari et al, 1989)***, also had a high latent heat of vaporization and contains oxygen ***(Environment Australia, 2002)***.

Ethanol is sometimes called grain alcohol because it can be produced from grains such as barley, wheat, corn and rice. In addition of being a constituent of alcoholic beverages, ethanol has many industrial uses. It is used in the preparation of many organic chemicals and it is an important industrial solvent. It is also used by the pharmaceutical industry as an ingredient in cough syrups and other medications and an antiseptic for skin disinfection ***(Buel and Girard, 2002)***. Most industrial ethanol was denaturized (to prevent oral consumption) by the addition of small amounts (1-5%) of unpleasant or poisonous substances ***(Zumdahl, 1998; Environment Australia, 2002)***.

1.6.3 Corn Oil (Maize Oil)

It was one of unsaturated food oils (like olive oil), so represent a natural fatty stimulant ***(Scottish statutory instrument, 2006)***. It is a pale yellow liquid with characteristic taste and odor, insoluble in water; soluble in ether, chloroform, amyl acetate, benzene, and carbon disulfide and slightly soluble in alcohol. It is used as foodstuffs, soap, lubricants, leather dressing factice, margarine; salad oil, hair dressings and as solvent ***(Hawley, 1971)***. Corn oil was a product to the essential fatty acids, which were necessary fats that human, could not synthesize and must be obtained through diet like linolenic, linoleic and oleic acids. These acids support the cardiovascular, reproductive, immune and the nervous systems, so their deficiency causes a serious problem. As that, corn oil represents an important dietary material and this requires an adequate supply through dietary intake ***(Dunlap et al, 1995; Budwig, 2006)***.

1.6.4 Acetic Acid (Ethanioc Acid) CH_3COOH

Acetic acid, CH_3COOH, also known as ethanoic acid, is an organic acid which gives vinegar its sour taste and pungent smell. Pure, water-free acetic acid

(glacial acetic acid) is a colourless liquid that absorbs water from the environment, and freezes at 16.7 °C to a colourless crystalline solid. It is a weak acid, in that it is only partially dissociated acid in aqueous solution. It is one of the simplest carboxylic acids, and an important chemical reagent and industrial chemical. In the food industry acetic acid is used as an acidity regulator ***(Akeroyd, 1993)***.

Acetic acid was produced up to about 8-10% concentration by bacterial oxidation of alcohol. More concentrated solutions were produced synthetically by oxidation of propane or butane, by reaction of methanol and CO and by oxidation of ethanol from acetylene ***(Shreve, 1956)***. The nomenclature of acetic acid often lead to confusion as to whether concentrations were expressed as percentages of concentrated acetic acid ($C_2H_4O_2$) or of this diluted form. Therefore, the percentage or the following titles: diluted acetic acid or acidum aceticum dilutum should be informed ***(Reyolds et al, 1996)***. It was miscible with water, alcohol and glycerol. Its solution was sterilized by autoclaving and stored in air tight containers ***(Budavari et al, 1989)***. Acetic acid was excellent solvent for many organic compounds, also dissolvent phosphorus, sulfur and halogen acids ***(Bryson, 1997)***, also it has well known role in metabolism of fat and carbohydrates. It's used as analgesic, antipyretic, anti-rheumatic and in treatment of black and hairy tongue ***(Patty et al, 1967)***.

1.6.5 Citric Acid $C_6H_8O_7$

Is a weak organic acid, and it is a natural preservative and is also used to add an acidic, or sour, taste to foods and soft drinks. In biochemistry, it is important as an intermediate in the citric acid cycle and therefore occurs in the metabolism of virtually all living things. It can also be used as an environmentally benign cleaning agent and acts as an antioxidant and a lubricant. Citric acid exists in greater than trace amounts in a variety of fruits and vegetables, most notably citrus fruits. Lemons and limes have particularly high concentrations of the acid; it can constitute as much as 8% of the dry weight of these fruits ***(Penniston et al, 2008)***.

Chapter two

Materials and methods

Chapter Two

Materials and Methods

2.1 Materials

The materials which were used in the present study could be classified into: orthodontic materials, chemical reagents, equipment and instruments.

2.1.1 Orthodontic Materials *(Fig 2–1)*

I. Stainless steel Roth bracket with 0.022 ×0.030 inch slot (Ortho Technology, USA, Bionic Bracket System, no. BM22-1345).
II. Rectangular stainless steel orthodontic archwire of 0.017×0.025 inch (Ortho Technology, USA, no.8000-109).
III. Stainless steel orthodontic bands (Ortho Technology, USA).
IV. Stainless steel ligature wire (Ortho Technology, USA, no. B11-F03).

2.1.2 Chemical Reagents

These materials are used as testing immersion media in the present study, they include:

A. Ethanol (Gainland chemical company, United Kingdom) (Fig 2–1).
B. Corn oil (Afia, Jordan) (Fig 2–1).
C. Acetic acid (E. Merck, Darmstadt, Germany) (Fig 2–1).
D. Citric acid (Fig 2–1).
E. Artificial saliva.
F. Distilled water (Al-Iraqia Company for water treatment) (Fig 2–1).

2.1.3 Other Materials and Instruments

1. Bracket clamping tweezers (Dentaurum, Germany) (Fig 2–1).
2. Artery forceps (Dentaurum, Germany) (Fig 2–1).
3. Side cutter (Dentaurum, Germany) (Fig 2–1).

4. Dental vernier (Dentaurum, Germany) (Fig 2–1).
5. Angle player (Dentaurum, Germany) (Fig 2–1).
6. Large cutter (Dentaurum, Germany) (Fig 2–1).
7. Glass tubes with screw cap (Citoglas, China) (Fig 2–1).
8. Class container of different size (Fig 2–1).
9. Class beaker of different size (Fig 2–1).
10. Acetone (Gainland chemical company, United Kingdom) (Fig 2–1).
11. Dental floss (Oral B,Ireland) (Fig 2–1).
12. Silica gel particle (Fig 2–1).
13. Iron kit (Spinreact, spin).
14. Fine end permanent marker (Staedtler, Germany) (Fig 2–1).
15. Nylon suture.
16. Parafilm (Laboratory film, American national can, Chicago, USA).
17. Buffer powder: used to prepare buffering solution (standard PH). NaH_2PO_4 represent the buffer system.
18. Mechanical micro-pipette (Slamed, Germany) (Fig 2–9).
19. Plane tube (Citotest, china).
20. Pipette tips (Citotest, china).

1.2.4 Equipment

- Analytic balance (precisa, model XB 220A, Switzerland) (Fig 2–2).
- Ultrasonic cleaning bath (Sonomatic/170-2-T80, Germany) (Fig 2–3).
- PH- meter (Jenway, model 3320, Germany) (Fig 2–4).
- Incubator (Fisher scientific, model 5500/Germany) (Fig 2–5).
- Atomic absorption spectrophotometer (Back scientific/graphite furnace, model 210 VGP, USA) (Fig 2–8).
- Spectrophotometer (Cecil ce 7200, Japan) (Fig 2–10).
- Optical microscope (Nikon Eclipse Me 600 with digital camera DXM1200F) (Fig 2–11).

Fig (2–1) Some of the material and instrument used in this study

Fig (2–2) Analytic balance

Fig (2–3) Ultrasonic cleaning bath

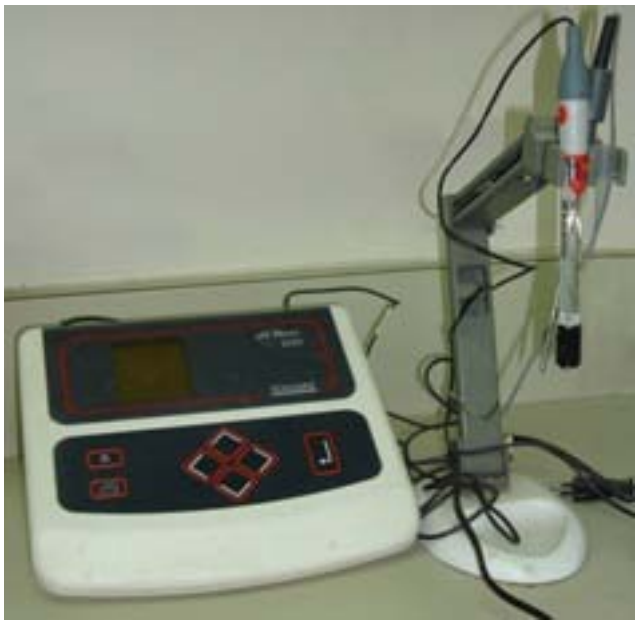

Fig (2–4) PH-meter

Fig (2–5) Incubator

2.2 Methods

2.2.1 Preparation of the Orthodontic Samples Used In the Study

Our sample includes seventy orthodontic sets. Each set represents half average fixed orthodontic appliance, from the central incisor to the first molar, for the maxillary arch. Each appliance consisted of five brackets, from second premolar to central incisor, a molar band with double tubes and a hook buccally and cleats lingually, and an archwire tied to the brackets and band with metal ligature wires. Because the same size bands may have different weights, so with the aid of analytical balance device (Fig 2–2), only the closely weights were chosen to be used ***(Al-Joboury, 2001; Kuhta et al, 2009)***.

The brackets used for a specific tooth (eg. lateral incisor) may have different weights, so again by using the analytical balance device, only the closely similar weights were used ***(Al-Joboury, 2001)***.

The archwire length was determined basing on a standard cast, and it was found that it was about 5.8cm. It measured after fixing the brackets on the proper position for each tooth and the band on the first permanent molar and placing stopper mesial to the molar band to prevent mesial slippage and sliding friction. The measurement was done from the midline point anteriorly and extended 1.5 mm distal to the first molar band tube posteriorly, the end of the wire was bent posteriorly to prevent slippage of the appliance during the experiments. Only the closely similar weights wires were used as that done for the bands and brackets ***(Al-Joboury, 2001)***.

Each piece of the appliance was cleaned ultrasonically with ethanol and acetone (Fig 2–3), rinsed with distilled water and then dried in hot air ***(Huang et al, 2003; Oh and Kim, 2005)***, and stored in closely packed glass container which contain silica gel particles to avoid any oxidation or contamination of the alloy. After that, the components of each sample were gathered and held securely to the 0.017 by 0.025 inch stainless steel archwire. The ligation was done by using 0.001 inch stainless steel ligature wires (Fig 2–6) ***(Al-Joboury, 2001)***.

The samples then were immersed again in acetone for 8 seconds and dried in hot air and restored again, this is to get rid of any contaminants from the fingers of the operator during ligation ***(Barret et al, 1993; Al-Joboury, 2001)***.

No attempt was made in the present study to cover either the inner surface of the bands or the bonding surface of the brackets as was previously suggested. This was done to eliminate the introduction of any possible extraneous sources of ions. In a clinical situation, the inner surface of bands would be coated with a cementing medium, and the mesh or bracket bases would be covered with a composite bonding material. Therefore it could be assumed that the surfaces available for biodegradation and metal release are approximately double what would be available clinically. The exposed surface area may be considered as equivalent to full maxillary and mandibular fixed appliance after its placement ***(Park and Shearer, 1983; Barret et al, 1993; Al-Joboury, 2001)***.

Fig (2–6) Orthodontic sample used in this study

2.2.2 Testing Solutions

The testing solutions include aqueous foods, alcoholic foods, acidic foods, and fatty foods as represented by dietary simulating liquids (10% and 50% ethanol, 3% acetic acid, 1% citric acid, corn oil) in addition to distilled water and artificial

saliva ***(Scottish Statutory Instrument, 2006;United States Food and Drug Administration Guideline, 2006;Geraldo et al,2007; Yousif, 2007; Hemed; 2008)***

2.2.2.1 Preparation of Testing Solutions

A. Artificial Saliva

The artificial saliva which is used in this study consisted of 0.7g NaCl , 1.2g KCl , 0.26g Na_2HPO_4 , 0.2g K_2HPO_4 , 1.5g $NaHCO_3$, 0.33g KSCN , 0.13g urea and 1000 ml deionized water, this formula is named modified Carter's solution which is a modification to the old one used by Gerdet and Hero in 1963 ***(Duffo and Quaezada, 2004)***.

The differences being that the old formula contains ($CaCl_2$). Since the presence of calcium ion interferes with the atomization of chromium in the atomic absorption spectrophotometer and so prevent the accurate measurement of chromium. As a result, it was decided to substitute calcium chloride with an equal amount of potassium chloride which more matches the corrosive properties of calcium chloride. Proteins were eliminated from the formula since its high endogenous concentration of nickel ***(Barrett et al, 1993)***.

All the components of this formula are present in the natural saliva ***(Durrant, 1982)***. These particular weights were measured by electronic balance then dissolved in deionized water to be added at the moment of use ***(Querioz et al, 2007)***.

The PH of the artificial saliva was adjusted to (7 ± 0.2) to simulate the oral environment (the range of normal saliva and dental plaque) ***(Ferriter et al, 1990)***. Buffer powder was used to prepare buffering solution (standard pH). The pH meter readings were calibrated using this solution before measuring the pH of the artificial saliva. Buffering solution prepared by dissolving this buffer powder in 100ml distilled water ***(Al-Joboury, 2001)***.

B. Preparation of 10% Ethanol

For the preparation of 300 ml of 10% ethanol solution, 270 ml of distilled water was added to 30 ml absolute ethanol (99.85%) according to the formula C1V1=C2V2 ***(Scopes, 1994; Farone and Farone, 1999; Seidman and Moore,***

2000; Gerstein, 2001; Reed et al, 2003). By using the PH meter it was found that the PH of 50% ethanol is 5.7 ± 0.2.

C. Preparation of 50 % Ethanol

According to the equation mentioned previously, 150 ml distilled water was added to 150 ml absolute ethanol (99.85%) for the preparation of 300 ml of 50% aqueous ethanol***(Scopes, 1994; Farone and Farone, 1999; Seidman and Moore, 2000; Gerstein, 2001; Reed et al, 2003)***. Then the PH of the solution was measured using PH meter, and it is found that it equal to PH= 5.5± 0.2.

D. Preparation of 3% Acetic Acid

Three percent of acetic acid was prepared by dissolution of nine ml of concentrated acetic acid in 291 ml distilled water ***(Seidman and Moore, 2000; Reed et al, 2003)***. Then the PH of the solution is measured using PH meter, and found to be equal to 2.9± 0.2.

E. Preparation of 1% Citric Acid

For the preparation of 300 ml of 1% citric acid and according to the same equation , 3 g of citric acid is dissolved in 300 ml of distilled water ***(Seidman and Moore, 2000; Reed et al, 2003)***, and the PH of the solution is found to be 2.7 ± 0.2.

2.2.3 Immersion Procedure

After the preparation of the orthodontic sets and testing solutions, the samples are divided into seven groups, each group contain ten sets.

