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Quantitative and qualitative analysis of metallic ion release of orthodontic brackets in three different pH conditions - An invitro study

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ABSTRACT

Background: Fluoridated mouth rinses improve anti-cariogenic environment but decrease oral pH below critical value, affecting orthodontic bracket surface topography and causing corrosive changes over prolonged use. This invitro study aimed to quantitatively and qualitatively assess the surface topography and metallic ion release of the stainless steel (SS) brackets at varying acidic and alkaline pH.
Materials and methods: Forty unused SS brackets were divided into four groups (Group A, B, C, D) and immersed for 48- hours in solutions of artificial saliva and sodium fluoride (0.2 %) mouth rinse at varying pH of 5.5,6.7,7 and 8. The surface morphologic changes were analyzed under scanning electron microscope (SEM) at 50×, 150×, and 500× magnification. The changes in slot area were scored using the customized scale. The Energy Dispersive Xray Spectroscopy Analysis (EDAX) was used to estimate the probed elements' atomic and weight percentage.
Results: The mean score of the scale was 3.4 for the brackets immersed in the acidic solution which was statistically significant ($p = 0.00$)and for alkaline and neutral solutions ($p = 0.00$). Chromium was found to be significantly higher in the alkaline solution ($p = 0.016$) followed by the neutral solution. Carbon was found excess in acidic solution than the neutral and alkaline solution.
Conclusion: Quantitative and qualitative analysis of the ion release in stainless steel brackets using SEM and EDAX revealed the corrosive effect of fluoride ion causing maximum surface changes in acidic medium and chromium release in alkaline pH.

1. Introduction

Metallic brackets and wires are integral to the force delivery system in orthodontic treatment. Due to prolonged intraoral placement, brackets are exposed to microbial attacks, varying oral pH, and temperature fluctuations. The physical, chemical, and electrochemical deviations are proven to corrode the surface topography of the bracket surfaces and slots, leading to pitting corrosion, elemental changes, and irregularities in the wire contact surfaces that create asperities, causing increased friction.[1,2] Wires are subjected to similar conditions, but the possibility of being replaced every 4–6 weeks indicates that the brackets bear the maximum effects of repeated clinical use. The commercially used brackets are predominantly stainless steel (SS) of 303, 304, 316, and 316 L type,[3] which, upon corrosion, releases byproducts of iron (Fe), chromium (Cr), and nickel (Ni) intraorally.[4] The ion release is more significant in an acidic environment, and even traces of copper and manganese have been found in the saliva of patients undergoing orthodontic treatment.[4–6] While dietary habits influence the ion release, orthodontic patients are often prescribed fluoride-containing mouthwashes that alter oral pH, potentially accelerating corrosion. Various studies have used a scanning electron microscope (SEM) to analyze the surface irregularities caused by such agents.[7,8] Typically, a nominal scale has been employed to segregate the images, and a few studies have used a discrete scale that demarcates the severity of the surface irregularities.[7,9] However, there is limited data to fully understand the complex interactions between fluoride, pH, and the oral environment.[10,11]

Hence, this study primarily intends to characterize the surface topographic changes using the SEM images of the brackets after exposure to fluoridating mouth rinses of varying acidic and alkaline pH concentrations. It also aims to compare and estimate the surface elemental changes of the altered surfaces on the SS brackets. The suspected corrosive changes were compared with the control brackets

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immersed in artificial saliva.

2. Materials and methodology

The invitro study was conducted at the Department of Orthodontics, and Sophisticated Analytical Instrument Facility (SAIF), Department of Electron Microscopy, after obtaining the institute's ethical committee approval (IECPG 307/May 29, 2019). The null hypothesis was that the fluoride containing mouth rinses do not cause any changes in surface topography and elemental composition of the brackets.

3. Materials and Methods

3.1. Adjustment of pH

The artificial saliva used as the control base solution in this study (Mouth Wet ™, United Biotech Pvt. Ltd, Delhi, India) had pH 6.7 and its contents has been listed in Supple. Table. The neutral solution was prepared by mixing artificial saliva and sodium fluoride mouth rinse (pH 4.84) (U.S.P. 0.2 % w/v Proflo mouth rinse, Sandika Pharmaceuticals Pvt. Ltd, New Delhi, India). The acidic and alkaline pH was optimised using the pH meter (pH 700, Eutech Instruments, Thermo Fisher Scientific, Massachusetts,USA). Four solutions including (artificial saliva) control, neutral solution (pH 7) (artificial saliva with fluoridated mouthwash), an acidic solution with 1 N hydrochloric acid (pH 5.5), and an alkaline solution with 1 N potassium hydroxide with pH 8 were prepared, labeled and incubated at 37 °C.

