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Surface characteristics and ion leaching of 17-4 precipitation hardening stainless steel orthodontic brackets exposed to salt water and fluoride-based oral rinses

Research Article

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Abstract: The aim of this laboratory study is to analyze, compare, and contrast qualitatively and quantitatively the surface roughness and metal ion leaching of two brands of orthodontic precipitation hardening 17-4 stainless steel (SS) brackets exposed to salt water and commercially available NaF-based oral rinses. A total of 60 SS maxillary premolar orthodontic brackets from two brands (Victory Series™, and Mini Diamond Twin™) were assigned to three groups (n = 10). The brackets were immersed in NaF-containing oral rinses (Listerine™ or Orex™), or salt water rinse for 28 days. Microhardness (Vickers hardness number, VHN) and surface roughness (R_a) before and after immersion in oral rinses were evaluated using a non-contact optical profilometer. Inductively coupled plasma mass spectrometry (ICP-MS) was used to analyze the ion leaching into oral rinses. The local surface changes and elemental analysis of corrosion-free and corrosion spots after immersion were assessed and compared using scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (EDS). Data analysis was performed using Statistical Package for the Social Sciences software using one-way analysis of variance, the Tukey's *post hoc* test, and paired t-test (α = 0.05). The VHN of the brackets was significantly different at baseline and after immersion in the oral rinses ($P < 0.05$). The mean R_a before immersion in oral rinses for the two groups of the orthodontic brackets was significantly different ($P < 0.01$). The brackets immersed in salt water for Victory™ (0.65 ± 0.07 µm) and Mini Diamond™ (0.76 ± 0.08 µm) demonstrated significantly lower roughness values than NaF-based oral rinses (Listerine™ or Orex™) ($P < 0.01$). However, the difference in R_a between the bracket systems was significant only for Listerine™ and Orex™ rinses ($P > 0.01$). The ICP-MS analysis showed significant differences for Cu and Zn ions in Listerine™ and all ions except Ni for salt water oral rinses. On the contrary, the comparison between the bracket systems for the metal ions showed no significant difference in the Orex™ group ($P > 0.05$). Salt water oral rinses demonstrated significantly fewer surface changes than NaF-containing oral rinses. The leaching of the metal ions was significant for Fe^{2+} and Cr^{3+} but did not vary considerably for Ni^{2+} between the evaluated oral rinses.

Keywords: *corrosion • ion leaching • oral rinses • orthodontic brackets • stainless steel*

1. Introduction

Correction of malocclusion through fixed orthodontic appliances demands the use of different components made of metal, ceramic, and polymers [1,2]. Nonetheless, metallic materials' balanced combination of good mechanical, biological, and chemical properties makes them the preferred material for manufacturing fixed orthodontic appliances [3]. Orthodontic brackets are an essential component of orthodontic treatment and are crafted mainly from stainless steel (SS) alloys due to their excellent mechanical properties, corrosion resistance, and biocompatibility [4]. However,

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despite their high corrosion resistance, SS brackets can corrode in the oral environment because of its changing pH, which is one of the main concerns during orthodontic treatment [5].

Corrosion occurs when the brackets are exposed to an oral environment, such as diet, temperature changes, salivary pH, microbial flora, and mechanical stress [6,7]. Bracket corrosion could seriously impact the patient's oral health and treatment duration. It may even weaken the brackets' structural integrity, resulting in breakage and possibly prolonging the orthodontic treatment. Corrosion products may also discolor the teeth and surrounding tissues, which would substantially harm the aesthetics [8,9]. Iron (Fe), chromium (Cr), and nickel (Ni), the primary corrosion products of SS brackets, have the potential to cause detrimental effects on the body, ranging from allergy to cytotoxicity [10]. That said, orthodontic brackets are nowadays crafted from 17-4 precipitation hardening (PH) SS using metal injection molding (MIM).

The 17-4 PH SS is an alloy of Cr, Ni, and Cu that can be precipitation hardened [11]. The combination of exceptional strength, high toughness, and better corrosion resistance has made 17-4 PH SS distinct among the other SS used. Overall, this SS has higher mechanical properties, which can be further improved by aging [12,13]. For these reasons, it is utilized in the chemical sector, for equipment manufacture in the automobile and aerospace industries, and for medical and computer applications. Furthermore, a number of studies have provided encouraging findings about its mechanical characteristics and biocompatibility for use in the medical field [12,13]. The orthodontic brackets fabricated from 17-4 PH SS alloy are durable against stress from the arch wire to the bracket slot, and its low nickel content (4%) can help lower the risk of allergies [14].