Each set was placed in a glass container contain 30ml of artificial saliva or the chosen food simulant, and held at the end of the archwire distal to the first molar tube using dental floss in such a way that the sample was fully immersed in the testing solution without touching the walls of the container (Fig 2–7). The glass containers were placed in the incubator at 37°C for 28 days ***(Al-Joboury, 2001)*** (Fig 2–5). Each container was closed by parafilm to control evaporation. Continuous solution exposure was selected to accelerate the weathering effect on the material tested ***(Lee et al, 1996; Lee et al, 1998)***.

To avoid saturation with the released ions during the incubation period, the solutions were changed at 7 days intervals, so that it will be changed on the days 7, 14, 21, and 28 days. At the end of 37°C incubation, one drop of 65% HNO_3 was added into each sample with a PH of ≥ 6.75 to keep the released ions stable in the solution ***(Elshahawy et al, 2009a; Kuhta et al, 2009)***.

Fig (2–7) Orthodontic Sample Immersed In Testing Solution

2.2.4 Weighing the Orthodontic Samples

Each orthodontic set was weighed for five times, the first time was before the immersion in the testing solution and the others were at the end of each time interval using analytic balance device. Each sample was weighed after they dried in hot air.

2.2.5 Estimation of Ions Concentration

The solution was analyzed for chromium, nickel, copper and iron concentration using atomic absorption spectrophotometer (Buck scientific, 210VGP, USA) and spectrophotometer (Cecil ce 7200, Japan) following standardized procedure, Fig (2–8), (2–10).

Fig (2–8) Atomic Absorption Spectrophotometer

For atomic absorption spectrophotometer settings were performed according to instrumental manufacturer's specifications. Hallow cathode lamps were used for Cr, Ni and Cu. Absorption was measured in fuel–rich flame in order to obtain maximum sensitivity. Single element hollow cathode were operated at currents or energies recommended by the manufacturer, optimum working condition regarding wave length and slit width of the instrument for each element are given in table (2–1).

Table (2–1) Optimum working condition and instrumental setting for each element using Buck Scientific, 210VGP AAS

Element	*Wave length (nm)*	*Mode*	*Slit width (nm)*
Cr	357.9	Air–acetylene flame(ABS)	0.7
Ni	232	Air–acetylene flame(ABS)	0.2
Cu	324.7	Air–acetylene flame(ABS)	0.7

2.2.5.1 Preparation of Samples

1. Stock standard 1000 ppm calibration standard of chromium, nickel and copper were prepared by dilution the standard sample with 1000 ml of deionized water.
2. Working standards of the element to be analyzed were prepared from zero concentration and upwards of at least five concentrations or more. Standards were prepared in polyethylene tube and prepared freshly before reading.
3. Instrumental and gas flame setting for each element studied was given in table (2–1). Aspiration of zero concentration standards was done to set baseline to read zero absorbance, this process was repeated frequently to correct baseline drift.
4. Working standards was analyzed sequentially from most diluted to most concentrated value. The resulting values were used to establish working regression curve.
5. Analysis of the collected sample after each time period, the sample was aspirated directly into the flame.
6. Calculation of specimen concentration from absorbance reading by interpolation from the working regression curve.

Diluted hydrochloric acid was periodically aspirated to clean nebulizer system and the burner head was cleaned before every run to obtain optimal results.

2.2.5.2 Determination of Chromium, Nickel and Copper Concentrations

Stock solution (1000ppm) was diluted with deionized water to give the following concentration of the working standard (0.0, 0.1, 0.2, 0.3, 0.4 μg/ml) for chromium, (0.0, 0.2, 0.4, 0.6, 0.8 μg/ml) for nickel and (0.0, 3, 6, 9, 12 μg/ml) for copper.

Precautions were taken to avoid contamination during sample preparation or from reagents. So 2 ml of each solution was aspirated using a mechanical micro-pipette (Fig 2–9) and new pipette tip at each time of measurement of each ion, the

aspirated solution was put in a new plane tube to avoid any contamination. Analysis of samples and standard was done. Calculation of values was obtained from working regression curve.

Fig (2–9) Mechanical Micro- Pipette

2.2.5.3 Determination of Iron Concentration

The method used to determine the concentration of iron is by colometric method and used spin kit according to the manufactured instructions. The principle of this method is that the iron is dissociated in weekly acid medium, and then liberated iron was reduced into the bivalent form by means of ascorbic acid. Ferrous ions give with ferrozine a colored complex. The intensity of the color formed was proportional to the iron concentration in the sample. The color was measured using spectrophotometer at 562 nm (Fig 2–10).

Fig (2– 10) Spectrophotometer

2.2.6 Microscopical Examination

Microscopical examination was performed to determine the type, position, and severity of corrosion in the components of fixed orthodontic appliance. The microscopical examination was done using optical microscope (Nikon Eclipse Me 600 with digital camera dxm1200F) (Fig 2–11). This microscope was supplied by many accessories to allow the observation of metal pieces and photomicrography.

The samples were examined at the end of the 4th interval (28 day) because the appearance of pits, crevices, inter-granular corrosion requires many days or weeks to be detected ***(Fontana and green, 1982)***.

In this study random samples were taken from each group to represent the different studied groups, and their surface were examined and photographs were taken at 50X, 100X, 200X, 500X, 1000X. The 100X and 200 X magnifications were considered to be more comparable; therefore, they were selected for this study, then the photographs were cropped and organized.

The sites that were examined thoroughly include:

1. Wire sides at the bracket slot - archwire interface and wire parts at the inter-bracket areas.
2. Wire section that pass through the molar band tube.
3. Areas of ligation of the ligature wires on the archwires and bracket wings.
4. The slot of the bracket.
5. Wings of the bracket.
6. Mesh of the bracket.
7. Molar bands including the areas of weld at the site of union between the band and the tube, and between the cleats of the bands.

Fig (2–11) Optical microscope

2.2.7 Pilot Study

A pilot study was carried out to:

1. Training on measurement procedure.
2. Test the workability and efficiency of the equipment that has been used for measuring the appliance weights.
3. Asses the validity of the measurement and eliminate as much as possible the limitation that could be faced during the course of the study, so the reliability of the researchers work (intra and inter examiner calibration) can be evaluated. The measurements were carried out twice by the examiner alone and for a third time by the professional using the same specimens at different time.

Ten orthodontic samples were prepared according to the method mentioned previously. The weights of these samples were taken using analytic balance before immersion in the solutions twice by the researcher and for the third time by the professional. Intra and inter examiner calibration show 100% agreement (Table 2– 2).

Table (2–2) Intra and inter examiner calibration of samples weight

	Intra-examiner calibration	*Inter-examiner calibration*
% of agreement	100%	100%

2.2.8 Statistical Analysis

Data were collected and analyzed using SPSS (statistical package of social science) software version 15 for windows XP Chicago, USA. The suitable statistical methods were used in order to analyze and assess the results; they include the followings ***(Sorlie, 1995)***:

A. Descriptive Statistics

- Summary statistics of the reading distribution (mean, SD, minimum, maximum).
- Statistical tables and graphical presentation.

B. Inferential Statistics

- ***ONE WAY ANALYSIS OF VARIANCE (ANOVA):*** To test any statistically significant difference among the weights of the sample and the amounts of the ion released concerning the test periods and the effect of different storage media.

- ***LEAST SIGNIFICANT DIFFERENCE (LSD)***: was performed when (ANOVA) test showed a statistical significant difference, to assess any statistical significant difference between:

I. Two successive point of time (linked pairs) to detect the effect of time on the weight and ion release behavior of orthodontic sample.

II. Each two group of storage media to detect the effect of different media on the weight and ion release behavior of orthodontic sample.

(P) Value of more than 0.05 was regarded as statistically insignificant as follows:

P>0.05 **NS** Non – significant

0.05≥ P>0.01 **S** significant

0.01≥ P>0.001 **HS** Highly significant

P≤ 0.001 **VHS** very highly significant

Chapter Three

Results

Chapter Three

Results

3.1 The Amount of Ion Release

Descriptive statistics were performed for the measured four ions (chromium, nickel, copper and ferrous ion) released from the tested appliance, at the four time interval (7, 14, 21, 28 days) for each storage medium (distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, 1% citric acid) in addition to artificial saliva.

These statistics included mean, standered deviation, minimum and maximum values, which are given in table (3–1). As showed in this table, the highest levels of ions were released when the tested appliances were immersed in 1% citric acid and 3% acetic acid, and the least amount of ions were released in corn oil and distilled water. This table also showed that the amounts of ion released were greatly affected by the time factor.

3.1.1 Effect of Time on the Ion Release in Different Storage Media

3.1.1.1 Changes in Chromium Ion Release

As clearly illustrated in table (3–1), the greatest amount of chromium ion were released in the first week of this study, then the released rate decreased till reach the minimal level thought day 28. The changes in chromium release were very sharp from day 7 downward as showed in figure (3–1).

One way analysis of variance (ANOVA) showed very highly significant difference in chromium ion release at different testing interval for each of the storage medium. Consequently, least significant difference (LSD) test was performed between each successive points of time over all the study period in the studied storage media table (3–2), the (LSD) test showed there were very highly significant differences in the time effect between 7 and 14 days for all of the storage media

except that for distilled water where there was a highly significant difference. Later on, since the amount of released ion was decreased, the (LSD) test show less significant or no significant difference among the time intervals.

3.1.1.2 Changes in Nickel Ion Release

It can be seen in table (3–1), and figure (3–2) that the greatest amount of nickel ion were released in the first week of this study, then the released rate decreased till reach the minimal level thought day 28. The changes in nickel ion release were very sharp from day 7 downward.

The result of (ANOVA) test showed very highly significant difference in nickel ion release at different testing interval for each of the storage medium. These result specified by (LSD) test which showed that there were very highly significant differences in the time effect between 7 and 14 days for all of the storage media except for corn oil where there was no significant difference, and in 50% ethanol there was highly significant difference. Then the amount of released ion was decreased, so the (LSD) test show less significant or no significant difference among the time interval.

3.1.1.3 Changes in Copper Ion Release

By inspecting the data value in table (3–1), and figure (3–3) ,it can be seen that the greatest amount of copper ion were released in the first two week of this study, then the release rate decreased till reach the minimal level thought day 28.

The result of (ANOVA) test showed very highly significant difference in copper ion release at different testing interval for each of the storage medium. This result specified by (LSD) test which showed that there were very highly significant differences in the time effect between all of the storage media in the first two week except for corn oil and distilled water where there was no significant difference, and in 50% ethanol there was significant difference. With the progress of the study, the amount of released ion was decreased, so the (LSD) test show less significant or no significant difference among the time intervals.

3.1.1.4 Changes in Iron Ion Release

It can be seen in table (3–1), and figure (3–4) that the greatest amount of iron ion were released in the first week of this study, then the released rate decreased till reach the minimal level thought day 28. The changes in iron ion release were very sharp from day 7 downward.

The result of (ANOVA) test showed very highly significant difference in iron ion release at different testing interval for each of the storage medium. These result specified by (LSD) test which showed that there were very highly significant differences in the time effect between 7 and 14 days for all of the storage media except for distilled water where there was highly significant difference. Then, the amount of released ion was decreased, so the (LSD) test show less significant or no significant difference among the time interval.

3.1.2 Effect of Different Storage Media on Ions Release

The effect of different storage media on the ion release from the orthodontic set used in this study appeared to be marked over the storage periods. The larger amount of chromium, nickel, copper and iron ion were released in the 1% citric acid and 3% acetic acid media followed by 10% ethanol and 50% ethanol then followed by artificial saliva and finally there were distilled water and corn oil as seen on table (3–1).

3.1.2.1 Chromium Ion Release Levels

Analysis of variance difference (ANOVA) has demonstrated a very highly significant difference on the chromium ion release in the entire tested media (artificial saliva, distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, and 1% citric acid) table (3–3). These results were specified by least significant difference (LSD) test which showed significant difference in the chromium ion release among the test solutions except between distilled water and artificial saliva. The LSD test showed no significant difference between distilled water and corn oil, 10%ethanol and50% ethanol, 3% acetic acid and 1% citric acid after 7 days. Then

differences between the chromium ion releases become little to indicate less significant difference with the progress of the study table (3–4); figure (3–1).

3.1.2.2 Nickel Ion Release Levels

The result of (ANOVA) test showed very highly significant difference on the nickel ion release in all of the tested media (artificial saliva, distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, and 1% citric acid), table (3–3), and least significant difference (LSD) test showed significant difference in the nickel ion release among the test solutions except between distilled water and corn oil. The LSD test showed no significant difference between 10%ethanol and50% ethanol after 7 days. Then differences between the nickel ion releases become little to indicate less significant difference with the progress of the study table (3–5); figure (3–2).

3.1.2.3 Copper Ion Release Levels

Analysis of variance difference (ANOVA) showed very highly significant difference on the copper ion release among the tested media (artificial saliva, distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, and 1% citric acid), table (3–3). These results were specified by least significant difference (LSD) test which showed significant difference in the copper ion release among the test solutions except between distilled water and corn oil. The LSD test showed no significant difference between 10%ethanol and 50% ethanol, 3% acetic acid and 1% citric acid after 7 days. Later on, differences between the copper ion releases become little to indicate less significant difference with the progress of the study table (3–6); figure (3–3).

3.1.2.4 Iron Ion Release Levels

By inspecting the result of (ANOVA) test, it can be seen that there were very highly significant difference on the iron ion release in all of the tested media (artificial saliva, distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, and 1% citric acid), table (3–3). The least significant difference (LSD) test showed significant difference in the iron ion release among the test solutions except between artificial saliva and corn oil and between 3% acetic acid and 1% citric acid after 7

days showed no significant difference. With the progress of the study the differences between the iron ion releases shows less significant difference table (3–7); figure (3–4).