Forty unused first premolar SS brackets with 0.022 × 0.028-inch slots (Orthodontic Supplies Pvt. Ltd. (OSL), Leicestershire, UK) were checked for any defects, washed with distilled water, and dried for 30 secs. They were decontaminated in 70 % alcohol; washed in double distilled water and air dried for 3 mins. They were divided into four groups (A,B,C,D) of ten brackets per group and immersed for 48 hrs in the solutions, considering the routine usage of 2 mins mouth wash/rinse.

3.2. Surface topography analysis and scoring

After 48 hrs, each bracket was retrieved, cleaned with double distilled water for 10 mins, dehydrated in an ascending grade of acetone, and critical point dried (E3100, Quorum tech Pvt. ltd). They were mounted on the aluminum stubs, sputter coated with colloidal gold in a super cool sputter system (Bal-Tec SCD 050 technology, Oerlikon Balzers, Liechtenstein) and observed using the EVO 18 scanning electron microscope (Carl Zeiss, Germany) under magnification of 50×, 150×, and 500× (20 Kilovolts) (Fig. 1A,B,C). Digital images were acquired through integrated charged coupled device (CCD) camera with SmartSEM GUI (Version 05.06). Bracket slots were analyzed for surface dullness, roughness, irregularities, cracks, craters, pitting, and crevice corrosion. The images were carefully assessed in the slot area, and scored using the customized index developed by the authors with scoring criteria (Fig. 2).

3.3. Elemental composition analysis (EDAX)

The study used energy dispersive analysis of xrays (EDAX) at 20 kV accelerating voltage. To investigate the compositional arrangement of brackets in neutral, acidic, alkaline, and control solutions. The atomic and weight percentages of probed elements, including carbon, sodium, oxygen, iron, chromium, and gold, were estimated and tabulated.

3.4. Statistical analysis

The SPSS statistical software version 22.0 was used to analyze the data. One-way ANOVA analysis was performed to compare the atomic and weight percentages of each probed element obtained via EDAX analysis. For the multivariant comparison,Mann-Whitney *U* test was done for carbon, oxygen, and iron. The 2/2 test was used to assess the sodium, and gold while Mann Whitney test was used to compare chromium levels in different pH solutions. The scoring of the SEM data was statistically analyzed using one-way ANOVA followed by the Bonferroni test. The level of significance was set at $p < 0.05$. Twenty random images were scored again after two weeks for intra and inter observer judgement by authors DP and CPK and found to have good inter and intra rater agreement.

4. Results

4.1. Surface topography

The study assessed brackets under various pH conditions and graded them from 0 to 4 based on slot area morphology. Results showed significant scores in all four pH conditions. No brackets scored 0 even in artificial saliva (Control) solution, indicating the inadvertent effects of salivary components on the surface topography of the brackets even after a brief 48-hrs immersion period. Surface irregularities were highest on brackets immersed in acidic solutions, with a mean score of 3.4 and their slots showed complete corrosion with indistinguishable margins and surface craters. Brackets immersed in alkaline solution also scored significantly higher, indicating the corrosive effect of the alkaline environment combined with fluoride ions. We also noted that the corrosion is greater when the pH is more acidic than alkaline (Table 1).

4.2. Atomic percentage

The study analyzed the surface adsorption of carbon, oxygen, and iron in SS brackets (Fig. 3) (Table 2). The atomic weights of oxygen and iron were similar in all four experimental solutions. The acidic group showed the highest carbon percentage (68.72 ± 6.12) compared to the control group brackets. The mean atomic weight of carbon was 67.70 ± 2.49 and 64.67 ± 6.54 in the alkaline and neutral groups, respectively.

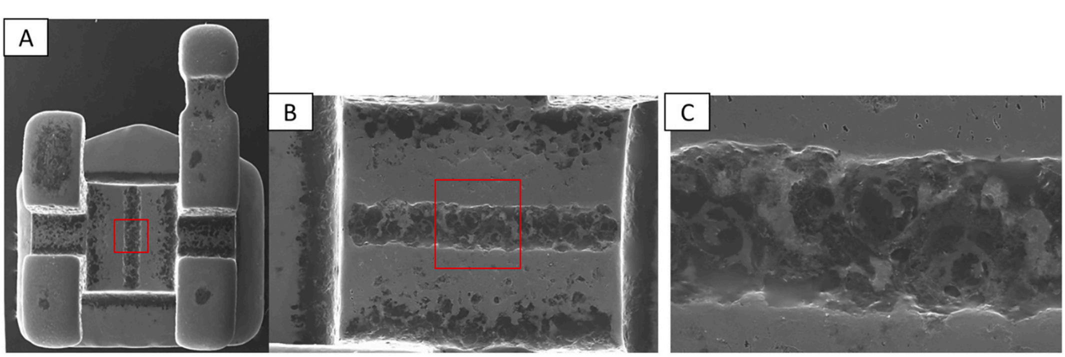


Fig. 1. SEM analysis at A) 50× B) 150× C) 500× magnification.