Corrosion resistance is one of the basic principles of biocompatibility for orthodontic brackets. It can be influenced by several factors, including the alloy type, manufacturing method, and surface characteristics [9]. Various processes, including casting, milling, and MIM, are used for manufacturing metal orthodontic brackets [15]. Nevertheless, the casting process is expensive as 50 and 75% of the material used during machining becomes waste, and, furthermore, 90% of the metal used may be wasted in sprues and runners. Milling is only economically advantageous for manufacturing parts with basic geometry [16,17]. MIM is the least expensive and most competitive technology, owing to material savings during manufacturing and complicated and sophisticated parts that can be produced in large quantities [17].

During the active phase of orthodontic treatment, fluoride-containing products such as toothpaste and fluoride oral rinses are recommended to reduce the risk of dental caries [18]. Oral rinses also supplement the primary hygiene method since they are clinically effective in reducing plaque accumulation, and their anti-inflammatory effect makes them valuable during orthodontic treatment [19,20]. Nevertheless, some fluoride rinses combined with an acidic oral environment may compromise brackets' resistance to corrosion, increase metal ion release, and discolor SS brackets, jeopardizing their integrity and possibly promoting microbial adhesion that can lead to periodontal inflammation and dental caries [7,21,22].

On the contrary, natural remedies like salt water based oral rinses would seem more beneficial due to the negative consequences of the sodium-fluoride (NaF) solutions [23]. Previous studies have demonstrated that oral rinses made with salt water could improve dental hygiene by significantly reducing the amount of bacteria in the oral cavity and lessening halitosis and xerostomia [19,22–25]. While highlighting the possible adverse effects of fluoride-based oral rinses on orthodontic brackets, existing literature lacks scientific evidence on the effect of natural salt water rinses on the corrosion of orthodontic brackets. Therefore, it is vital to fill this gap by investigating the effect of salt water and commercially available NaF-containing oral rinses on the corrosion of orthodontic brackets.

In particular, the aim of this laboratory study is to analyze, compare, and contrast qualitatively and quantitatively the surface roughness and metal ion leaching of two widely used brands of orthodontic brackets exposed to salt water or commercially available NaF-based oral rinses. The study hypothesis was that there is no significant difference in the roughness and metal ion leaching of brackets exposed to salt water or NaF oral rinses.

2. Materials and methods

2.1 Study design, specimens, and test solutions

This laboratory-based study employed a quantitative and qualitative analysis. A total of 60 upper premolar SS orthodontic brackets from two manufacturers, Victory Series™ Low Profile (3M Unitek, Monrovia, CA, USA) and Mini Diamond Twin™ (Ormco, Glendora, CA, USA), were included in this study. Both the orthodontic brackets are manufactured by MIM.

Two commercially available NaF oral rinses, Listerine™ (Johnson & Johnson, Neuss, Germany) and Orex™ (Fushima, SL, Cantabria, Spain for Nahdi Medical Co.), and a readily prepared salt water rinse were used as test solutions.

Accordingly, the study had two bracket groups further divided randomly into six subgroups (n = 10) based on oral rinses. Table 1 details the orthodontic brackets and oral rinses used in this laboratory investigation. The surface characteristics and corrosion behavior of the orthodontic brackets immersed in oral rinses were analyzed using an optical profilometer, inductively coupled plasma mass spectrometer (ICP-MS), and scanning electron microscope (SEM) equipped with energy-dispersive X-ray analysis (EDS).

2.2 Evaluation of microhardness

The microhardness of the specimens was assessed using Vickers hardness tester (INNOVA TEST, Borgharenweg, Maastricht, Netherlands). A 200 g load was applied and stabilized for 15 s on the external wing surface of the study brackets. For each bracket, three Vickers indentation readings were randomly obtained and their mean value represented the hardness (Vickers hardness number, VHN) of that particular specimen [26].