Table (3–1) Descriptive statistic of chromium, nickel, copper and iron ion released in different test period and storage media

Groups	Media	Days	Ions															
			Cr				Ni				Cu				Fe			
			Mean	SD	Min	Max	Mean	SD	Min	Max	Mean	SD	Min	Max	Mean	SD	Min	Max
Gr1	Artificial Saliva	7	0.090	0.015	0.070	0.110	0.121	0.016	0.090	0.140	2.050	0.190	1.800	2.300	0.240	0.059	0.167	0.333
		14	0.055	0.011	0.040	0.070	0.068	0.023	0.030	0.100	1.510	0.247	1.300	2.000	0.118	0.023	0.087	0.167
		21	0.043	0.008	0.030	0.050	0.046	0.016	0.025	0.080	1.200	0.236	0.900	1.500	0.096	0.011	0.083	0.117
		28	0.023	0.009	0.010	0.040	0.033	0.012	0.020	0.050	0.430	0.142	0.300	0.700	0.069	0.020	0.033	0.103
Gr2	DW	7	0.071	0.019	0.040	0.090	0.079	0.017	0.050	0.100	0.150	0.014	0.127	0.169	0.070	0.024	0.043	0.103
		14	0.050	0.017	0.030	0.090	0.047	0.019	0.030	0.090	0.138	0.014	0.119	0.165	0.041	0.020	0.020	0.083
		21	0.034	0.010	0.020	0.050	0.031	0.010	0.020	0.050	0.067	0.023	0.032	0.102	0.022	0.010	0.007	0.033
		28	0.020	0.008	0.010	0.030	0.015	0.005	0.010	0.025	0.042	0.015	0.030	0.072	0.012	0.009	0.003	0.033
Gr3	10% Ethanol	7	0.146	0.025	0.120	0.190	0.221	0.027	0.180	0.260	4.310	0.331	4.000	5.000	0.492	0.050	0.433	0.567
		14	0.082	0.019	0.050	0.110	0.154	0.015	0.140	0.190	0.640	0.178	0.400	0.900	0.159	0.038	0.093	0.203
		21	0.046	0.016	0.020	0.070	0.036	0.012	0.020	0.060	0.400	0.082	0.300	0.500	0.108	0.028	0.067	0.147
		28	0.037	0.013	0.018	0.050	0.018	0.013	0.003	0.030	0.240	0.052	0.200	0.300	0.102	0.026	0.067	0.143
Gr4	50% Ethanol	7	0.148	0.030	0.100	0.200	0.243	0.032	0.200	0.290	4.000	1.155	2.000	5.000	0.403	0.030	0.367	0.467
		14	0.085	0.010	0.070	0.100	0.199	0.014	0.180	0.230	3.100	0.738	2.000	4.000	0.171	0.023	0.143	0.203
		21	0.071	0.009	0.050	0.080	0.105	0.041	0.050	0.150	2.190	0.540	1.000	2.700	0.098	0.021	0.067	0.133
		28	0.052	0.011	0.035	0.070	0.041	0.025	0.020	0.100	1.230	0.562	0.400	2.000	0.040	0.017	0.023	0.067
Gr5	3% Acetic Acid	7	0.249	0.038	0.200	0.320	0.525	0.069	0.450	0.640	15.070	0.691	14.000	16.000	0.910	0.050	0.853	0.970
		14	0.066	0.020	0.040	0.100	0.163	0.029	0.130	0.220	13.150	1.021	11.500	14.500	0.377	0.033	0.333	0.433
		21	0.046	0.011	0.030	0.070	0.072	0.017	0.050	0.100	2.720	0.257	2.300	3.000	0.213	0.039	0.167	0.267
		28	0.031	0.012	0.010	0.050	0.063	0.019	0.045	0.100	0.800	0.115	0.600	1.000	0.113	0.028	0.067	0.157
Gr6	Corn Oil	7	0.051	0.017	0.020	0.080	0.076	0.013	0.060	0.100	0.050	0.008	0.042	0.070	0.223	0.016	0.200	0.243
		14	0.015	0.006	0.008	0.028	0.065	0.012	0.050	0.080	0.043	0.006	0.034	0.052	0.110	0.027	0.067	0.147
		21	0.011	0.006	0.004	0.022	0.040	0.012	0.010	0.060	0.040	0.005	0.030	0.050	0.077	0.021	0.053	0.107
		28	0.008	0.004	0.004	0.015	0.020	0.016	0.000	0.040	0.021	0.008	0.012	0.039	0.049	0.010	0.033	0.060
Gr7	1% Citric Acid	7	0.248	0.031	0.190	0.290	0.628	0.065	0.550	0.750	15.280	0.911	13.400	16.500	0.931	0.045	0.867	1.000
		14	0.181	0.025	0.120	0.210	0.413	0.055	0.360	0.500	8.020	1.361	6.600	10.500	0.620	0.075	0.533	0.763
		21	0.083	0.019	0.050	0.110	0.132	0.030	0.100	0.200	5.540	0.749	4.700	6.600	0.197	0.041	0.150	0.253
		28	0.053	0.016	0.030	0.080	0.088	0.011	0.070	0.110	1.950	0.268	1.700	2.500	0.041	0.014	0.027	0.067

Table (3–2) Difference in the chromium, nickel, copper and iron ions release between two successive time interval at different storage periods (ANOVA test, LSD test, p– values)

Gps	Media	Ions	ANOVA (d.f. = 39)		LSD (p level)		
			F	p	7 day/14 day	14 day/21 day	21 day/28 day
Gp 1	Artificial Saliva	Cr	63.698	0.000	0.000	0.021	0.000
		Ni	51.835	0.000	0.000	0.007	0.097
		Cu	105.992	0.000	0.000	0.002	0.000
		Fe	49.804	0.000	0.000	0.156	0.084
Gp 2	Distilled water	Cr	23.587	0.000	0.002	0.017	0.035
		Ni	39.298	0.000	0.000	0.014	0.014
		Cu	93.564	0.000	0.131	0.000	0.003
		Fe	22.177	0.000	0.001	0.018	0.200
Gp 3	10% Ethanol	Cr	69.435	0.000	0.000	0.000	0.281
		Ni	301.200	0.000	0.000	0.000	0.030
		Cu	1007.335	0.000	0.000	0.009	0.074
		Fe	255.971	0.000	0.000	0.004	0.733
Gp 4	50% Ethanol	Cr	59.664	0.000	0.000	0.076	0.018
		Ni	92.967	0.000	0.002	0.000	0.000
		Cu	22.806	0.000	0.015	0.014	0.010
		Fe	466.063	0.000	0.000	0.000	0.000
Gp 5	3% Acetic Acid	Cr	195.286	0.000	0.000	0.060	0.154
		Ni	300.137	0.000	0.000	0.000	0.615
		Cu	1301.560	0.000	0.000	0.000	0.000
		Fe	861.956	0.000	0.000	0.000	0.000
Gp 6	Corn Oil	Cr	42.047	0.000	0.000	0.342	0.570
		Ni	35.306	0.000	0.075	0.000	0.002
		Cu	31.209	0.000	0.055	0.271	0.000
		Fe	156.064	0.000	0.000	0.000	0.003
Gp 7	1% Citric Acid	Cr	144.435	0.000	0.000	0.000	0.007
		Ni	314.477	0.000	0.000	0.000	0.036
		Cu	383.178	0.000	0.000	0.000	0.000
		Fe	686.330	0.000	0.000	0.000	0.000

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

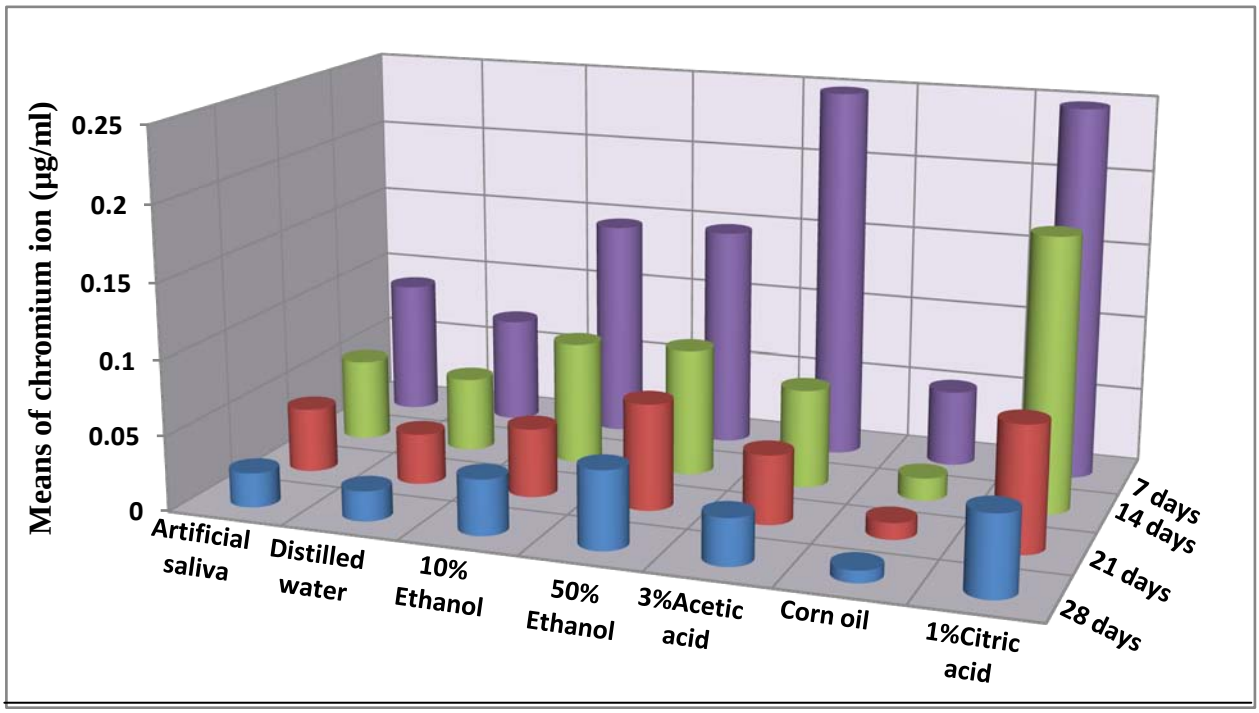

Figure (3–1) Mean distribution of Chromium–ion (Cr) levels (µg/ml) in the related periods for different studied group's Media

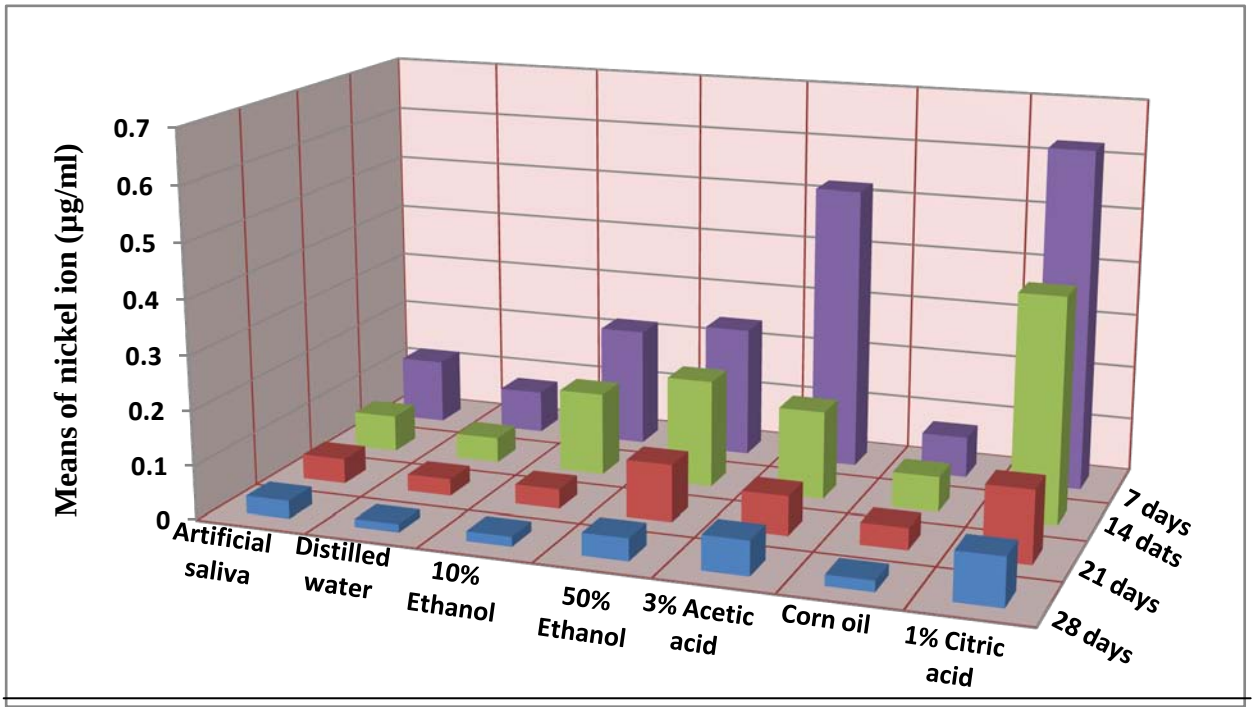

Figure (3–2) Mean distribution of nickel –ion (Ni) levels (µg/ml) in the related periods for different studied group's Media

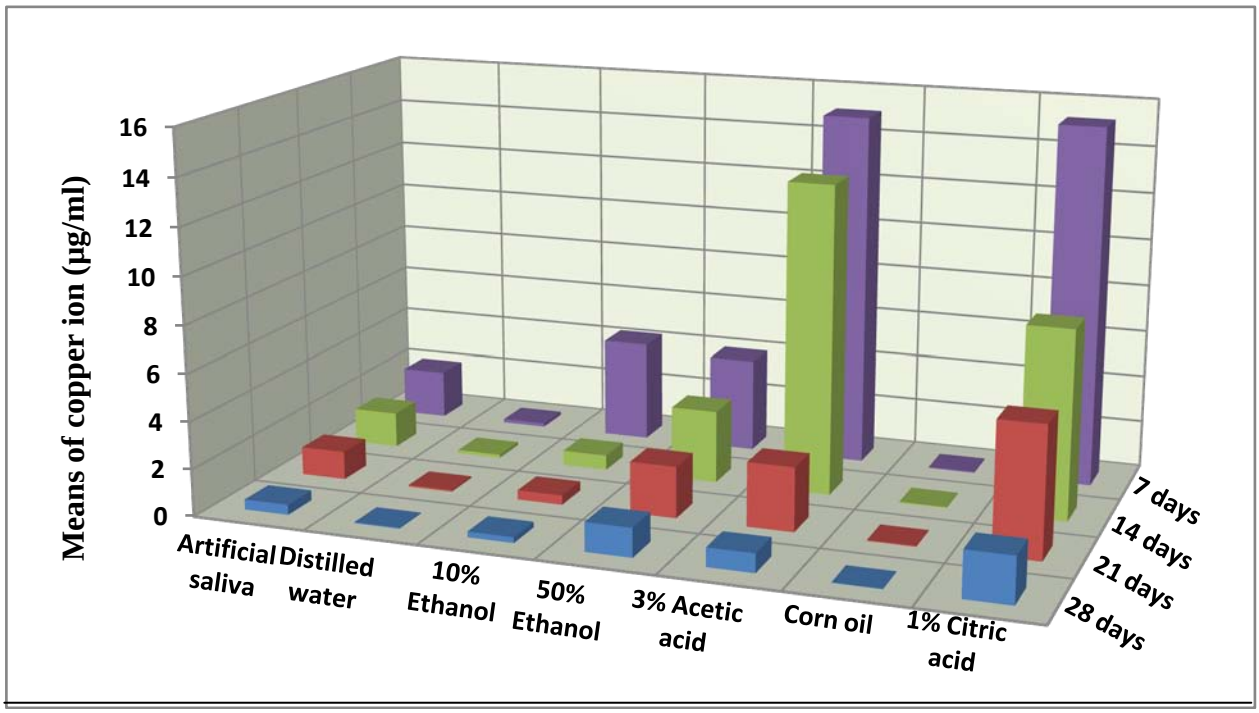

Figure (3–3) Mean distribution of copper –ion (Cu) levels (µg/ ml) in the related periods for different studied group's Media

