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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF NICKEL AND CHROMIUM RELEASE FROM THREE DIFFERENT METAL BRACKETS IN ARTIFICIAL SALIVA

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ABSTRACT

Background and Objectives: Orthodontic brackets are important part of fixed appliances, which are used to deliver forces from the wires or other power modules to the teeth and will be in the oral environment for long periods of time. The most widely used stainless steel bracket in orthodontics is the austenitic type, containing 18-20% chromium and 8-10% nickel. The combination of chromium and nickel increases the bracket resistance to corrosion. The oral environment is highly conducive to corrosion and pH changes. Metallic orthodontic appliances are known to release metal ions leading to changes in the mechanical properties. Among the metal ions released nickel is the most common cause of metal-induced allergic contact dermatitis and second in frequency is chromium. The objective of this study is to compare and evaluate the rate of release of nickel and chromium ions from stainless steel brackets of three different manufacturers at two different time intervals. **Methods:** 90 standard stainless steel orthodontic brackets of three different manufacturers (Group A-3M ESPE, Group B- JJ Orthodontics, Group C-Modern Orthodontics) were collected. The brackets were divided into three equal groups. Each bracket was incubated at a room temperature of 37°C in individual 30ml plastic containers containing 15ml of artificial saliva at pH 6.7. 2 ml of artificial saliva was taken from each plastic containers for inductively coupled plasma optical emission spectrometric (ICP-OES) evaluation on seventh day and twenty eighth day. The artificial saliva was replaced after each sample collection to avoid saturation with corrosion products. **Result and Discussion:** In all the three groups, Nickel release was more on seventh day. And chromium release was more on twenty eighth day for 3M and Modern. And for JJ Orthodontics, Chromium release was more on seventh day. **Conclusion:** Stainless steel brackets of 3M shows less amount of Nickel ion release on twenty eighth day compared to JJ and Modern. Stainless steel brackets of Modern shows less amount of Chromium ion release compared to 3M and JJ on both days.

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INTRODUCTION

Orthodontic brackets are important part of fixed appliances which are temporarily attached to the teeth during the course of orthodontic treatment. They are used to deliver forces from the wires or other power modules to the teeth. (1)

The bracket is an important element of the appliance because it determines the style of braces and will be in the oral environment for long periods of time. In earlier days, orthodontists made attachments from Gold, Platinum, Iridium and Silver alloys (noble metals and their alloys) which were aesthetically pleasing and corrosion-resistant, but lacked flexibility and tensile strength. Stainless steel is widely used in orthodontics because of its good mechanical properties, strength, good biocompatibility and has good corrosion resistance.

The most widely used type in the orthodontic field is the austenitic type of stainless steel, which is 18-8 containing 18-20% chromium and 8-10% nickel. The combination of chromium and nickel increases bracket resistance to corrosion (2). Orthodontic appliances to correct maligned teeth have been found in Greek, Etruscan and Egyptian artifacts. Pliny the Elder (23-79 AD) was the first to mechanically align the teeth. Pierre Fauchard (1678 –1761) a French dentist was the first to make a scientific attempt to align irregular teeth by an appliance named *Bandeau* appliance. Fauchard also used to reposition irregular teeth with his *Pelican* forceps and then ligate them with neighboring teeth until healing took place. Fauchard published his work in 1728 in his landmark book entitled *The Surgeon Dentist: A Treatise on the Teeth*.