2.3 Evaluation of surface roughness

Surface roughness (R_a) of the bracket surface was evaluated using a non-contact optical surface characterization and roughness assessment profilometer device (Bruker Contour GT, Billerica, MA, USA), working on a vertical scan interferometry principle. The bracket was placed on the turret, and a measurement area of approximately 1 mm^2 at the center (between the arch wire slot) was scanned using a ×5 Michelson magnification lens, ×1 scan speed, and 4% threshold [27]. The surface roughness, defined as the mean arithmetic heights measured through the surface area of the bracket, was expressed in micrometers (μm) and was analyzed by simple Vision 64 software (Bruker, Billerica, MA, USA). The software was also used to control the turret movements and to convert the roughness data into high-resolution 2D images. Each bracket was scanned at five random spots and averaged [28]. The roughness measurement was obtained at two intervals, before and after immersion in oral rinses.

2.4 Immersion procedure

Before immersing the randomly selected brackets in the test solutions, they were cleaned ultrasonically in ethanol for 2 min, then rinsed with water and dried with water-oil-free compressed air. Ten brackets from each manufacturer were immersed in glass vials containing either Listerine™ or Orex™ or salt water. The ratio between the volume of the test solutions and the surface area of the bracket specimen was continual (1.00 mL solution per 1 cm^2 of the bracket surface area). The salt water rinse (2%) was readily prepared by dissolving 5.00 g of commercially available NaCl in 250 mL of purified water [23]. The orthodontic brackets were immersed in the respective oral rinses for 28 days in closed glass vials to prevent evaporation of the oral rinses and maintained at 37°C in an

Material	Composition	Manufacturer	Batch No.
Victory Series™ Low Profile orthodontic bracket	Precipitation hardened 17-4 alloy [6,29,30] Fe: 70%, Cr: 15.5–17.5%, Ni: 3–5%, Cu: 3–5%, Si: <1%, Mn: <1%, C: <0.07%, P: <0.04%, S: <0.04%, Nb: <0.3%, Al: 0.67%, Others: 0.05%	3M Unitek, Monrovia, CA, USA	LX2MH
Mini Diamond Twin™ orthodontic bracket		Ormco, Glendora, CA, USA	071723188
Listerine™ fluoride mouthwash	Water, propylene glycol, sorbitol, poloxamer 407, sodium lauryl sulfate, eucalyptol, benzoic acid, sodium benzoate, methyl salicylate, thymol, sodium saccharin, menthol, sucralose, aroma, and sodium fluoride (220 ppm F^-)	Johnson & Johnson, Neuss, Germany	691760
Orex™ fluoride mouthwash	Water, sorbitol, PEG-40 hydrogenated castor oil, glycerin, thymol, poloxamer 407, aroma, sodium methylparaben, sodium propylparaben, citric acid, cetylpyridinium chloride, sodium benzoate, sodium saccharin, and sodium fluoride (225 ppm F^-)	Manufactured for Nahdi Medical Co. by Fushima, SL, Cantabria, Spain	230241
Salt water oral rinse	5.00 g of NaCl dissolved in 250 mL of distilled water (2% saline)		

Key: ppm F^- = parts per million of fluoride.
Table 1. Materials used in the laboratory study.

incubator (Thermo Fischer Scientific, Waltham, MA, USA). The pH of the oral rinses before immersion was 5.1, 7.2, and 6.8 for Listerine™, Orex™, and salt water rinse, respectively (Benchtop Meters: pH Basic Series, Sartorius Lab Instruments, Göttingen, Germany).

2.5 Analysis of the metal ion leaching using ICP-MS

The elemental analysis for ion release was performed according to the International Organization for Standardization (ISO) standard 10271:2011, which defines the corrosion test methods of metallic dental materials [6]. The oral rinses recovered at the end of the 28-day immersion period were quantitatively analyzed for the metal ions with ICP-MS (iCAP™ RQ, Thermo Fisher Scientific, Waltham, MA, USA) [6,7]. The ICP-MS parameters included 10 keV applied voltage and a 0.05 ppb (parts per billion) detectable limit.

2.6 Qualitative and quantitative evaluation of brackets with SEM equipped with EDS

The representative orthodontic brackets from each subgroup were analyzed using SEM (NeoScope JCM-6000Plus, JEOL, Tokyo, Japan) equipped with a fully integrated EDS. Surface micrographs of the noticeable corrosion (COR) and the adjacent corrosion-free (COR-F) spots on the bracket slot (rectangular area as in Figure 1) either on right or left side were imaged using SEM for qualitative analysis. The COR-F and COR spots were distinguished as per a previous study [6]. The COR-F spots were imaged if it did not demonstrate any corrosion signs, plaque, or debris. The microanalysis was performed using the SEM equipped with EDS to determine the chemical elemental composition of the distinguished local COR-F and COR bracket spots. The SEM was operated at ×500 magnification in a vacuum, 15 kV voltage, and 0–20 keV energy range.