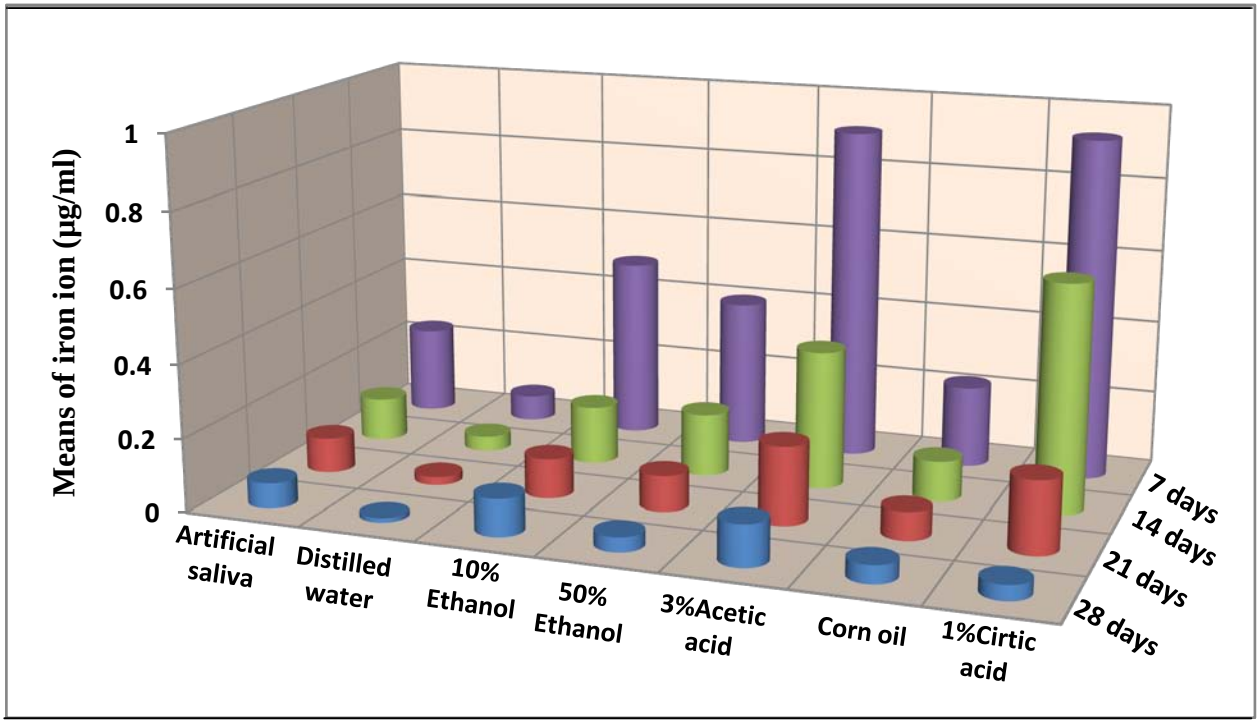

Figure (3–4) Mean distribution of iron –ion (Fe) levels (µg/ml) in the related periods for different studied group's Media

Table (3–3) Difference in the chromium, nickel, copper and iron ions release in different storage media during each storage period (ANOVA test, p– values)

Days	Ions	ANOVA	
		F	p
7	Cr	94.122	0.000
	Ni	298.397	0.000
	Cu	1089.205	0.000
	Fe	654.484	0.000
14	Cr	96.611	0.000
	Ni	213.285	0.000
	Cu	490.303	0.000
	Fe	275.655	0.000
21	Cr	38.497	0.000
	Ni	30.008	0.000
	Cu	277.877	0.000
	Fe	61.802	0.000
28	Cr	21.906	0.000
	Ni	30.193	0.000
	Cu	83.331	0.000
	Fe	35.803	0.000

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

Table (3–4) Difference in chromium ion release in different storage media during each storage period (LSD test, p– values)

Media		Cr			
		7	14	21	28
Artificial Saliva	DW	0.110	0.504	0.099	0.549
Artificial Saliva	10% Ethanol	0.000	0.001	0.579	0.007
Artificial Saliva	50% Ethanol	0.000	0.000	0.000	0.000
Artificial Saliva	3% Acetic Acid	0.000	0.145	0.579	0.113
Artificial Saliva	Corn Oil	0.001	0.000	0.000	0.004
Artificial Saliva	1% Citric Acid	0.000	0.000	0.000	0.000
DW	10% Ethanol	0.000	0.000	0.029	0.001
DW	50% Ethanol	0.000	0.000	0.000	0.000
DW	3% Acetic Acid	0.000	0.036	0.029	0.031
DW	Corn Oil	0.093	0.000	0.000	0.022
DW	1% Citric Acid	0.000	0.000	0.000	0.000
10% Ethanol	50% Ethanol	0.865	0.738	0.000	0.004
10% Ethanol	3% Acetic Acid	0.000	0.036	1.000	0.249
10% Ethanol	Corn Oil	0.000	0.000	0.000	0.000
10% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.002
50% Ethanol	3% Acetic Acid	0.000	0.016	0.000	0.000
50% Ethanol	Corn Oil	0.000	0.000	0.000	0.000
50% Ethanol	1% Citric Acid	0.000	0.000	0.023	0.764
3% Acetic Acid	Corn Oil	0.000	0.000	0.000	0.000
3% Acetic Acid	1% Citric Acid	0.932	0.000	0.000	0.000
Corn Oil	1% Citric Acid	0.000	0.000	0.000	0.000

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

Table (3–5) Difference in nickel ion release in different storage media during each storage period (LSD test, p– values)

Media		Ni			
		7	14	21	28
Artificial Saliva	DW	0.023	0.091	0.141	0.012
Artificial Saliva	10% Ethanol	0.000	0.000	0.324	0.037
Artificial Saliva	50% Ethanol	0.000	0.000	0.000	0.255
Artificial Saliva	3% Acetic Acid	0.000	0.000	0.012	0.000
Artificial Saliva	Corn Oil	0.015	0.807	0.553	0.063
Artificial Saliva	1% Citric Acid	0.000	0.000	0.000	0.000
DW	10% Ethanol	0.000	0.000	0.621	0.647
DW	50% Ethanol	0.000	0.000	0.000	0.000
DW	3% Acetic Acid	0.000	0.000	0.000	0.000
DW	Corn Oil	0.869	0.146	0.374	0.493
DW	1% Citric Acid	0.000	0.000	0.000	0.000
10% Ethanol	50% Ethanol	0.239	0.000	0.000	0.002
10% Ethanol	3% Acetic Acid	0.000	0.465	0.001	0.000
10% Ethanol	Corn Oil	0.000	0.000	0.692	0.819
10% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.000
50% Ethanol	3% Acetic Acid	0.000	0.005	0.002	0.002
50% Ethanol	Corn Oil	0.000	0.000	0.000	0.003
50% Ethanol	1% Citric Acid	0.000	0.000	0.009	0.000
3% Acetic Acid	Corn Oil	0.000	0.000	0.002	0.000
3% Acetic Acid	1% Citric Acid	0.000	0.000	0.000	0.001
Corn Oil	1% Citric Acid	0.000	0.000	0.000	0.000

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

Table (3–6) Difference in copper ion release in different storage media during each storage period (LSD test, p– values)

Media		Cu			
		7	14	21	28
Artificial Saliva	DW	0.000	0.000	0.000	0.001
Artificial Saliva	10% Ethanol	0.000	0.008	0.000	0.089
Artificial Saliva	50% Ethanol	0.000	0.000	0.000	0.000
Artificial Saliva	3% Acetic Acid	0.000	0.000	0.000	0.001
Artificial Saliva	Corn Oil	0.000	0.000	0.000	0.000
Artificial Saliva	1% Citric Acid	0.000	0.000	0.000	0.000
DW	10% Ethanol	0.000	0.119	0.051	0.077
DW	50% Ethanol	0.000	0.000	0.000	0.000
DW	3% Acetic Acid	0.000	0.000	0.000	0.000
DW	Corn Oil	0.724	0.767	0.872	0.846
DW	1% Citric Acid	0.000	0.000	0.000	0.000
10% Ethanol	50% Ethanol	0.276	0.000	0.000	0.000
10% Ethanol	3% Acetic Acid	0.000	0.000	0.000	0.000
10% Ethanol	Corn Oil	0.000	0.065	0.035	0.051
10% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.000
50% Ethanol	3% Acetic Acid	0.000	0.000	0.002	0.000
50% Ethanol	Corn Oil	0.000	0.000	0.000	0.000
50% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.000
3% Acetic Acid	Corn Oil	0.000	0.000	0.000	0.000
3% Acetic Acid	1% Citric Acid	0.460	0.000	0.000	0.000
Corn Oil	1% Citric Acid	0.000	0.000	0.000	0.000

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

Table (3–7) Difference in iron ions release in different storage media during each storage period (LSD test, p– values)

Media		Fe			
		7	14	21	28
Artificial Saliva	DW	0.000	0.000	0.000	0.000
Artificial Saliva	10% Ethanol	0.000	0.020	0.312	0.000
Artificial Saliva	50% Ethanol	0.000	0.003	0.826	0.002
Artificial Saliva	3% Acetic Acid	0.000	0.000	0.000	0.000
Artificial Saliva	Corn Oil	0.376	0.658	0.121	0.027
Artificial Saliva	1% Citric Acid	0.000	0.000	0.000	0.002
DW	10% Ethanol	0.000	0.000	0.000	0.000
DW	50% Ethanol	0.000	0.000	0.000	0.002
DW	3% Acetic Acid	0.000	0.000	0.000	0.000
DW	Corn Oil	0.000	0.000	0.000	0.000
DW	1% Citric Acid	0.000	0.000	0.000	0.001
10% Ethanol	50% Ethanol	0.000	0.488	0.427	0.000
10% Ethanol	3% Acetic Acid	0.000	0.000	0.000	0.203
10% Ethanol	Corn Oil	0.000	0.006	0.012	0.000
10% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.000
50% Ethanol	3% Acetic Acid	0.000	0.000	0.000	0.000
50% Ethanol	Corn Oil	0.000	0.001	0.078	0.299
50% Ethanol	1% Citric Acid	0.000	0.000	0.000	0.912
3% Acetic Acid	Corn Oil	0.000	0.000	0.000	0.000
3% Acetic Acid	1% Citric Acid	0.272	0.000	0.173	0.000
Corn Oil	1% Citric Acid	0.000	0.000	0.000	0.353

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

3.2 Weight Changes of the Orthodontic Appliances

Weights were measured for all orthodontic sets used in this study. All of these orthodontic sets were measured before and at the four time intervals (7, 14, 21, 28 days) on each a storage medium used in this study. These storage media represented a food simulating liquids (distilled water, 10% ethanol, 50% ethanol, 3% acetic acid, corn oil, 1% citric acid) in addition to artificial saliva.

These statistics included mean, slandered deviation, minimum and maximum values, which are given in (Table 3–8).

3.2.1 Effect of Time on the Weight in Different Storage Media

The results of ANOVA (analysis of variance) tests showed that there were no significant differences between the weights of orthodontic sets at different time interval in each storage medium except for the 10% ethanol and corn oil which showed very highly significant difference(Table 3–9 and Figure 3–5).

These results were specified by LSD (least significant difference) test which showed that there were very highly significant differences in the weight of the sets immersed in 10% ethanol after 7 days of immersion, between 7 and 14 days, and between 21 and 28 days. By inspecting the result of LSD test, it can be seen that there were no significant difference in the weight of appliances after immersion in corn oil at each time interval (Table 3–9).

3.2.2 Effect of Different Storage Media on the Weight

Statistically, there was no significant difference in the weight among the studied groups before immersion (at zero time), as is detected by ANOVA (analysis of variance) test and specified by LSD (least significant difference) test (Table 3–10, Table 3–11 and Figure 3–5).

The ANOVA (analysis of variance) test showed that there were a very highly significant difference in the weight among studied groups on day 7 and day 28 , and there was a highly significant difference on day 21, while there was only a significant difference on day 14 (Table 3–10, Table 3–11 and Figure 3–5).

By specifying these results using LSD (least significant difference) test, it can be seen that the weight of the appliance showed a marked significant difference (VHD, HD) between 10% ethanol and each of the other testing media on day 7 and day 28, while there were a significant difference between 10% ethanol and each of 3% acetic acid, corn oil and 1% citric acid on day 21(Table 3–10, Table 3–11 and Figure 3–5).

The LSD (least significant difference) test result showed that there were a significant difference between the weight of the appliances that were immersed in corn oil and other testing media on day 14 and day 28, also there were a marked significant difference between corn oil and each of distilled water, artificial saliva, 10% ethanol an 50% ethanol on day 21 (Table 3–10, Table 3–11 and Figure 3–5).