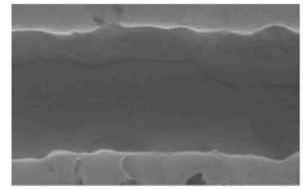
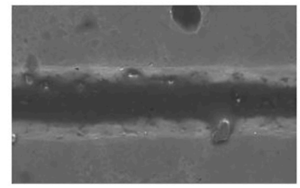
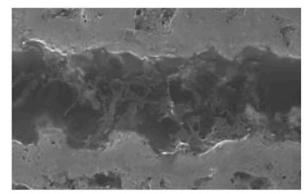
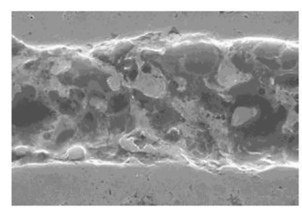
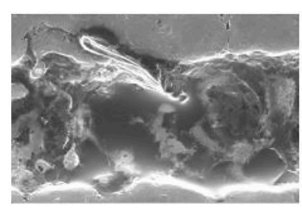
Observed Characteristics	Scoring	Images
• Well differentiable slot margins • No surface corrosion and roughness • No surface irregularity • No cracks • No craters	0	
• Wavy slot margins but still appreciable • Mild surface corrosion and roughness • Pitting irregularity • Cracks and craters confined to borders	1	
• Irregular slot margins • Surface corrosion in more than 25% but less than 50% • Surface roughness spread throughout slot area • Cracks and craters spreads to outer surface of the slot	2	
• Long wavy and irregular margins • Surface corrosion and roughness more than 50% area • Surface irregularity in more than 50% of the area • Cracks and craters reaching outer borders of the area	3	
• Indistinguishable slot margins • Corrosion in entire slot area • Surface irregularity in total area • Cracks and craters in the complete slot area	4	

Fig. 2. Quantitative analysis of SEM images of brackets at 500× magnification with customized Scoring criteria.

Table 1
Comparison of mean values of scoring index for each sample from each test solution.

pH	Number of brackets per group (n = 10)	Mean score	Standard Deviation (Mean ± SD)	P value
Acidic	10	3.4[a]	0.69	0.00
Alkaline	10	2.4[a,b,c]	0.51	0.00
Neutral	10	1.7[a,b]	0.67	0.00
Control	10	1.3[a,c]	0.48	0.00

[a] Statistical difference between acidic, alkaline, neutral and control solutions.
[b] Statistical difference between alkaline and neutral solutions.
[c] Statistical difference between alkaline and control solutions.

Gold had a greater percentage in acidic solution with a statistically significant p-value of <0.016 and was absent in alkaline and neutral solutions. Chromium release was increased in alkaline and neutral solutions than in the acidic solution, indicating the corrosive nature of fluoride ions in an alkaline environment. The alkaline condition also increased the release of sodium ions, while it was not seen in the acidic pH.The maximum atomic percentage of chromium was detected in brackets of neutral and alkaline solutions.

4.3. Weight percentage

The study analyzed the release of carbon, oxygen, and iron in various pH environments (Fig. 3) (Table 3). The acidic solution caused a release of carbon (44.15 ± 10.06), oxygen, and iron, but the release was less compared to the alkaline solution, which showed maximum release of carbon (46.88 ± 5.71) and oxygen. The control solution showed the greatest iron release. Base elements were released at comparable levels in all pH environments. Chromium release was observed in acidic, alkaline, and neutral conditions, with a maximum of 7.37 % in the neutral solution and 3.02 % in the alkaline solution. Gold percentage was traceable in the control group and almost untraceable across other test solutions. Sodium percentage was significantly greater in alkaline groups.