Edward Hartley Angle (1855-1930) was the most dominant and influential figure in orthodontics and is regarded as the "Father of Modern Orthodontics." Contemporary orthodontic brackets are modification of a standard edgewise brackets developed by Edward H Angle. Conventional stainless steel brackets with softer base component and harder slot/wings component should be preferred (1). Metallic orthodontic appliances are known to release metal ions in electrolytes such as saliva and body fluids, which leads to changes in mechanical properties and the surface morphologies of the appliances. Toms reported serious clinical implications for the corrosion of orthodontic appliances, ranging from a loss of dimension resulting in lower forces being applied to the teeth, through stress corrosion failure of the appliance, to the production of possibly toxic corrosion products. In order to ensure safe use in the oral cavity, appliances should exhibit high corrosion resistance and the requisite biomechanical properties for orthodontic treatment. Although orthodontic appliances such as brackets, wires, bands, etc., may feature high corrosion resistance, the possibility of corrosion may be increased when these appliances are combined for orthodontic treatment in the oral cavity. pitting corrosion of orthodontic appliances is common due to the aggressive action of Chloride ions in saliva, or from food or drink. The salivary solution that exists in the oral cavity is often under conditions of low pH and with high chloride ion concentration. These environments reduce the stability of the stainless steel passive film, which allows pitting and crevice corrosion to occur more easily(3).

Signs of corrosion include subtle physical changes in metals such as color change and rough surface, and chemical changes resulting from the release of metal ions, known as corrosion products, in the surrounding solution. Among these corrosion products, the release of nickel and chromium ions is most often studied because of their negative effects, which include allergies, dermatitis, gingivitis, and enamel staining, and they have been found to be mutagenic, carcinogenic, cytotoxic, and can cause DNA damage.(4) Nickel is the most common cause of metal-induced allergic contact dermatitis in man and produces more allergic reactions than all other metals combined. Second in frequency is chromium.(5) 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.

The incidence for nickel allergy was reported to be 1% in male subjects and 10% in female subjects. On the other hand, the incidence for chromium allergy is estimated at 10% in male subjects and 3% in female subjects (6). The potential of an alloy to cause an allergic reaction is related to the pattern and mode of corrosion.(7) For nickel compounds, risk is inversely related to its solubility in an aqueous media. For chromium compounds, carcinogenic risk has only been identified with compounds in which the chromium is in a hexavalent oxidation state. 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(6). This study is aiming to compare and evaluate the release of metal ions like nickel and chromium from three different stainless steel brackets of three different manufactures at seventh day and twenty eighth day when put in artificial saliva at pH 6.4. The samples were analyzed to identify the

traces of metal ions using inductively coupled plasma optical emission spectrophotometry.

MATERIALS AND METHODS

This study was aimed to assess and compare the rate of release of Nickel and Chromium ions at two different time intervals from stainless steel brackets by three different manufacturers when immersed in artificial saliva and also to evaluate the difference in the rate of ion release at seventh day and twenty eighth day.

INCLUSION CRITERIA: Standard upper first premolar stainless steel brackets of different manufacturers divided into three equal groups.

- a) 3M ESPE
- b) JJ ORTHODONTICS.
- c) MODERN ORTHODONTICS

MATERIALS USED

1. Standard stainless steel brackets:
 - a) 3M ESPE (30)
 - b) JJ Orthodontics (30)
 - c) Modern Orthodontics (30)
2. Artificial saliva
3. Bracket holder
4. Measuring Jar
5. Ninety small plastic containers (30 ml)

SAMPLE SIZE: A total of ninety standard stainless steel orthodontic brackets were used for this study. The brackets will be divided into three equal groups. Each group containing thirty standard stainless steel brackets, which will be of 3M ESPE, JJ orthodontics and modern orthodontic brands.

- Group A= 30 Stainless Steel 0.022 Brackets, 3M ESPE
 Group B = 30 Stainless Steel 0.022 Brackets, JJ Orthodontics
 Group C= 30 Stainless Steel 0.022 Brackets, Modern Orthodontics