2.7 Statistical analysis

Statistical Package for the Social Sciences software (v.21, IBM® SPSS®, Chicago, IL, USA) was used to analyze the available data. The continuous variables were expressed as mean values and standard deviation (SD), and one-way analysis of variance was used to compare the means. Tukey's *post hoc* test was applied to determine if a significant difference exists between the groups. A paired *t*-test was used to compare the groups regarding surface roughness, metal ions leaching, and chemical element analysis between oral rinses and bracket systems ($\alpha = 0.05$).

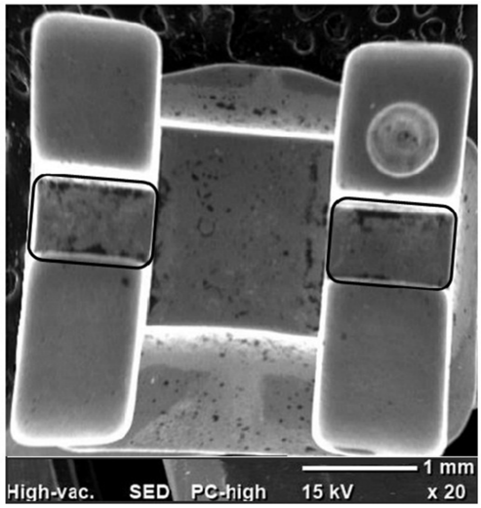

Figure 1. Bracket slot (marked rectangular area) used for imaging of COR-F and COR spots.

3. Results

3.1 VHN of the tested brackets

Table 2 presents the VHN of the orthodontic brackets at baseline and after exposure to oral rinses. The baseline VHN of Victory and Twin Diamond brackets were 312.76 ± 1.86 and 300.55 ± 2.89, respectively. For the Victory brackets, the immersion of orthodontic brackets in Listerine and Orex oral rinses produced significantly lower hardness ($P = 0.002$) compared to baseline VHN, while there was no significant difference between baseline and salt water oral rinses ($P > 0.05$). For the Twin Diamond brackets, the immersion of orthodontic brackets in Orex oral rinses produced significantly lower hardness ($P = 0.04$) compared to baseline VHN, while there was no significant difference between baseline and Listerine or salt water oral rinses ($P > 0.05$).

3.2 Surface roughness (R_a)

Table 3 presents the mean R_a values before and after immersion in oral rinses for the two groups of the orthodontic brackets. For the Victory™ brackets, the mean R_a was around 0.2 µm, but after immersion, the R_a of the

Brackets	Hardness				
	Baseline	Listerine™	Orex™	Salt water	
Victory™	312.76 ± $1.86^{a,A}$	301.52 ± $2.98^{b,A}$	303.9 ± $6.66^{b,A}$	312.20 ± $5.47^{a,A}$	$P = 0.002^*$
Twin Diamond™	300.55 ± $2.89^{a,B}$	295.18 ± $2.84^{a,b,B}$	294.5 ± $2.32^{b,B}$	298.74 ± $4.66^{a,b,B}$	$P = 0.03^*$
	$P < 0.05$				

*Statistically significant ($P < 0.01$).
Different lower-case letters between the oral rinse groups for the bracket system implies significant difference in R_a between them ($P < 0.01$).
Different upper-case letters between the bracket groups implies significant difference in R_a before and after immersion ($P < 0.01$).
Table 2. VHN of the tested brackets at baseline and after exposure to oral rinses.

brackets increased significantly for all the tested oral rinses ($P < 0.01$). Orthodontic brackets immersed in Orex™ and salt water demonstrated the highest (0.93 ± 0.02 µm) and lowest (0.65 ± 0.07 µm) roughness, respectively. For the Mini Diamond™ brackets, the mean R_a before immersion was around 0.3 µm, but after immersion, the R_a of the brackets increased significantly for all the tested oral rinses ($P < 0.01$). Orthodontic brackets immersed in Listerine™ and salt water demonstrated the highest (1.23 ± 0.03 µm) and lowest (0.76 ± 0.08 µm) roughness, respectively. The post-immersion roughness did not differ significantly between Listerine™ and Orex™ compared to the salt water group for both the brackets evaluated ($P > 0.01$).