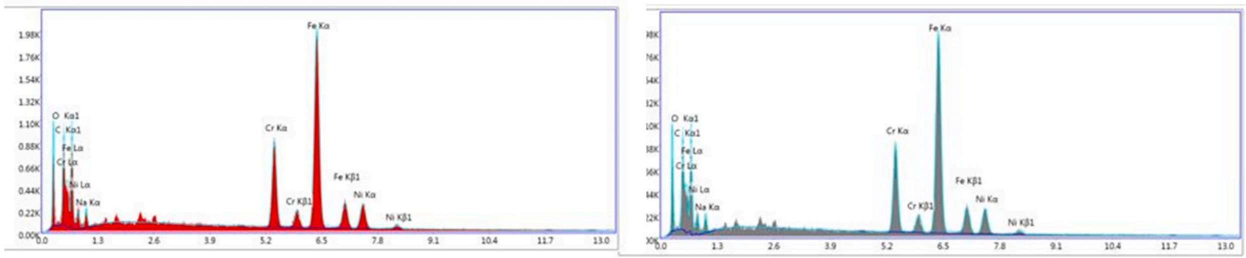


Fig. 3. A:Representative EDAX graph of the atomic percentage of brackets. B:Representative EDAX graph of weight percentage of brackets.

Table 2
Comparison of atomic percentage of elements on the surface of the stainless-steel brackets on each test solutions.

Probed elements	Acidic solution Mean ± SD	Alkaline solution Mean ± SD	Neutral solution Mean ± SD	Control solution Mean ± SD	P Value (<0.05)
Carbon	68.72[a] ± 6.12	67.70 ± 2.49	64.67 ± 6.54	61.30[a] ±5.47	0.0178
Oxygen	15.13 ± 11.74	20.10 ± 8.89	17.96 ± 4.44	21.89 ± 3.083	0.2651
Iron	14.11 ± 9.24	11.66 ± 11.08	11.46 ± 4.50	16.63 ± 7.65	0.4906
	Acidic solution (Median ± Q1, Q3)	**Alkaline solution (Median ± Q1, Q3)**	**Neutral solution (Median ± Q1, Q3)**	**Control solution (Median ± Q1, Q3)**	
Sodium	0 (0,0)	0.65 (0,2.8)	0 (0,3.44)	0 (0,0)	NS
Chromium	0 (0,2.68)	1[a] (0,3.74)	2.61[b] (0,3.28)	0[a],[b] (0,0)	[a],[b] <0.016
Gold	0.115[a] (0.07,0.38)	0 (0,0)	0[a],[b] (0,0)	0.115[b] (0.07,0.38)	[a],[b] <0.016

[a] Statistical difference of Sodium ion between acidic, alkaline, neutral and control solutions.
[b] Statistical difference of Chromium ion between neutral and control solutions.

5. Discussion

Bio-degradation of the metallic bracket occurs after intraoral use due to variations in the dietary pH, temperature fluctuations, mechanical abrasion, oral microflora, and their byproducts.[1] Fluoride decay prevention protocols for high-risk patients[12] include mouth rinses for 1–2 mins in a biweekly regime and fluoridated toothpaste.[13,14] Nevertheless, these chemicals have an adverse impact on the surface topography and frictional resistance of orthodontic wires and brackets and increase surface irregularities over time.[10,11,15–20] Hence, this study evaluated solely the effects of 0.2 % sodium fluoride mouth rinse on received SS brackets under different acidic and alkaline pH settings.

Studies examining the effects of different pH conditions on intraoral appliances with fluoride intake mainly focus on wires rather than brackets,[19–23] our study fills this gap. The brackets need to be evaluated over short-term appliances to understand the long-standing effects of the fluoridating agents.

The study's findings are significant, revealing surface degradation in all brackets, even those immersed in artificial saliva with a pH of 6.7. The surface topographic degradation showed a clear correlation with decreasing pH levels, indicating the profound influence of fluoride ions and pH on the brackets. The average score was 3.4 in the group with an acidic environment (pH 5.5) and 2.4 in the group with an alkaline environment (pH 8). In contrast, the group with a neutral environment (pH 7) had an average score of 1.7. This damage is caused by the reaction between the fluoride ion in sodium fluoride mouth rinse and hydrogen ions, resulting in the formation of hydrofluoric acid. The acid then combines with chromium oxide on the protective layer, causing corrosion. The metal brackets exhibit the highest level of corrosion when exposed to NaF at an acidic pH of 4. Nakagawa et al. (2001) found that even at low concentrations, the fluoride ions with an acidic pH increase the corrosion of the titanium, which is in line with our findings.[24]

Remarkably, the alkaline pH brackets exhibited more significant surface imperfections than the control group in the study. Mouth rinses purchased in stores often have a pH range of 4.23–7.34. Many research have utilized mouth rinses with different pH levels, particularly within the range of 4–7.[8,12,17,25] The permissible concentrations of NaF in the 0.05 % and 0.2 % mouth rinse solutions are 230 ppm and 920 ppm, respectively.[12]

Consuming dairy products can lead to a more alkaline intraoral pH, while consuming foods high in citrus, vinegar, and aerated drinks have a more acidic pH.[26] This study used sodium fluoride mouthwash with HCl/KOH to simulate these pH fluctuations. The temperature of a solution, even at the same pH, can significantly influence the surface of brackets, indicating a synergistic effect of fluoride, pH, and oral environment temperature.[27]

The study used energy dispersive spectroscopy to estimate the atomic and weight percentages of elements such as iron, chromium, oxygen, sodium, gold, and carbon in unused brackets. Fluorides cause

Table 3
Comparison of weight percentage of elements on the surface of the stainless-steel brackets on each test solutions.