PROCEDURE

Each bracket is incubated at a room temperature of 37°C in individual 30ml plastic containers containing 15ml of artificial saliva at pH 6.7. After incubation, on the seventh day, 2 ml of artificial saliva was taken from each of the plastic containers for inductively coupled plasma optical emission spectrometric evaluation (ICP-OES). The artificial saliva solutions will be replaced after each sample collection to avoid saturation with corrosion products. The plastic containers are then restored at 37°C, and similar such 2 ml of artificial saliva is collected from all the samples containing stainless steel brackets immersed in artificial saliva after twenty eight days. The immersed solution will be tested for Nickel and Chromium ion release in ICP-OES. The instrument will be calibrated using commercially available nickel and chromium standard stock solution to prepare the working standards with distilled and deionized water. More dilute solutions (0.1-10 mg/mL) of each ion were freshly prepared daily by appropriate dilutions of their stock solutions. To minimize the matrix effect in ICP measurements, the stock solution of each ion was diluted with artificial saliva. Calibration plots will be generated at the start of every run using freshly prepared working standards. This instrument allows measurement of extremely low concentrations (ppb) of released nickel and chromium ions. Inductively coupled plasma optical emission spectrophotometric evaluation (ICP-OES) is used mainly for elemental analysis. Around seventy elements were analysed in a single run. Liquid media are commonly analysed using this method. In case of any solid media, first it is digested by means of microwave method or an open digestion is done in the presence of an acidified media which is filtered into a liquid nature and then analysed using ICP-OES. A sample collecting tube is present, which is dipped in the sample to be analysed. From the sample collecting tube it is passed onto the peristaltic pump, which undergoes peristaltic movement and the sample reaches the spray chamber. In the spray chamber, the sample gets converted to aerosol and then to fine

droplets by means of a nebulizer present in the spray chamber. These fine droplets are then allowed to pass through plasma. Argon gas is commonly used for creating plasma. About 6000 kelvin temperature is needed for creating plasma. Nitrogen gas is used as the shear gas. An exhaust, chiller, and compressor are also needed for the efficient working of ICP-OES. In the presence of plasma, these fine droplets get atomized, ionized, and get converted to their excited form. As they are not stable in nature in their excited form, they release energy in the form of particular photos and get converted to their ground state. Each element has its own corresponding wavelength. According to the wavelength present, the concentration of the element in that particular medium can be analyzed. The standard stock solutions of the elements to be analyzed are individually made and three or five serial dilutions are made from the standard stock solution. These diluted solutions are initially analyzed and a standard calibration curve is drawn. Into that calibration made, the samples which are going to be analyzed are given and individual elemental concentrations are calculated.



Figure 1. 3M ESPE Stainless Steel Brackets



Figure 2. Modern Orthodontic Stainless Steel Brackets



Figure 3. JJ Orthodontic Stainless Steel Brackets

RESULTS

Data was analyzed using the statistical package SPSS 26.0 (SPSS Inc., Chicago, IL) and level of significance was set at $p < 0.05$. Descriptive statistics were performed to assess the mean and standard deviation of the respective groups. Normality of the data was assessed using Shapiro Wilkinson test. Since the data was following normal distribution and parametric test were used for the data analysis. Inferential statistics to find out the difference within the groups was done using paired t test.