Table (3–8) Descriptive statistic of weight factor for different test period and in each storage media

Dependent Variable	Groups	Mean	S D	Min.	Max.
Weight – zero time	Artificial saliva	0.994	0.007	0.985	1.010
	Distilled water	0.997	0.012	0.978	1.015
	10% Ethanol	0.992	0.012	0.972	1.007
	50% Ethanol	0.998	0.010	0.993	1.018
	3% Acetic acid	0.995	0.009	0.983	1.006
	Corn oil	0.997	0.009	0.986	1.017
	1% Citric acid	0.997	0.013	0.975	1.016
Weight-7days	Artificial saliva	0.998	0.008	0.988	1.013
	Distilled water	0.999	0.012	0.978	1.020
	10% Ethanol	1.022	0.022	0.997	1.075
	50% Ethanol	1.005	0.009	0.992	1.019
	3% Acetic acid	0.997	0.010	0.984	1.014
	Corn oil	1.008	0.006	1.002	1.020
	1% Citric acid	1.001	0.014	0.977	1.020
Weight-14days	Artificial saliva	0.998	0.008	0.987	1.013
	Distilled water	1.006	0.015	0.981	1.029
	10% Ethanol	0.996	0.013	0.976	1.016
	50% Ethanol	1.005	0.009	0.993	1.018
	3% Acetic acid	1.004	0.011	0.983	1.021
	Corn oil	1.017	0.014	1.000	1.049
	1% Citric acid	1.004	0.014	0.980	1.025
Weight-21days	Artificial saliva	1.000	0.009	0.988	1.016
	Distilled water	1.002	0.012	0.984	1.020
	10% Ethanol	0.993	0.012	0.974	1.009
	50% Ethanol	1.005	0.009	0.993	1.020
	3% Acetic acid	1.007	0.015	0.986	1.032
	Corn oil	1.017	0.009	1.005	1.032
	1% Citric acid	1.013	0.020	0.997	1.056
Weight-28days	Artificial saliva	1.004	0.013	0.988	1.033
	Distilled water	1.003	0.014	0.985	1.025
	10% Ethanol	1.019	0.011	0.999	1.031
	50% Ethanol	1.007	0.009	0.996	1.021
	3% Acetic acid	1.000	0.011	0.984	1.012
	Corn oil	1.025	0.018	1.005	1.054
	1% Citric acid	1.006	0.011	0.988	1.022

Table (3–9) Difference in the weight between two successive time interval at different storage periods (ANOVA test, LSD test, p– values)

Media	ANOVA (d.f. = 49)		LSD (P level)			
	F	p	0-7	7-14	14-21	21-28
Artificial Saliva	1.375	0.258	0.363	0.952	0.773	0.304
DW	0.699	0.597	0.761	0.247	0.496	0.774
10% Ethanol	10.180	0.000	0.000	0.000	0.677	0.000
50% Ethanol	1.369	0.260	0.091	0.861	0.973	0.554
3% Acetic Acid	1.870	0.132	0.649	0.211	0.501	0.150
Corn Oil	7.770	0.000	0.069	0.095	0.940	0.140
1% Citric Acid	1.638	0.181	0.531	0.673	0.179	0.322

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

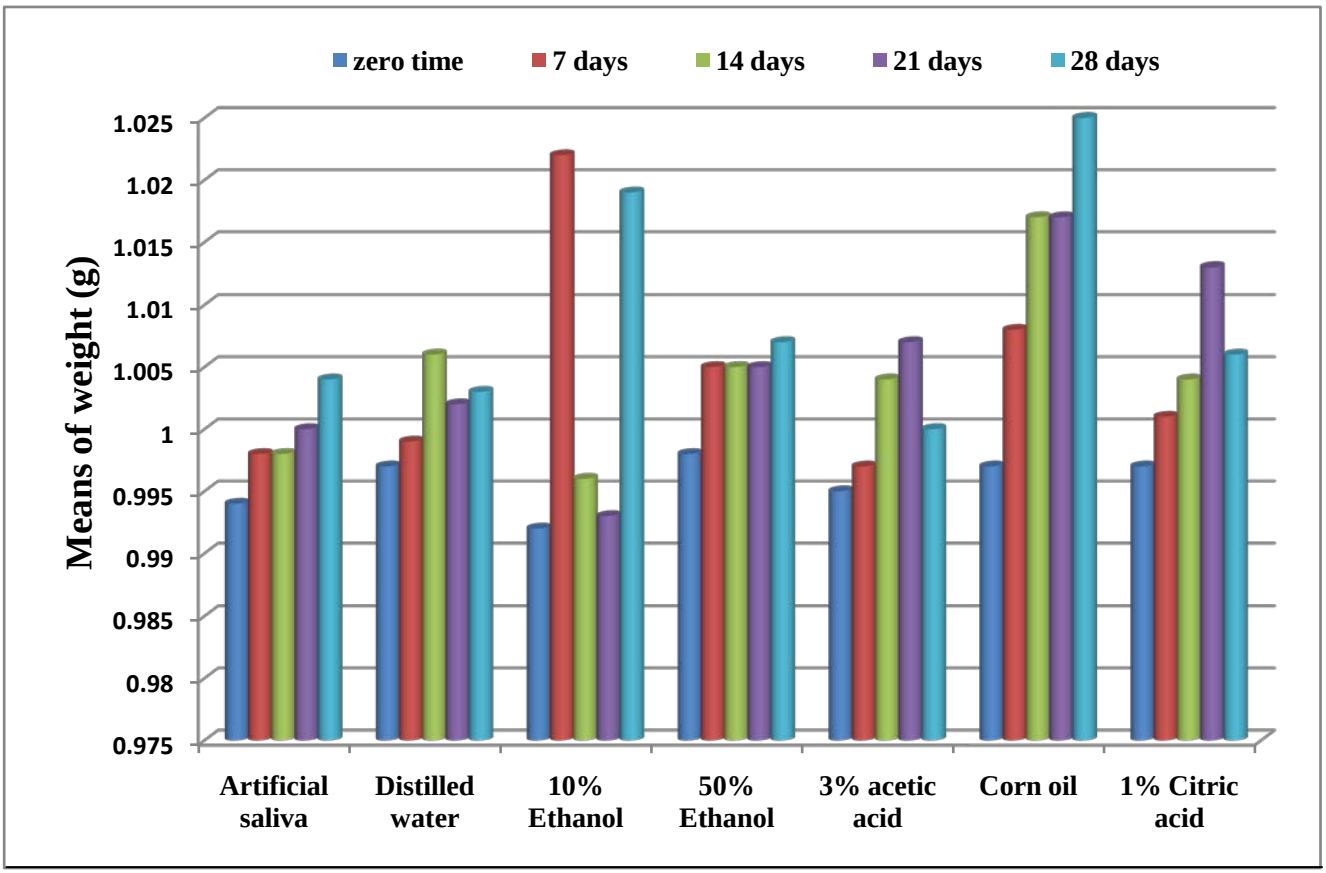

Figure (3–5) Mean distribution of Weight factor in the related periods for different studied group's Media

Table (3–10) Difference in the weight in different storage media during each storage period (ANOVA test, p– values)

Time	F	p
0	0.465	0.831
7	4.783	0.000
14	2.892	0.015
21	4.000	0.002
28	5.384	0.000

Table (3–11) Difference in the weight in different storage media during each storage period (LSD test, p– values)

Media		LSD				
		0	7	14	21	28
Artificial Saliva	DW	0.551	0.887	0.185	0.705	0.941
Artificial Saliva	10% Ethanol	0.602	0.000	0.686	0.285	0.010
Artificial Saliva	50% Ethanol	0.407	0.204	0.258	0.366	0.555
Artificial Saliva	3% Acetic Acid	0.902	0.879	0.333	0.187	0.471
Artificial Saliva	Corn Oil	0.509	0.098	0.001	0.003	0.000
Artificial Saliva	1% Citric Acid	0.548	0.577	0.305	0.023	0.653
DW	10% Ethanol	0.265	0.000	0.086	0.149	0.008
DW	50% Ethanol	0.815	0.258	0.843	0.598	0.506
DW	3% Acetic Acid	0.636	0.768	0.717	0.344	0.518
DW	Corn Oil	0.949	0.130	0.047	0.009	0.000
DW	1% Citric Acid	0.997	0.677	0.761	0.055	0.601
10% Ethanol	50% Ethanol	0.179	0.004	0.127	0.051	0.042
10% Ethanol	3% Acetic Acid	0.519	0.000	0.172	0.019	0.001
10% Ethanol	Corn Oil	0.239	0.012	0.000	0.000	0.266
10% Ethanol	1% Citric Acid	0.263	0.000	0.155	0.001	0.030
50% Ethanol	3% Acetic Acid	0.480	0.155	0.870	0.674	0.192
50% Ethanol	Corn Oil	0.865	0.695	0.030	0.033	0.002
50% Ethanol	1% Citric Acid	0.818	0.472	0.916	0.158	0.887
3% Acetic Acid	Corn Oil	0.591	0.072	0.020	0.083	0.000
3% Acetic Acid	1% Citric Acid	0.633	0.478	0.954	0.319	0.244
Corn Oil	1% Citric Acid	0.952	0.268	0.023	0.453	0.001

P>0.05 NS Non – significant
0.01≥ P>0.001 HS Highly significant
P≤ 0.001 VHS very highly significant

3.3 Microscopical Examinations

The examination of different parts in the new components of fixed appliances, which are cleaned and dried, reveals smooth fibrous structure of stainless steel archwire and ligature wire. Orthodontic brackets examined extensively demonstrating mainly a smooth and fibrous structure of both brackets slot and wings, on the other hand small manufacturing defects can be seen in different parts of orthodontic brackets. Molar bands and tubes also have smooth fibrous surface except for the welded sites in which the grain growth was very clear (Figure 3–6, Figure 3–7, Figure 3–8, Figure 3–9, Figure 3–10, Figure 3–11, Figure 3–12).

At the end of 28 days immersion period, the most common types of corrosion were pitting, and crevice corrosion. The pits appeared as circular or semicircular area (single or in groups), while the crevice regions were presented with finger like projection which are comprised of the microscopic pits sometimes.

By examination of the archwire, these corroded areas can be seen only on the wire at the bracket slot-archwire interface and the part that pass through the molar tube. The archwire parts between the brackets remain intact in most of the cases, the archwire that were immersed in 3% acetic acid and 1% citric acid exhibited more crevice and larger number of pitting corrosion when compared with other groups (Figure 3–6, Figure 3–7). The ligature wires were less subjected to any type of corrosion (Figure 3–8).

The brackets slot were subjected to small areas of crevice corrosion and only minor pitting corrosion occur (Figure 3–9), the wing of the bracket were subjected to pitting and crevice corrosion, the smallest amount of corrosion could be seen in appliances immersed in corn oil, and larger size of crevice and pitting corrosion could be seen in appliances immersed in 10% ethanol, 50% ethanol, 3% acetic acid, 1% citric acid (Figure 3–10).The base of the bracket that were immersed in 3% acetic acid and 1% citric acid showed the most progressive corrosion than other groups (Figure 3–11).

The molar bands were affected at the welded areas expected to be an inter-granular corrosion, and at the molar tube area with both crevice and pitting corrosion, the less amount of corrosion could be seen in appliances immersed in corn oil (Figure 3–12).

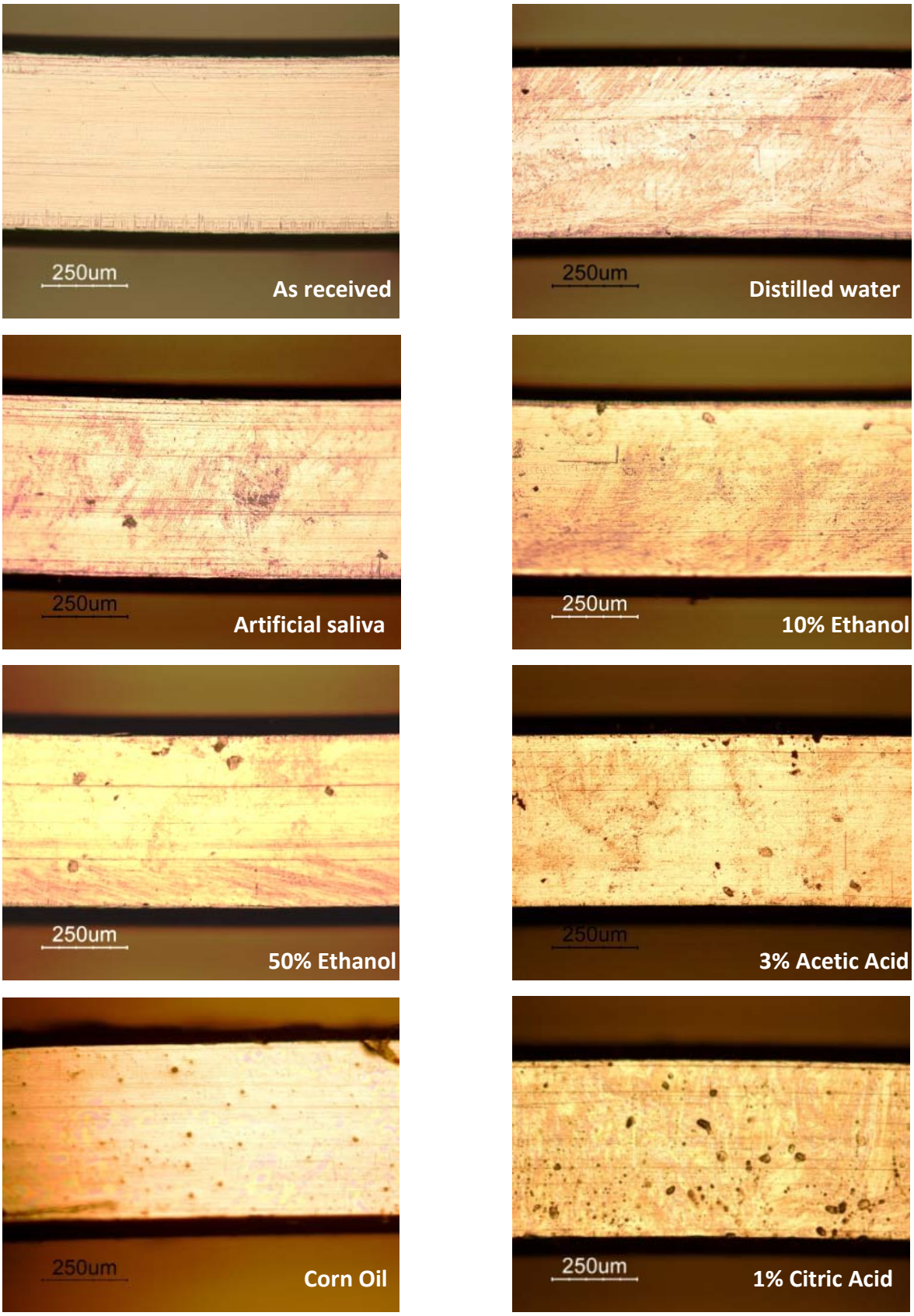

Figure (3– 6) Surface of stainless steel archwire (at the bracket slot - archwire interface area) before and after immersion in each of the testing media