Immersion	Victory™			
	Listerine™	Orex™	Salt water	
Before	0.21 ± $0.02^{a,A}$	0.20 ± $0.04^{a,A}$	0.21 ± $0.01^{a,A}$	$P = 0.89$
After	0.92 ± $0.01^{a,B}$	0.93 ± $0.02^{a,B}$	0.65 ± $0.07^{b,B}$	$P < 0.01^*$
	$P < 0.01$			
	Twin Diamond™			
	Listerine™	Orex™	Salt water	
Before	0.35 ± $0.05^{a,A}$	0.37 ± $0.04^{a,A}$	0.35 ± $0.05^{a,A}$	$P = 0.77$
After	1.23 ± $0.03^{a,B}$	1.16 ± $0.01^{a,B}$	0.76 ± $0.08^{b,B}$	$P < 0.01^*$
	$P < 0.01$			

*Statistically significant ($P < 0.01$). Different lower-case letters between the oral rinse groups within the bracket system implies significant difference in Ra between them ($P < 0.01$).
Different upper-case letters between the brackets groups implies significant difference in Ra before and after immersion ($P < 0.01$).
Table 3. Surface roughness (R_a in µm) of the bracket surface before and after immersion in media.

Comparing the R_a between the brackets, the R_a before immersion significantly differed for the tested bracket systems. After immersion, the difference in R_a of both bracket systems was statistically significant for Listerine™ and Orex™ rinses ($P > 0.01$). However, no significant difference was found between the salt water rinse ($P < 0.01$).

3.3 Metal ion leaching obtained via ICP-MS

The most apparent metal ion concentration in the test solutions that were extracted after 28 days of bracket immersion is presented in Figure 2. The metals Fe, Cu, Cr, and Ni were the most detectable, in addition to the detected trace amounts of Al, Zn, and Mn. The Al^{3+} and Zn^{2+} ions, not listed as the bracket constituents by the manufacturer, were also present in the corrosion test solutions. The concentration of Fe^{2+} ions in the test solutions was significantly higher than that of other ions present, which were directly related to the Fe composition. Among the oral rinses used, salt water demonstrated comparatively lower ion concentration than the NaF-based oral rinses for both tested brackets. However, only Victory™ brackets demonstrated significantly fewer ions than Mini Diamond™ brackets in salt water except for Ni ($P < 0.05$).

The comparison of the metal ion concentration between the oral rinses showed significant differences for Cu^{2+} and Zn^{2+} ions in Listerine™ and all ions except Ni for salt water. On the contrary, the comparison between the bracket systems for the metal ions showed no significant difference in the Orex™ group ($P > 0.05$).

3.4 Qualitative SEM micrograph analysis

The representative SEM micrographs of COR spots and the adjacent COR-F spots of the Victory™ and Mini Diamond™ brackets immersed in different oral rinses are presented