Figure 4. Artificial Saliva

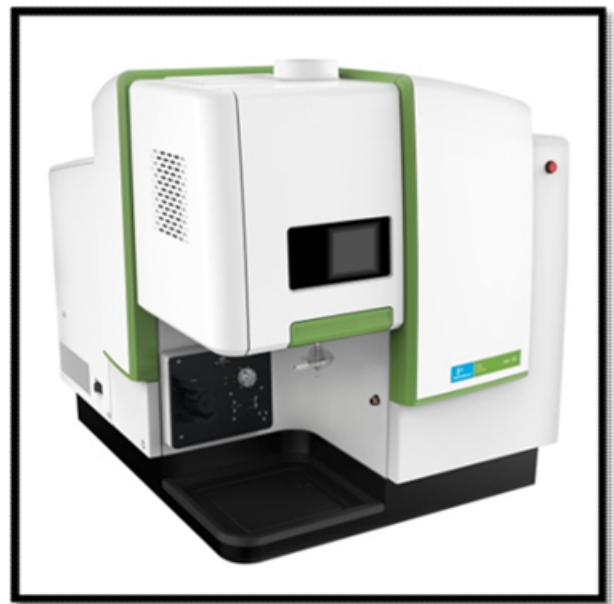
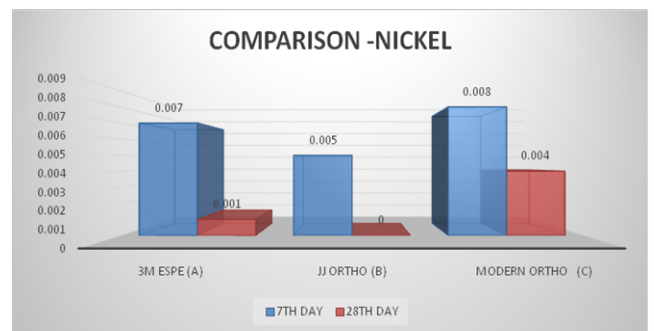
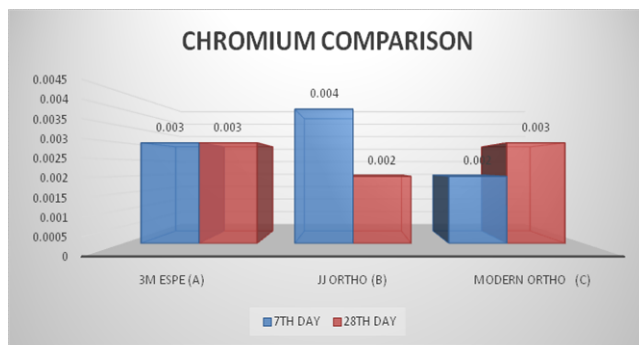


Figure 5. ICP-OES



Graph 1. Comparison of Nickel release

One way ANOVA test followed by Tukey's HSD test has been used to check the difference between three groups.



Graph 2. Comparison of Chromium release

Two tailed One way Anova test reported significant difference regarding the NICKEL release on both days. Post hoc test analysis was done to analyse the pair group significance at each time interval. During seventh day, the significant difference was seen between JJ and MODERN ORTHO ($P=0.009$). During twenty eighth day the significant difference was seen regarding all the three pair groups. Within group analysis using two tailed Paired T test reported statistically significant result regarding 3M and MODERN. JJ group did not report significant difference from over the time line. 3M group reported more change compared to MODERN. Two tailed one way Anova test reported significant difference regarding the CHROMIUM release on seventh day only. Post hoc test analysis was done to analyse the pair group significance at each time interval. During seventh day, significant difference was seen regarding all the three pair groups comparison. Within group analysis using two tailed paired T test reported statistically significant result regarding JJ and MODERN. 3M group did not report significant difference from over the time line. JJ group reported more change compared to MODERN.

DISCUSSION

Metallic orthodontic appliances are known to release metal ions in electrolytes like saliva and body fluids, which leads to changes in mechanical properties and the surface morphologies of the appliances.⁽³⁾ Corrosion is an electrochemical process that results in the loss of essential metallic properties of a metal. Depending on the thermodynamics and kinetics of the overall reaction, corrosion may proceed slowly or rapidly and occur as a general or localized attack. Corrosion occurs either through loss of metal ions directly into solution or by progressive dissolution of a surface film, typically an oxide or sulfide, on the metal. Mechanical or chemical disruption of the protective film, or exposure to a medium that dissolves the film and prevents its reformation, will result in corrosion. Even in the absence of abrasion, oxide films commonly dissolve slowly and then reform as the exposed metal surface is exposed to oxygen from its surrounding medium⁽⁸⁾. Corrosion involves two concomitant reactions, an oxidation reaction at the anode and a reduction reaction at the cathode. Commonly anodic reaction will continue until there is total consumption of the metal, by forming a protective surface film, the process known as passivation or the cathodic reactant is consumed⁽⁸⁾. The amount of nickel ions released from group A (3M ESPE) was 0.223 ppm on seventh day and 0.053 ppm on twenty eighth day and that of group B (JJ ORTHO) was 0.165 ppm on seventh day and 0.150 on twenty eighth day, and the nickel ions released from group C (MODERN ORTHO) was 0.267 ppm on seventh day and 0.145 ppm on twenty eighth day. All the above mentioned results were within the biologic safe limits (2.5-50 ppm) and does not cause any serious health hazard. More chromium, iron, and manganese ions were released in solutions with a pH 6.7 and pH 10. This was in line with Weisman's (1968) study, which stated that acidity created a condition in which the oxide films contained in stainless steel became unstable, resulting in reduced corrosion resistance. In the Huang et al. (2004) study, metal brackets immersed in a pH 4 solution released more ions when compared to that of metal brackets immersed in an artificial saliva solution with a pH of 6.7.⁽⁹⁾ This was in accordance with my study where less amount of metal ions were released at pH 6.7 (pH of artificial saliva taken for this study) compared to an acidic