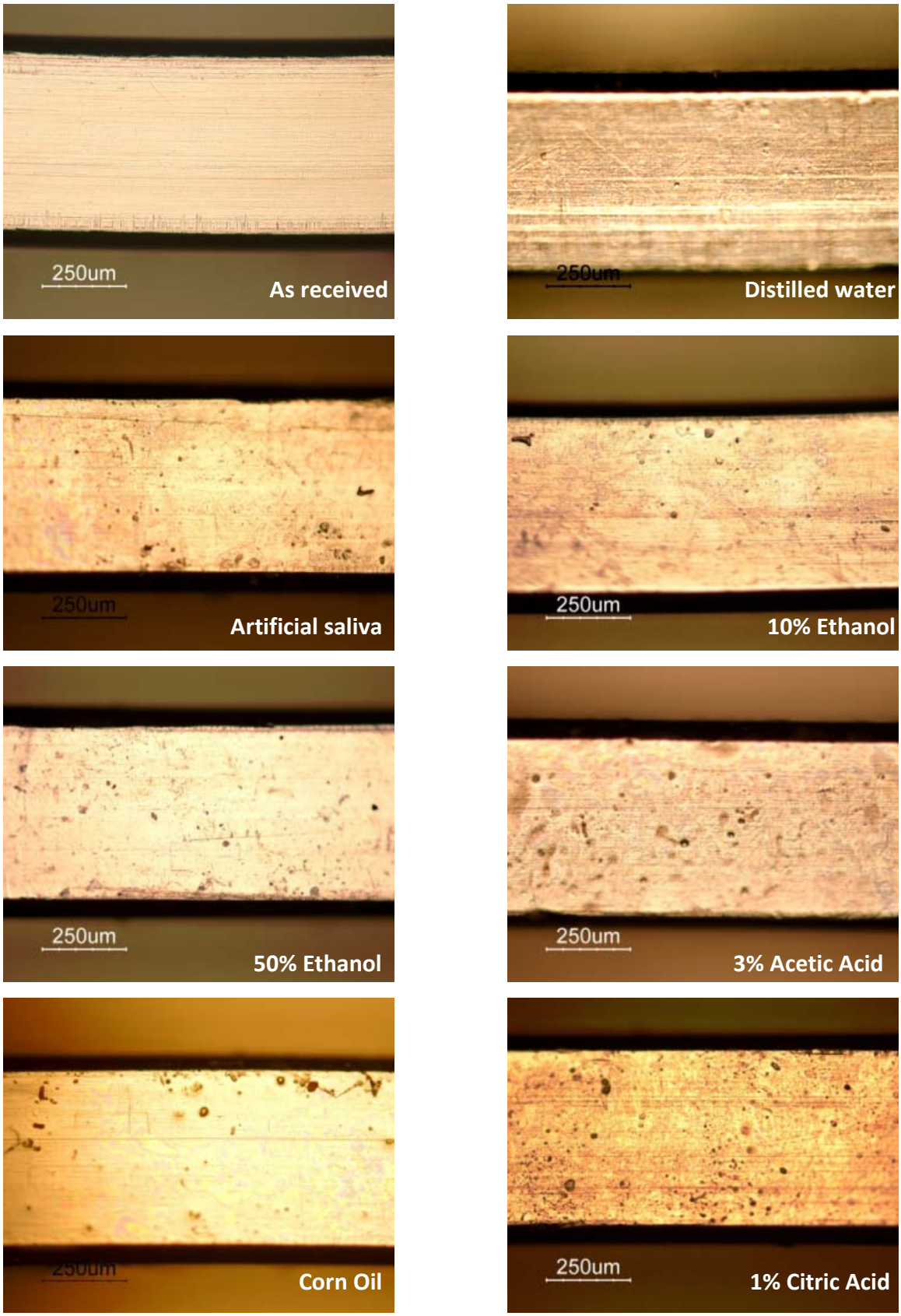

Figure (3– 7) Surface of stainless steel archwire (Wire section that pass through the molar band tube) before and after immersion in each of the testing media

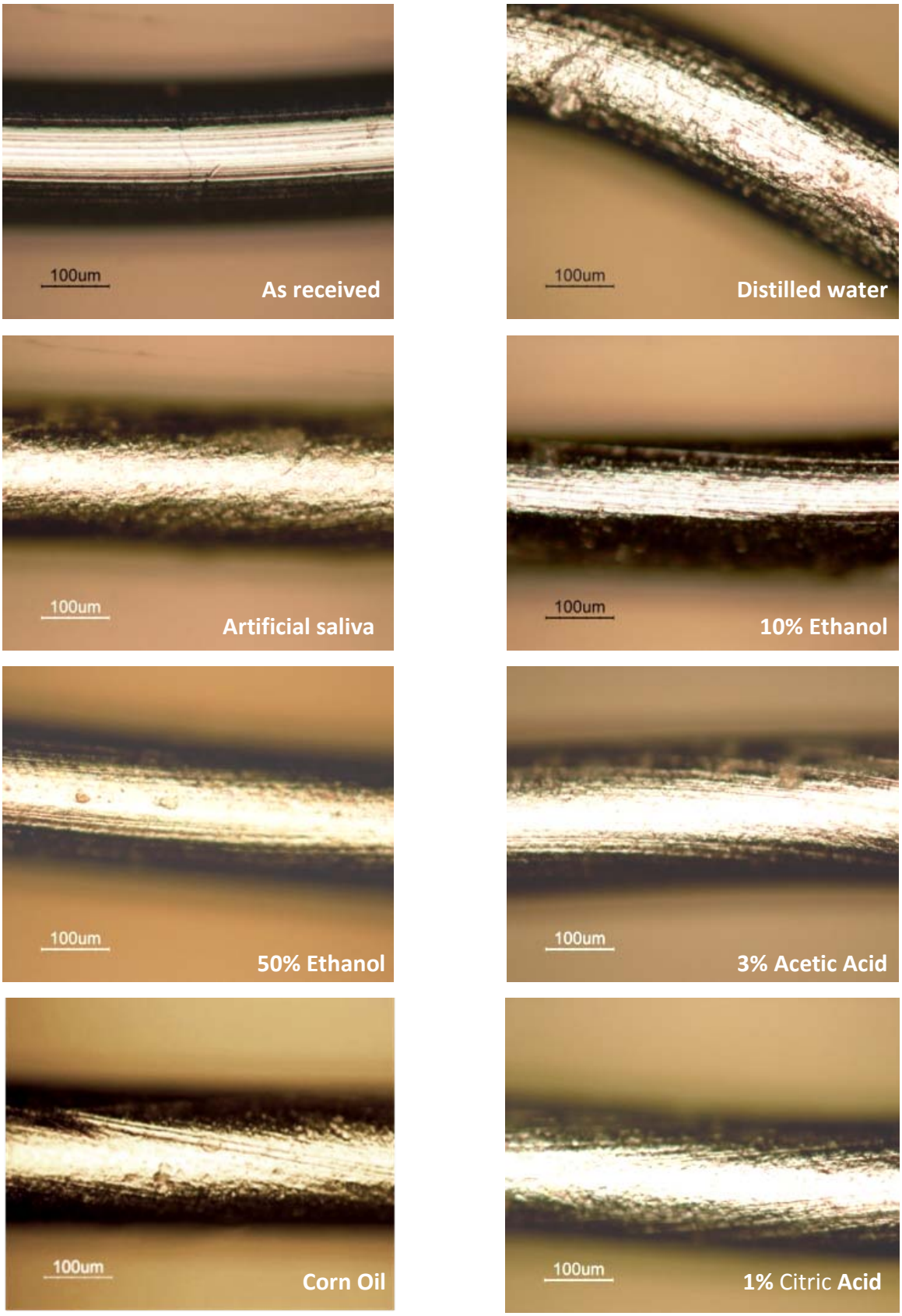

Figure (3– 8) Surface of ligature wire before and after immersion in each of the testing media

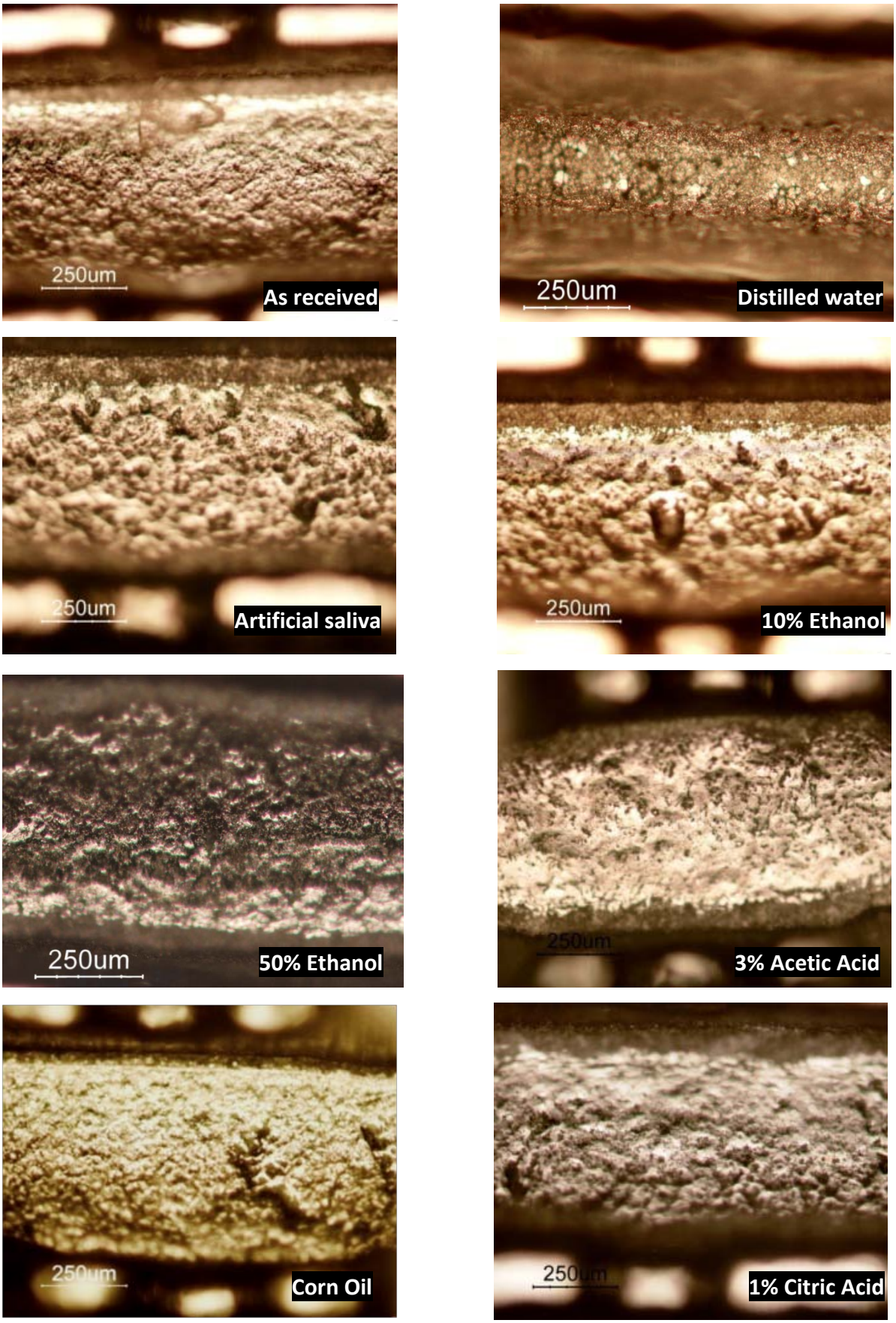

Figure (3– 9) Slot of stainless steel bracket before and after immersion in each of the testing media

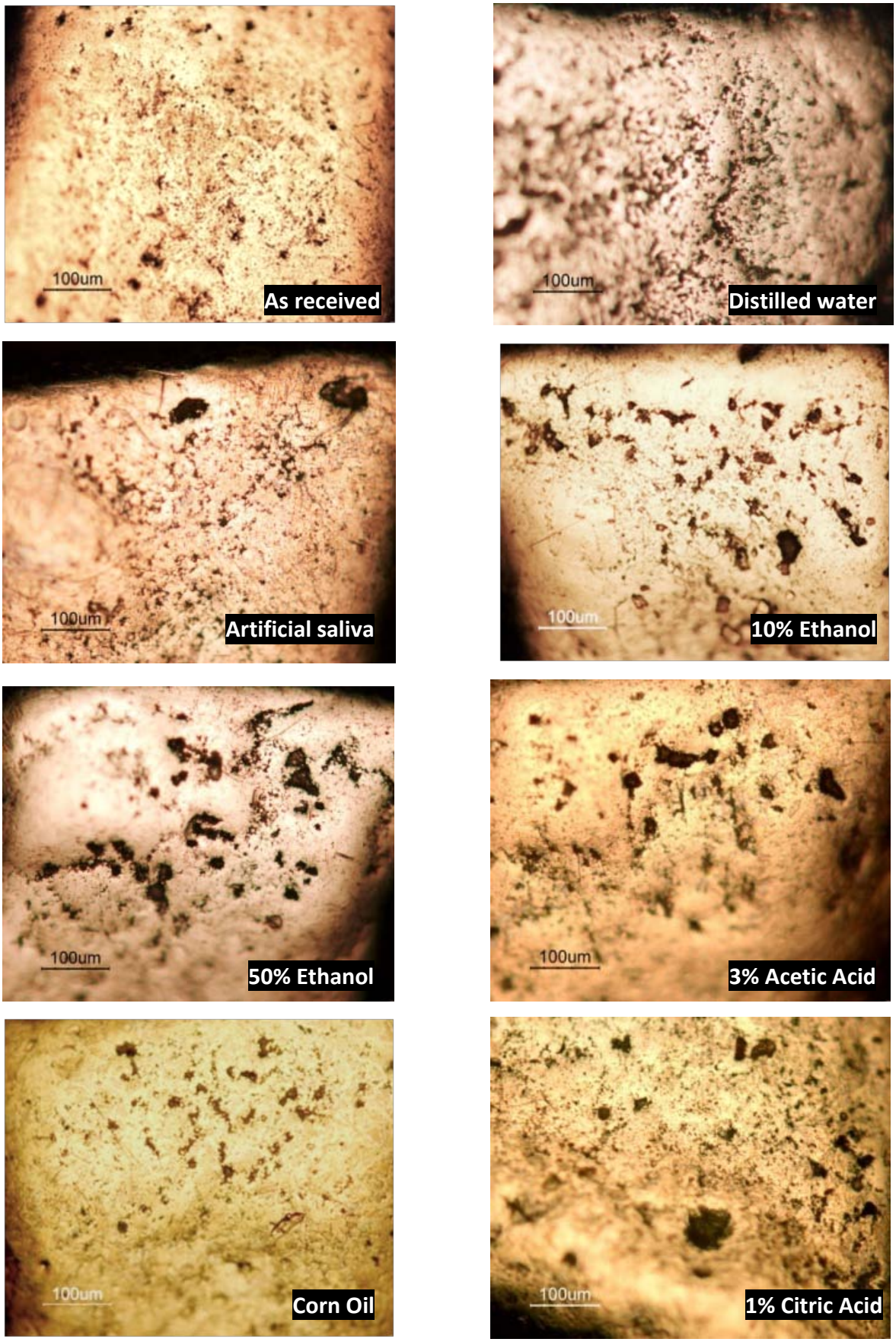

Figure (3– 10) Wing of stainless steel bracket before and after immersion in each of the testing media