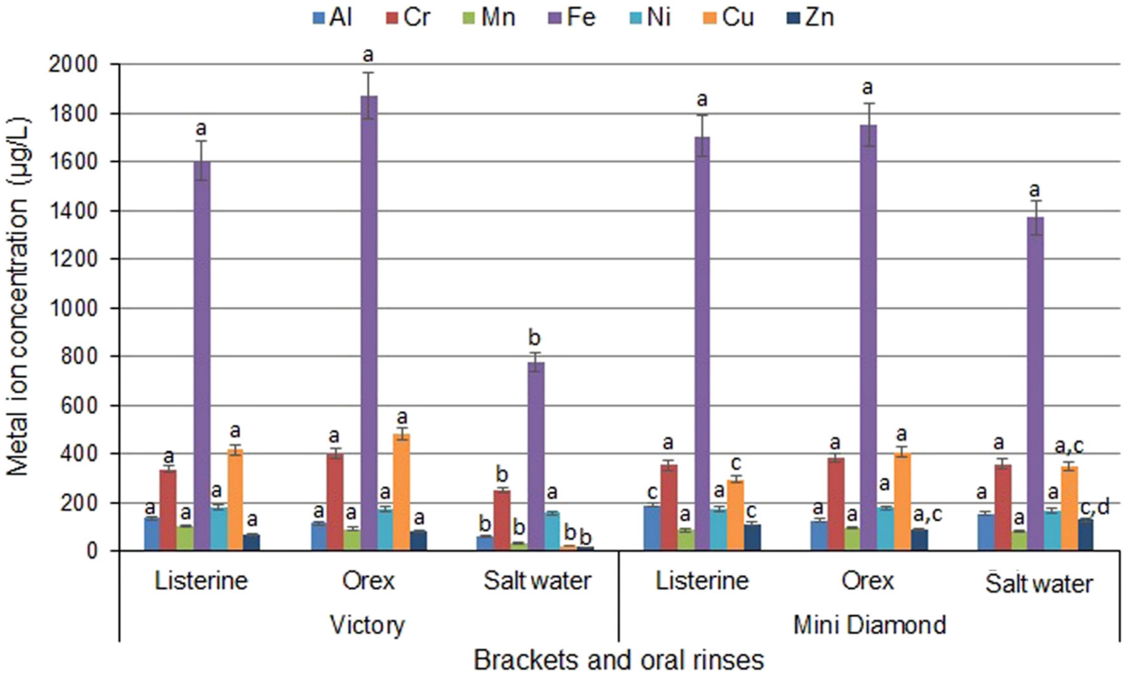

Figure 2. Mean values and standard deviations of metal ion concentration in the test solutions. Different lower-case letters between the oral rinse groups within the bracket system imply a significant difference for the listed metal ion ($P < 0.01$).

in Figures 3 and 4, respectively. The morphological features on the COR spots revealed a superficial granulated spot, with the formation of multiple pitting corrosion and release of metal scraps from the surface of the bracket. On the contrary, adjacent COR-F spots revealed irregular surfaces, which could be related to the manufacturing defects.

Regarding the groups, significant changes occurred with Victory™ brackets immersed in Orex™ (Figure 3) and Mini Diamond™ brackets immersed in Listerine™ (Figure 4). The Victory™ brackets immersed in salt water (Figure 3) showed minor surface morphological changes, followed by Mini Diamond™ immersed in salt water (Figure 4), compared to the other study groups.

3.5 Chemical elemental composition analysis via EDS

Table 4 presents the concentrations of the most apparent chemical elements (Cr, Mn, Fe, Co, and Ni) at the COR-F and suspected COR spots of the brackets obtained via EDS. The elemental concentrations significantly decreased from COR-F to COR spots for both bracket systems.

In the Victory™ brackets, there was no significant difference between the oral rinses for Co^{2+} and Ni^{2+}. However, a significant difference was found for Cr^{2+} and Fe^{2+} between salt water and NaF-based oral rinses ($P < 0.01$). There was no significant difference between the oral rinses for Mn^{2+}, Co^{2+}, and Ni^{2+} in the Mini Diamond™ brackets. However, a significant difference was found for Cr^{2+} and Fe^{2+} between salt water and NaF-based oral rinses ($P < 0.01$).

Figure 5 presents the representative EDS spectra to demonstrate COR-F and COR spots of brackets. The EDS spectra showed the presence of solid peaks of Fe followed by Cr, Co, and Ni. Small amounts of Si, S, Mn, and Cu were also detected in the COR-F spots of brackets. The COR spots, irrespective of the oral rinses, showed declined Fe, Cr, and Ni peaks.

Table 5 presents the % mean loss of Fe^{2+}, Ni^{2+}, and Cr^{2+} ions from COR-F to COR surfaces. For Fe^{2+} ions, no difference was observed in the % mean loss between Listerine™ and Orex™ in both bracket systems. However, a significant difference was observed in the % mean loss of Fe between the bracket systems for the oral rinses except for salt water rinse ($P = 0.976$). For Ni^{2+} ion, no difference was observed between the oral rinses for the % mean loss of Ni in both bracket systems or between the bracket systems ($P > 0.05$). For Cr^{2+} ion, no difference was observed between Listerine™ and Orex™ in both bracket systems for the % mean loss. However, significant difference was observed between the bracket systems for the oral rinses except for Orex™ rinse ($P = 0.619$). The highest mean % loss of Fe^{2+} (98%), Ni^{2+}

(99.4%), and Cr^{2+} ions (98.95%) was seen with Mini Diamond bracket system immersed in Listerine™.