Probed elements	Acidic solution Mean ± SD	Alkaline solution Mean ± SD	Neutral solution Mean ± SD	Control solution Mean ± SD	P Value (<0.05)
Carbon	44.15 ± 10.06	46.88 ± 5.71	43.32 ± 7.32	37.37 ± 9.82	0.099
Oxygen	15.99 ± 11.20	19.60 ± 11.57	15.85 ± 5.76	17.82 ± 5.04	0.758
Iron	31.04 ± 19.23	27.58 ± 12.24	32.93 ± 8.33	43.29 ± 14.20	0.092
	Acidic solution (Median ± Q1, Q3)	**Alkaline solution (Median ± Q1, Q3)**	**Neutral solution (Median ± Q1, Q3)**	**Control solution (Median ± Q1, Q3)**	
Sodium	0[a](0,0)	1.29[a](0,3.44)	0 (0,3.98)	0 (0,0)	[a]<0.016
Chromium	0 (0,7.73)	3.02[a](0,8.36)	7.37[b](0,8.81)	0[a],[b](0,0)	[a],[b] <0.016
Gold	0 (0,1.35)	0 (0,0)	0[a](0,0)	1.2[a](0.69,2.65)	[a]<0.016

[a] Statistical difference of Sodium ion between acidic, alkaline, neutral and control solutions.
[b] Statistical difference of Chromium ion between neutral and control solutions.

surface elemental changes in orthodontic materials,[16] reflected in the composition of surface adsorbed elements, as corroded brackets show changes in color and texture.[28]

The study found that iron and oxygen atomic percentages were similar across all pH groups, similar to Doomen and Houb-dine et al.[7,29] However, the atomic percentage of carbon was found to be greater in the acidic group, due to carbon adsorption from the atmosphere on the bracket surfaces caused by oxidation of the brackets.[29]

Iron leached out was similar across all pH groups, with no statistical significance, with the control group having the highest weight percentage of iron.[30] Chromium release was highest in neutral and alkaline pH solutions, as expected in Refs. 7,30; the results deviated from the assumption that iron and chromium leach out more in acidic solutions, possibly due to the essential nature of the solution causing increased metal ion release. Iron and chromium release began on the first day and peaked in the first and second week after intraoral placement.[31] Fluoride anions increase the dissolution of the manganese by decreasing the pH.[17] Oxygen distribution was similar across different pH conditions. Negligible amounts of gold were detected in the control and acidic groups brackets.

The study found that sodium and chromium are released higher in alkaline solutions due to the presence of sodium hydroxide and sodium fluoride. Sodium fluoride was used as the base solution in all the pH solutions, which, along with alkaline pH, may have an additive effect on the sodium release.

Ehrami et al. compared the effects of 0.05 and 0.2 % of sodium fluoride mouthwashes on the surface topography of the wires and concluded that the weekly use of 0.2 % of NaF than 0.05 %.[15] As per our study 0.2 % fluoride mouthwash used 2 mins per day, releases chromium and carbon in acidic as well as neutral and alkaline solutions, indicating the corrosive nature of the mouth rinses. The literature suggests controlling fluoridating agents and cleaning bracket surfaces to overcome frictional resistance. The authors recommend replacing the premolar bracket before sliding mechanics when discoloration is clinically visible.

Strength and Limitations:

The in vitro study solely analyses the role of fluoride ions under varying pH without other confounding factors. The quantitative scoring method used in this study can be used to compare the surface deterioration changes in future. Oral cavity is a dynamic complex environment with bacteria, brushing, salivary action, and food interactions, hence results should be read with caution. A larger cohort with brackets from multiple manufacturers will increase the generalizability of the results.

6. Conclusion

Acidic pH significantly increases the release of carbon and gold, while chromium, sodium, and gold were released incrementally in alkaline as well as acidic pH. The fluoride ion synergized to increase carbon release in acidic environments and chromium release in neutral conditions.

Conflict of interest

We have no conflict of interest to disclose.

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