pH. Faccioni et al stated that both metals (Nickel and chromium) induced DNA damage in oral mucosal cells.⁽¹⁰⁾ Park and Shearer found that the precipitated corrosion products contained much higher amounts of chromium than nickel, whereas the solution itself contained more nickel than chromium. They also found that nickel was released primarily as soluble compounds, whereas chromium was released primarily as insoluble compounds. Shearer and Menne et al. found that the corrosion of the appliances reached a plateau after 6 days and did not increase appreciably thereafter. Marek and Treharne had similar findings and they found that the nickel present on the surface of the stainless steel may quickly corrode during the first 7 days of the experiment, then the rate of release drops off as the surface nickel is depleted. Then the corrosion products may have formed on the surface after 7 days slowing the corrosion of nickel.⁽⁹⁾ Barrett et al in his study stated that the nickel release reached a maximum after approximately 1-week following which the rate of release diminished while the chromium release increased during the first 2 weeks and leveled off during the subsequent 2 weeks.⁽¹¹⁾ This was in accordance with my study that Ni release was more following the first week and gradually diminishes due to the stable oxide layer formed. In a study conducted by Kuhta et al. (2009), the pH factor significantly affected ion release; more visible ions were released at pH 3.5 compared to pH 6.75 after a 28-day immersion.⁽¹²⁾ Von Fraunhofer (1997) discovered that the microstructure of metals is another parameter that affect the mechanical and corrosion properties of a metal. The variation in the manufacturing process and the polishing process after manufacture might affect the corrosive properties of orthodontic appliances.⁽²⁾

In my study, the intra group findings for GROUP A (3M ESPE), showed maximum amount of nickel ions was released on day seven and gradually declined till the end of day twenty eight. The maximum peak value at the end of seventh day was found to be 0.223 ppm, which is well below the critical concentration (600-2500 micro grams) necessary to induce nickel allergy. A similar study was done by Maja Kuhta for an immersion period of 28 days where the maximum amount of nickel and chromium ions were released during the first week of appliance immersion and a gradual decline was seen thereafter due to the formation of a stable oxide layer which slows down further ion release.^(13,14) The intra group findings for GROUP B (JJ ORTHODONTICS) showed that both nickel and chromium ions released were more on day seven compared to day twenty eight. This was in accordance with a study done by Sfondrini et al who did a similar study with an immersion period of 5 days, where the bracket showed a significant increase in nickel release at all time intervals 1, 24, 48, and 120 hours. For Chromium release, the brackets showed a significant increase in metal release among all-time intervals (0.25 to 120 hours; $P < .002$).⁽¹⁵⁾ The intra group findings for GROUP C (MODERN ORTHODONTICS) showed nickel release was more on seventh day compared to twenty eighth day and chromium release was little more on twenty eighth day compared to seventh day. This was in accordance with a study done by Gabriel and Schmidt et al, who did a similar study with an immersion period of 60 days, where maximum nickel and chromium ions were released in the first week (7 days) followed by the stabilization of the ionic release till day 60 for nickel. The chromium ion release decreased only by the 8th week, similar to the study done by Hwang.⁽¹⁶⁾ This was also in accordance with another study done by Singh et al, where there was a statistical difference in the nickel and chromium concentration between the first and third week. The highest concentration of nickel was found in the first week which gradually decreased by third week⁽¹⁷⁾ From the results of various studies suggests that the nickel concentration on the surface of the appliance is being depleted at a faster rate than that of chromium. The orthodontic appliances release measurable amounts of nickel and chromium when placed in an artificial saliva medium. The nickel release reaches maximum after approximately one week, then the rate of release diminishes with time. The chromium release increases during the first two weeks and levels-off during the subsequent two weeks.⁽⁷⁾ The decrease in the rate of release in the fourth week of appliance therapy is due to the formation of a stable oxide layer which further diminishes the metal ion release.