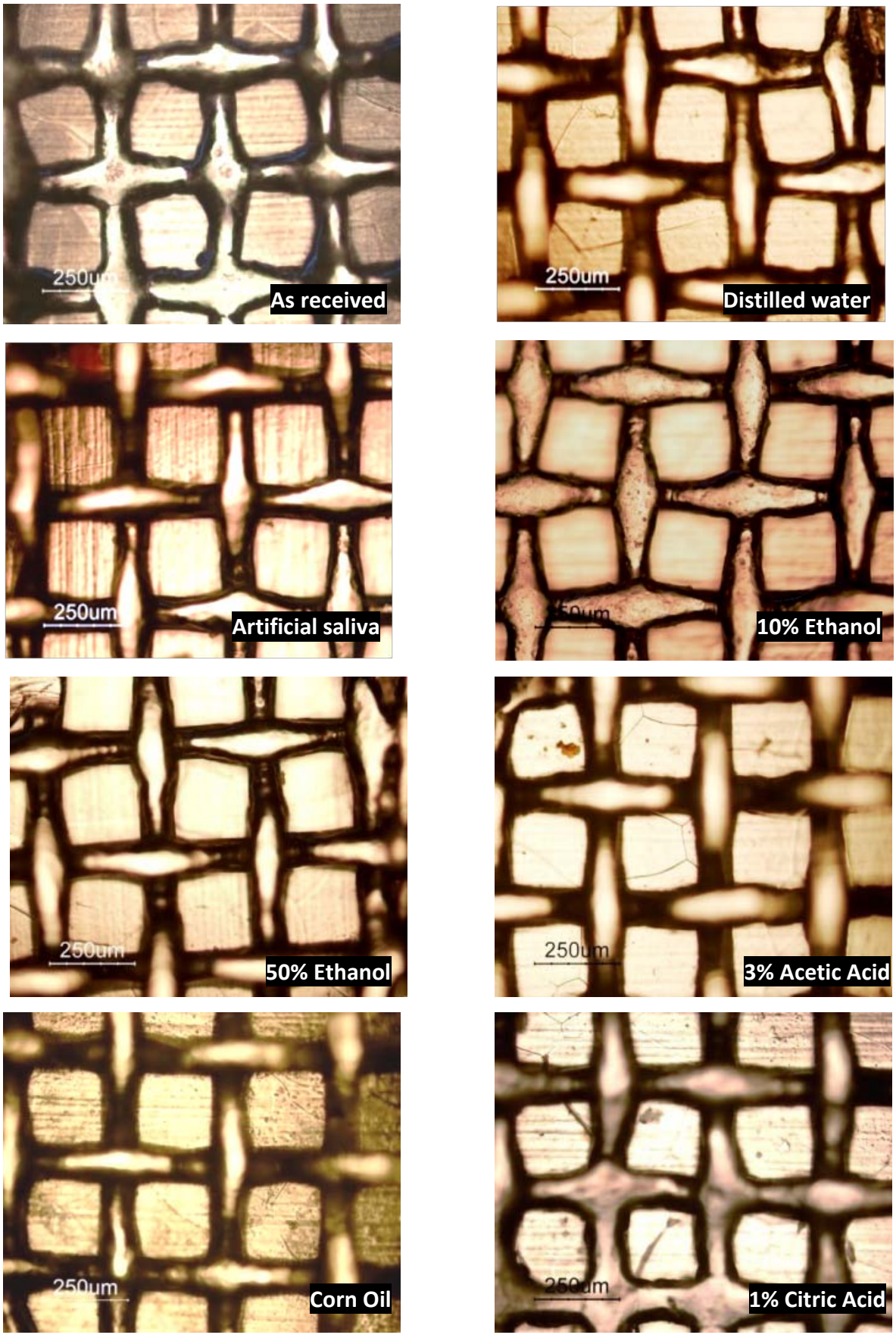

Figure (3– 11) Base of stainless steel bracket before and after immersion in each of the testing media

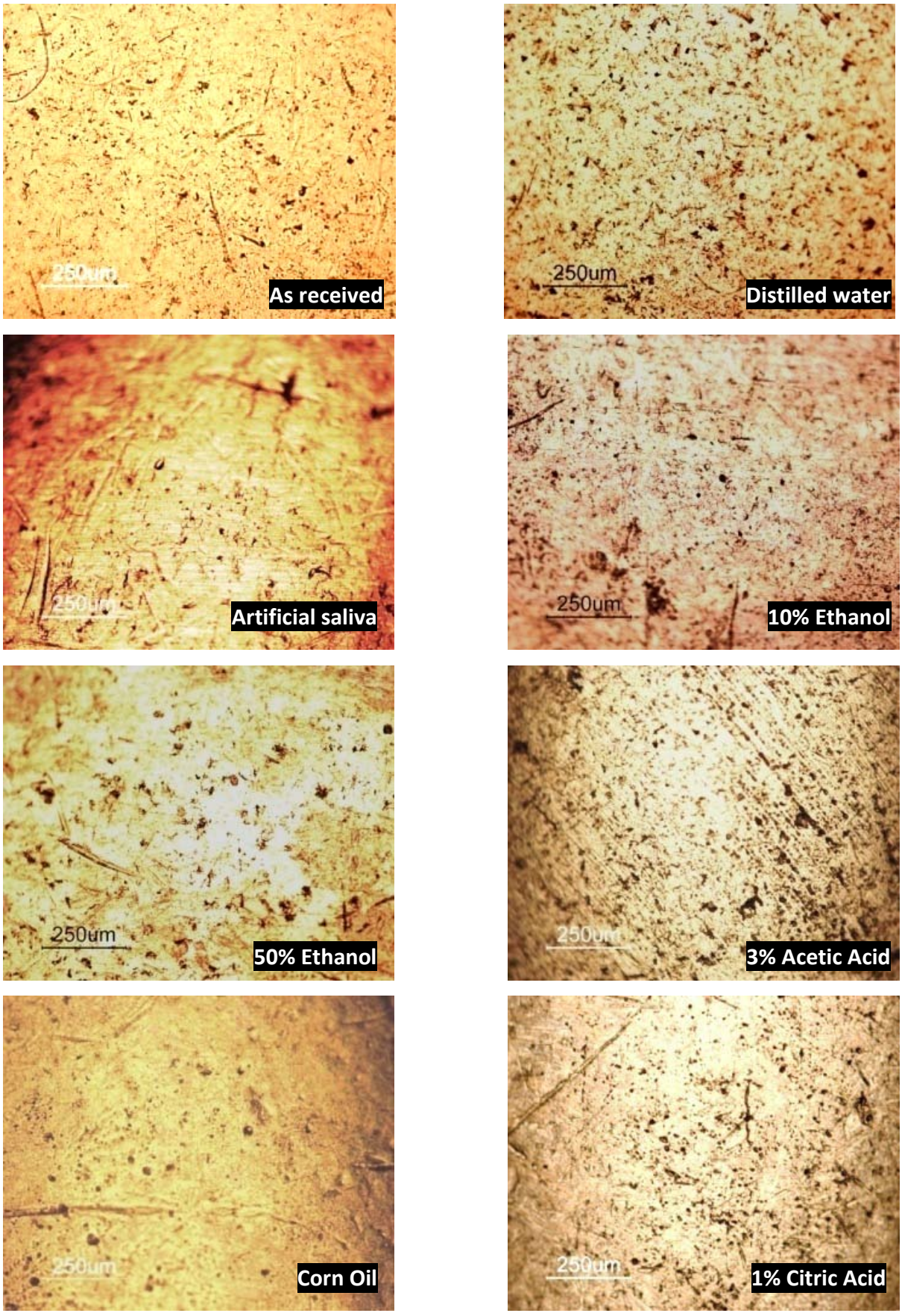

Figure (3– 12) Surface of stainless steel molar band before and after immersion in each of the testing media

Chapter Four

Discussion

Chapter Four
Discussion

4.1 Corrosion Studies

The corrosion rate of metals and alloys can be determined using chemical methods. The chemical methods for finding the corrosion tendency are as follow ***(Kedici et al, 1998)***:

I. By the mass loss of corroding metal and alloy.
II. By the amount of corrosion products in the corrosive media.
III. From the amount of gas produced during the corrosive reaction.

It is important to note that the oral environment is very conducive to the formation of corrosion products, because the mouth is moist and continually subjected to fluctuations in temperature. Saliva acts as an electrolyte, which can cause corrosion, and also as a medium for chemical reactions between metals. Foods and drinks cause transitory, but important and wide, variations in the chemistry of the environment, as the ingested food and liquids have wide ranges of PH. Acids are liberated during breakdown of foodstuffs. This food debris often adheres tenaciously to the metallic restoration providing a localized condition that is extremely conducive to an accelerated reaction between the oral media and the metal or alloy ***(Oh and Kim, 2005; Duffó and Farina, 2009)***.

It is virtually impossible to design an in vitro experimental model that mimics the real-life situation. This is why we chose to assay ion release in extreme conditions which were much more favorable to the corrosion of the orthodontic appliances than those normally found in the oral cavity ***(Staffolani et al, 1999)***.

The corrosion studies have demonstrated that the level of metal release from simulated fixed orthodontic appliances extends over one week and that all release is completed within four weeks ***(Park and Shearer, 1983; Barrett et al, 1993; Hwang et al, 2001)***. Considering this factor and by using chemical methods,

we studied the corrosion through 28 days at 37° which represent the normal temperature of the oral cavity.

4.2 The Amount of Ion Release

4.2.1 The Effect of Time

Time was a significant factor in this study, as seen from table (3–1), (3–2), and figure (3–1), (3–2), (3–3), (3–4), there was a significant difference in the release of chromium, nickel, copper and iron ion between the successive time interval in all the studied groups. Our results showed the greatest release of ions during the first week of the study, and gradual decline thereafter. This cannot be ascribed to saturation of the solution with metal ions because the solution was changed for every time period. Such decrease of ions release can be explained by natural ability of stainless steel of passivation, by the formation of a film of chromium oxides that prevents corrosion in many environments, when this protective film is formed, the corrosion rate will be reduced ***(Lopez et al, 2002; Juraga et al, 2007)***.

For the chromium and nickel ions, as seen in table (3–1), (3–2), and figure (3–1), (3–2), the greatest ion release occur during the first week of this study, then there is a gradual decrease in ion release. This kinetics of ion release coincides with the result of several studies ***(Park and Shearer, 1983; Barrett et al, 1993; Hwang et al., 2001; Hassoon, 2008)***. ***Staffolani et al (1999)*** found that the largest daily ion release was noticed after the first day of exposure to the solution. Also, ***Al- Joboury (2001) and Kuhta et al (2009)*** found that the actual reduction in the release occur after the 7th day reaching several fold smaller values at the end point.

Nickel release was found to be greater than chromium and this can be explained on the basis that nickel has less ability to form a protective layer against the environment as done by chromium so its release was governed by the formation and restoring the Cr-oxide. Otherwise, it is exposed to the environment and continues in leaching the nickel in large quantities. The release of nickel is not necessarily proportional to the nickel content ***(Kerosuo et al, 1995)***.

The result clearly showed that the iron ion was released at the higher level within the first week of this study, then progressively diminished in all the studied groups table(3–1), (3–2) and figure (3–4). This result was in consistent with the finding of ***Hwang et al (2001)*** and by ***Kuhta et al (2009)***, and in contrast to ***Hassoon (2008)***, whose result showed that the release of iron ions increased as the immersion time progressed.

The higher level of the release of iron ion compared with chromium and nickel seems to comply with the abundance of the elements in the alloy composition. This can be explained as iron is the major component of the stainless steel, iron oxide (Fe_2O_3) is not adherent, and because of the low amount of chromium comparing with the high amount of iron, it may not enough to produce such strongly adherent passivating layer of chromium oxide on the surface to prevent corrosion of iron ***(Elshahawy et al, 2009a)***.

The finding of this study showed that large amount of copper ion was released for two weeks with the higher level in the first 7 days period, then there was a decreased in the level of released ion table(3–1), (3–2) and figure (3–3). This result comes in accordance with ***Kuhta et al (2009)*** who reported that the greatest release of ions occur during the first 7 days and a gradual decline thereafter.

Most metal brackets are not cast or fabricated in one piece. Instead, the wing and the base portion of the metal bracket are connected by solder, which is primarily comprised of Cu ***(Huang et al, 2004)***. The presence of soldered joints however, has exacerbated corrosion susceptibility since they have a tendency to emit electro galvanic currents with saliva and consequently release metal ions ***(Hwang et al, 2001; Vahed et al, 2007)***. According to ***Zinelis et al (2004)***, Ag- based soldering alloy introduce a galvanic couple with stainless steel alloys, inducing release of metallic ion with Cu, the elements most easily leached out from silver soldering alloys. This was the reason for the higher levels of copper ion release compared with other ions.

4.2.2 The Effect of Food Simulants

This study emphasizes the importance of the immersed solution on the release of metal ions from fixed orthodontic appliances. Although the quantities of released metal ions measured in this study and other similar studies cannot be directly applied to in vivo conditions, but they are useful for relative comparisons and for determination of the effect of each individual solution on ions release without the influence of external factors.

The result showed a significant difference between studied groups in the amount of ions released, table (3–1), (3–3) and figure (3–1),(3–2), (3–3), (3–4).
The higher levels of ions were released in 1% citric acid group (PH= 2.7 $\pm$ 0.2) and in 3% acetic acid group (PH=2.9$\pm$ 0.2) followed by 10% ethanol (PH= 5.5$\pm$ 0.2) and 50% ethanol (PH=5.7 $\pm$ 0.2) groups, and then the artificial saliva group (PH=7$\pm$ 0.2). The least amount of ions was released in distilled water (PH=8$\pm$ 0.2) and corn oil groups. In the first week of this study which represents the higher level of ions release, the LSD test showed that there was no significant difference between 1% citric acid and 3% acetic acid groups, and between 10% ethanol and 50% ethanol groups, and between corn oil and distilled water groups for most of the measured ions, table (3–4), (3–5) (3–6), (3–7).

So the levels of released ions were gradually increased with decreasing solution PH. These results agree with the finding of ***Staffolani et al (1999) and Kuhta et al (2009)***. Also this finding also coming with the result of ***Huang et al (2004), Okazaki and Gotoh (2008), Elshahawy et al (2009a)***.This occurred because the acidic condition provide a reducing environment in which the stainless steel oxide film required for corrosion resistance is less stable ***(Sfondrini et al, 2009)***.While our finding disagree with the result of ***Duffó and Farina (2009)*** who showed that the aggressiveness of the different liquids is independent on the PH of the solution.

The corrosion rate of iron and copper increase in the acidic media, conversely alloys containing chromium and nickel proved to be more resistant;

however, small variation in their compositions affect this corrosion resistance ***(Kedici et al, 1998)***. The quantity of Cr released gradually increased with decreasing PH (PH ≤ 6) and botto med out at PH 6 ***(Okazaki and Gotoh, 2008)***. This explained the non significant difference in the release of chromium ion between artificial saliva and distilled water groups at all time periods, and the less significant difference between artificial saliva and distilled water groups at all time periods in the amount of released nickel ion, but we found a significant difference in the release of copper and iron ion between these two groups.

In this study the least amount of chromium, nickel, copper ion release was demonstrated in the corn oil group. This could be explained by the formation of a thin layer of an 'oily phase' on the metal surface. The adsorbed oily phase is considered to facilitate the cathodic partial reaction (O_2 reduction), because of the higher solubility of oxygen in oil, whereas it inhibits the anodic dissolution reaction of the steel due to low solubility of ionic species in the oil ***(Becerra et al, 2000; Tian and Cheng, 2008)***. The oil adsorption to the metal surface can be a result of physical adsorption, chemical adsorption, or chemical reaction ***(Eliezer et al, 2008)***, so instead of reacting with or removing an active corrosive species, the filming corrosion inhibitor function by strong adsorption and decrease the attack by creating a barrier between the metal and their environment ***(Al-Juhni and Newby, 2006; Rosliza and Wan nik, 2010)***.When the steel is pre-oxidized, a layer of pre-oxide film of $Fe(OH)_2$ is formed , the corrosion rate of steel increase by partial dissolution of $Fe(OH)_2$ ***(Tian and Cheng, 2008)***, and this explain the higher level of iron ion when compared to other ions in corn oil.

The higher value of chromium, nickel, and iron ions release for a four week period was observed in 1% citric acid group. By doubling this amount to represent maxillary and mandibuler arch, the result was 1.13 µg/ml for chromium ion, 2.522 µg/ml for nickel ion, 3.578 µg/ml for iron ion. The higher value of cupper ion was 63.48 µg/ml, which was measured in 3% acetic acid. All of these ions released were below average daily intake of these ions according to Food and Drug administration

(FDA) and World Health Organization ***(WHO)*** values ***(Appendix 1)***. All previous immersion corrosion tests from ***(Park and shearer, 1983)*** to ***(Kuhta et al, 2009)*** also found that the amount of ion released is below the average daily intake of these ions.

4.3 Weighing the Orthodontic Appliances

Decreasing the weight of the appliance may indicate released of ions, and the increase of weight was an indication for adsorption of ion from the surrounding media during the formation of oxide layer.

The result of this study as seen table (3–8), (3–9) and figure (3–9), showed that the weight of orthodontic sets in all groups was increased after immersion in testing media; this may be explained by adsorption of ions from immersed media during the process of oxide layer formation. The result of LSD test showed no significant difference on the weight of orthodontic sets between the successive time intervals in the studied groups except for 10% ethanol group. The greater increase in the weight of the appliances after 7 days can be explained by formation of oxide layer, so the release of ions as seen in table (3–1) was greatly reduced after 7 days especially for copper ion.

There was no significant difference in the weight of the appliances before immersion in solutions (at zero time), table (3–10), (3–11). After immersion in the testing solution, the results showed a significant difference between 10% ethanol and corn oil groups and other testing media. These findings for the weight difference of 10% ethanol with other groups occur due to larger increase in the weight of 10% ethanol after immersion compared to other groups, while the difference between the weights of appliances immersed in corn oil compared to other groups could be explained by the formation of thin layer of an ‘oily phase’ on the metal surface ***(Becerra et al, 2000; Tian and Cheng, 2008)***, which increase the weight of these appliances more than other groups.

There was a decrease in the weight of the appliances immersed in the 3% acetic acid and 1% citric acid after 21 day of immersion, this reduction could occur because of distraction of oxide layer and release of some ions. This difference

occurred because the acidic condition provide a reducing environment in which the stainless steel oxide film required for corrosion resistance is less stable ***(Sfondrini et al, 2009)***. The weight of all appliances was increased at the end of the study when compared to zero line, which give indication for absorption of ions during oxide layer formation.

4.4 Microscopical Examinations

The microscopical examination showed the presence of some surface roughness in parts of orthodontic appliances as received from the manufacture, these defect could be seen mostly in the bracket slot and wings, also small defects could be seen in molar band especially near the welded area. These surface defects produced during the manufacturing processes. Such manufacturing defect was observed also by ***(Lin et al, 2006***), who observed these defects in many types of stainless steel orthodontic brackets. The most common types of corrosion which could be seen in different parts of orthodontic appliances were pitting and crevice corrosion ***(Kim and Johnson 1999; Al-Joboury, 2001; Juraga et al, 2007; Hassoon, 2008; Luft et al, 2009)***.

Pitting corrosion was noted over the most component of the fixed appliances, the morphology of pits is similar to those obtained by ***Sutow and Milne (1985)***. The oxide layer couldn't be maintained in the presence of aggressive ions like (Cl). This ion competes with the oxidizing species and becomes incorporated into the passive film, thereby reducing the resistivity of the oxide layer. When the metal or alloy dissolution is high at one particular point, chloride ions will migrate to this point, this change tend to produce condition favorable for further rapid dissolution at this point, this point will behave anodically in comparison to other protected surface layer which behaves cathodically***(Uhlig, 1980;Fontana and Greene, 1982; Anusavice, 1996)***.

Crevice corrosion is obvious in different parts of fixed orthodontic appliances including the bracket slot, the archwire at the bracket slot-archwire interface, wire part which passes through the molar band tube, and the molar band

tube. The morphology of the crevices agrees with those mentioned by ***(Sutow and Milne, 1985)***. The previous sites represent harbors of stagnant solution and under the deposit area, oxygen depletion takes place. After oxygen is depleted, no further oxygen reduction occur, although the dissolution of the metal continues, this tend to produce an excess of positive charges in the solution that is balanced by migration of chloride ions to the crevice. This result increased the concentration of metal-chloride within the crevice. As the corrosion within the crevices increased, the rate of oxygen reduction on the adjacent surface increases and this cathodically protects the external surface, thus during crevice corrosion, the attack is localized within the shielded areas while the remaining surface suffers little or no corrosion ***(Jones, 1996)***.

The presence of rust colored precipitate at the weld spots and welded areas was indicative for inter-granular corrosion. Such type of corrosion caused usually due to the precipitation of chromium carbides at the grain boundaries at high temperature, when chromium combines with carbon in this manner, its passivating qualities were lost. As a consequence, the corrosion resistance reduced because that portion of the grain adjacent to the grain boundaries was depleted from chromium. Additionally, impurities are mainly present at the grain boundaries areas which increase its susceptibility to corrosion. This type of corrosion in the fixed orthodontic appliance was denoted in many experiments ***(Maijer and Smith, 1986; Grimsdottir et al, 1992; Barrett et al, 1993; Platt et al, 1997)***.

Greater extended areas of crevices and pits can be seen in the parts of fixed orthodontic appliances which immersed in acidic solutions. Other groups were affected by crevices and pits also, but for less extent than those in acidic solutions, This occur because the acidic condition provide a reducing environment in which the stainless steel oxide film required for corrosion resistance is less stable ***(Sfondrini et al, 2009)***.

4.5 Clinical Considerations

Most orthodontic bands, brackets, and archwires were considered to be a source of human exposure to different elements used in manufacturing the components of fixed orthodontic appliances. A static immersion test combined with released ions measurement seems to deliver information of high relevance for clinical application and biocompatibility. In this study, the amount of chromium, nickel, copper, and iron ions released from orthodontic appliances were measured, it was found that the total released amounts of chromium, nickel, copper, and iron ions at the end of this study from the studied groups were less than recommended daily intake of these elements which is for chromium 120 μg, for nickel < 1 mg, for copper 2 mg, and for iron 15 mg based on Food and Drug Administration **(FDA)** values, and the World Health Organization **(WHO)** values ***(Appendix 1)***. Thus, the release of metal ions from these reused materials may have no biological effects.

In addition to the aspect of biocompatibility the corrosion might have clinically relevant effects on the surface microstructure. This change of the surface roughness might result in higher friction during the sliding of the bracket along the archwire.

As organic acids facilitate the release of metal ions, so the acidic nature of accumulated dental plaque and production of acids as the major result for the metabolism of oral microorganism will provide aggressive nature for corrosion. So a good oral hygiene could be an important factor in reducing corrosive events.

Chapter Five

Conclusion
and
Suggestion for further studies

Chapter Five
Conclusions and Suggestions for Further Studies

5.1 Conclusions

I. Both the time of exposure to the solutions and the solution types was significantly influenced ions release.

II. The greatest amount of ions release occurred in the first week, followed by a progressive decline in ion release till the end of the test period in all storage media, which supports the role of oxide layers in slowing down a corrosive process on the metal surface.

III. The release of ions was increased with decreasing the PH of the solutions, and this is reflected by the marked ion release in the citric acid and acetic acid solutions, while it's decreased in corn oil, due to the formation of oily phase on metal surface which acting as corrosive inhibitor.

IV. The total released amounts of metals ions released from orthodontic appliances used in this study were less than the amounts ingested during daily food intake. Thus, the release of metal ions from these materials has no biological effects.

V. Weighing the orthodontic samples revealed that there were increases in the weight of the orthodontic appliances in all studied groups at the end of the study.

VI. The microscopical examination showed that there were crevice and pitting corrosion of most parts of orthodontic appliance surfaces with more severe in those immersed in acidic solutions, and the least amount of corrosion seen in corn oil.

VII. The corroded areas seen mostly on the wire at the bracket slot-archwire interface and the part that pass through the molar tube. The molar bands were affected wholly at the welded areas reflecting the inter-granular corrosion.

5.2 Suggestions for Further Studies

A. It is highly suggested to study the effect of food simulant on corrosion of orthodontic appliances in dynamic situation.

B. Use electrochemical test to study the effect of food simulant on the corrosion of orthodontic appliances.

C. Study the effect of food simulant on the corrosion of orthodontic appliances with different material such as nickel titanium.

D. Study the effect of food simulant on the physical properties of plastic, ceramic and composite brackets.

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APPENDIX 1

Recommended daily intakes of minerals

Minerals	Recommended daily intake	Over dosage
Boron	**< 20 mg**	**No information found**
Calcium	**1000 mg**	**Doses larger than 1500 mg may cause stomach problems for sensitive individuals**
Chlorine	**3400 mg (in chloride form)**	**No information found**
Chromium	**120 µg**	**Doses larger than 200 µg are toxic and may cause concentration problems and fainting**
Copper	**2 mg**	**As little as 10 mg of copper can have a toxic effect**
Fluorine	**3,5 mg**	**No information found**
Iodine	**150 µg**	**No information found**
Iron	**15 mg**	**Doses larger than 20 mg may cause stomach upset, constipation and blackened stools**
Magnesium	**350 mg**	**Doses larger than 400 mg may cause stomach problems and diarrhea**
Manganese	**5 mg**	**Excess manganese may hinder iron adsorption**
Molybdenum	**75 µg**	**Doses larger than 200 µg may cause kidney problems and copper deficiencies**
Nickel	**< 1 mg**	**Products containing nickel may cause skin rash in case of allergies**
Phosphorus	**1000 mg**	**Contradiction: the FDA states that doses larger than 250 mg may cause stomach problems for sensitive individuals**
Potassium	**3500 mg**	**Large doses may cause stomach upsets, intestinal problems or heart rhythm disorder**
Selenium	**35 µg**	**Doses larger than 200 µg can be toxic**
Sodium	**2400 mg**	**No information found**
Vanadium	**< 1,8 mg**	**No information found**
Zinc	**15 mg**	**Doses larger than 25 mg may cause anemia and copper deficiency**

الخلاصة

تعد اجهزة تقويم الاسنان احد مصادر تعرض الانسان لمختلف العناصر الداخلة في صناعة مكونات هذه الاجهزة . الخواص الفيزيائية والكيميائية للاطعمة والسوائل تستطيع ان تؤثرعلى تأكل هذه الاجهزة. لذا تم اعداد هذه الدراسة المختبرية لتحديد تأثير المواد المحاكية للمنتجات الغذائية الدهنية والحامضية الاكثر شيوعا والسائل الفموي في درجة احتكاك ومقدار تحرر الايونات من الاجهزة التقويمية الثابتة.

في هذه الدراسة تم اعداد سبعين نموذجا من الاجهزة التقويمية الثابتة وتألف كل نموذج من نصف جهاز تقويم ثابت للفك العلوي. تم تقسيم نماذج الاجهزة التقويمية الثابتة الى سبع مجاميع, كل مجموعة تحتوي على عشرة نماذج. ثم تم غمر هذه الاجهزة في احد وسائط التخزين التي تتضمن: اللعاب الصناعي, ماء مقطر,١٠ % ايثانول,٥٠ % ايثانول, ٣% حامض الخليك, زيت الذرة, ١% حامض الستريك. لقد تم غمر هذه الاجهزة في درجة حرارة ٣٧ ° لمدة ٢٨ يوما, ويتم تغيير وسائط التخزين كل ٧ ايام لمنع تشبع المحاليل بالايونات.

في نهاية كل فترة تخزين, تم جمع نماذج من المواد المختبرة لغرض التحليل العناصري, ان قياس ايونات الكروم(Cr), النيكل(Ni), النحاس(Cu), و الحديد(Fe) تم من خلال جهاز المطياف الامتصاص الذري وجهاز المطياف. لقد تم وزن النماذج قبل وبعد كل فترة غمر, فيما تم دراسة توزيع وانواع انماط التاكل بواسطة مجهر ضوئي خاص.

اظهرت نتائج دراسات الغمر ان اعلى كمية من الايونات تحررت خلال ال(٧) الايام الاولى من الدراسة. واظهرت ان اعلى مستوى من الايونات قد تحررت في محاليل ١% حامض الستريك و ٣% حامض الخليك, واقل مستوى من الايونات قد تحررت في محاليل زيت الذرة والماء المقطر.

نتائج الفحص المجهري اظهرت ان التآكل النقري و التآكل الشقي (الصدعي) هي من اكثر انواع التآكل التي يمكن رؤيتها في مختلف اجزاء الجهاز, والتآكل الاكثر حدة يمكن رؤيته في الاجهزة المغمورة في المحاليل الحامضية,واقل نسبة من التآكل يمكن رؤيته في الاجهزة المغمورة في زيت الذرة والماء المقطر.

اظهرت نتائج الوزن للنماذج التقويمية وجود تغير في وزن الاجهزة خلال الدراسة, وكل الاجهزة كان لديها وزن اعلى في نهاية الدراسة.

ان النتائج الكلية تشير الى ان نسبة التآكل للاجهزة التقويمية تزداد مع تناقص الدالة الحامضية(PH) للمحاليل, ووجود الزيت يقلل من نسبة تآكل هذه الاجهزة. ان التآكل يمكنه تغيير خشونة الاسطح مما قد يؤدي الى زيادة الاحتكاك خلال انزلاق الحاصرات على طول اسلاك التقويم. بما ان الحوامض العضوية تسرع تحرر الايونات المعدنية, لذلك فأن صحة الفم يمكن ان يكون عامل مهم في تقليل الاحتكاك

ثاتير المواد المحاكية للغذاء على احتكاك الاجهزةالتقويمية الثابثة

(دراسة مختبرية)

رسالة مقدمة الى مجلس كلية طب الاسنان – جامعة بغداد

كجزء من متطلبات نيل درجة الماجستير في تقويم

الاسنان

مقدمة من قبل

عفاف حميد حسين

بكالوريوس في طب وجراحة الفم والاسنان

باشراف

الاستاذ الدكتور اسامة الملا

بغداد – العراق

ذو الحجة /١٤٣٠ هـ كانون الاول /٢٠٠٩ م