Table 1. Analysis of chromium

	GROUP	SEVENTH DAY	TWENTY EIGHT DAY	T VALUE	P VALUE	DIFFERENCE
CHROMIUM RELEASE	3M ESPE (GROUP A)	0.003±0.001	0.003±0.003	0.01	0.99	0.0±0.002
	JJ ORTHO (GROUP B)	0.004±0.002	0.002±0.001	4.89	0.0001*	0.002±0.001
	MODERN ORTHO (GROUP C)	0.002±0.001	0.003±0.002	2.44	0.017*	0.001±0.001
P VALUE (ONE WAY ANOVA)		0.0001*	0.012			
POST HOC TEST	A Vs B	0.02*	0.17			
	A Vs C	0.02*	0.99			
	B Vs C	0.0001*	0.17			

*P<0.05 IS STATISTICALLY SIGNIFICANT

Table 2. Analysis of nickel

	GROUP	SEVENTH DAY	TWENTY EIGHT DAY	T VALUE	P VALUE	DIFFERENCE
NICKEL RELEASE	3M ESPE (GROUP A)	0.007±0.004	0.001±0.001	7.97	0.0001*	0.006±0.003
	JJ ORTHO (GROUP B)	0.005±0.002	0.005±0.002	0.01	0.99	0.0±0.0
	MODERN ORTHO (GROUP C)	0.008±0.005	0.004±0.001	4.29	0.0001*	0.004±0.004
P VALUE (ONE WAY ANOVA)		0.019*	0.0001*			
POST HOC TEST	A Vs B	0.11	0.0001*			
	A Vs C	0.57	0.0001*			
	B Vs C	0.009*	0.02*			

*P<0.05 IS STATISTICALLY SIGNIFICANT

The Inter Group Comparison of this study reveals that the Ni release was less for JJ Ortho followed by 3M and Modern Ortho on seventh day. On twenty eighth day, Ni release was less for 3M followed by Modern Ortho and JJ Ortho. It also reveals that the Cr release was less for Modern Ortho followed by 3M and JJ Ortho on seventh day. On twenty eighth day, Cr release was same for Modern Ortho and 3M followed by JJ Orthodontics. The overall picture of the Intra-group and Inter-group findings for Nickel and Chromium at different time intervals in this study, when compared to other articles has given the impression that most of the findings were similar. The ions released from the brackets were well below the average dietary intake [nickel - 300 to 500 µg/day; chromium- 5 to more than 100 µg/day] and the critical concentration level to induce a nickel hypersensitivity reaction [600-2500 µg].

CONCLUSION

Based on the result of this study it can be concluded that stainless steel brackets of 3M shows less amount of Nickel ion release on 28th day compared to JJ and Modern. Stainless steel brackets of Modern shows less amount of Chromium ion release compared to 3M and JJ on both days. Sometimes variations has occurred when compared with other findings may be due to the difference in the concentration of ingredients present in the artificial saliva, sample size or may be due to the difference in the manufacturing process of stainless steel materials used.

GLOSSARY OF ABBREVIATIONS

ANOVA	Analysis of Variance
Cr	Chromium
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrophotometry
Ni	Nickel
P value	Probability value
SD	Standard Deviation

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