4. Discussion

The current *in vitro* study analyzed and compared qualitatively and quantitatively the surface roughness and metal ion leaching of two brands of orthodontic brackets exposed to commercially available NaF-based oral rinses and salt water oral rinse. The study hypothesis was that there would be no significant difference in the roughness and metal ion leaching of brackets exposed to salt water or NaF-based oral rinses. The outcome of the statistical analysis suggests partial rejection of the study hypothesis.

Previous studies have reported beneficial effect of salt water oral rinse. Kim and Kim [24] demonstrated that salt water oral rinse at a particular concentration can improve oral hygiene. The authors also demonstrated that salt water oral rinse would reduce xerostomia, halitosis, and considerably reduce the bacterial load in the oral cavity of older adults living in long-term care institutions. On the other hand, Collins et al. [23] compared the anti-inflammatory effect of oral rinses containing NaCl and 0.12% chlorhexidine (CHX) in patients receiving minimally invasive periodontal surgery. The authors reported that using salt water (saline) as an oral rinse had an anti-inflammatory effect comparable to 0.12% CHX. Similarly, Aravinth et al. [31] assessed the effectiveness of salt water rinse in conjunction with CHX mouth rinse in lowering dental plaque

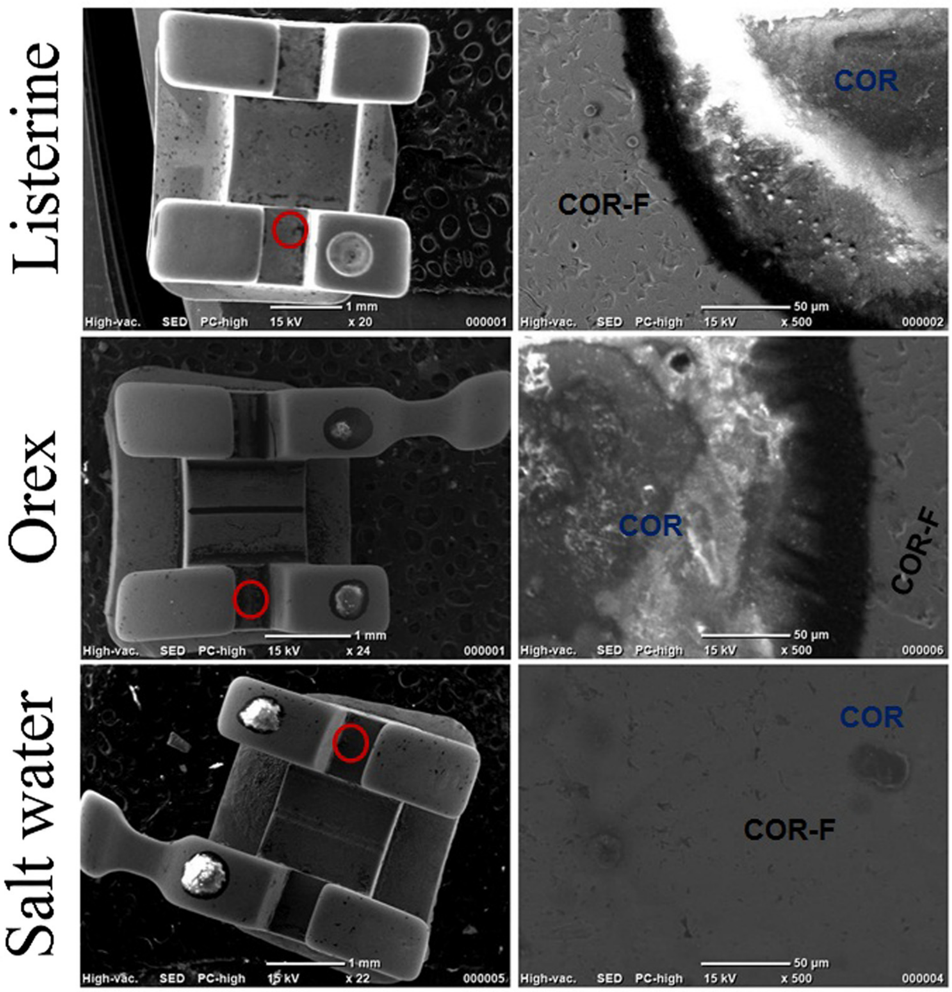

Figure 3. SEM micrographs demonstrating the noticeable COR and adjacent COR-F spots of the Victory™ brackets (magnification ×500). The red circle indicates the bracket slot area used for imaging of COR-F and COR spots.

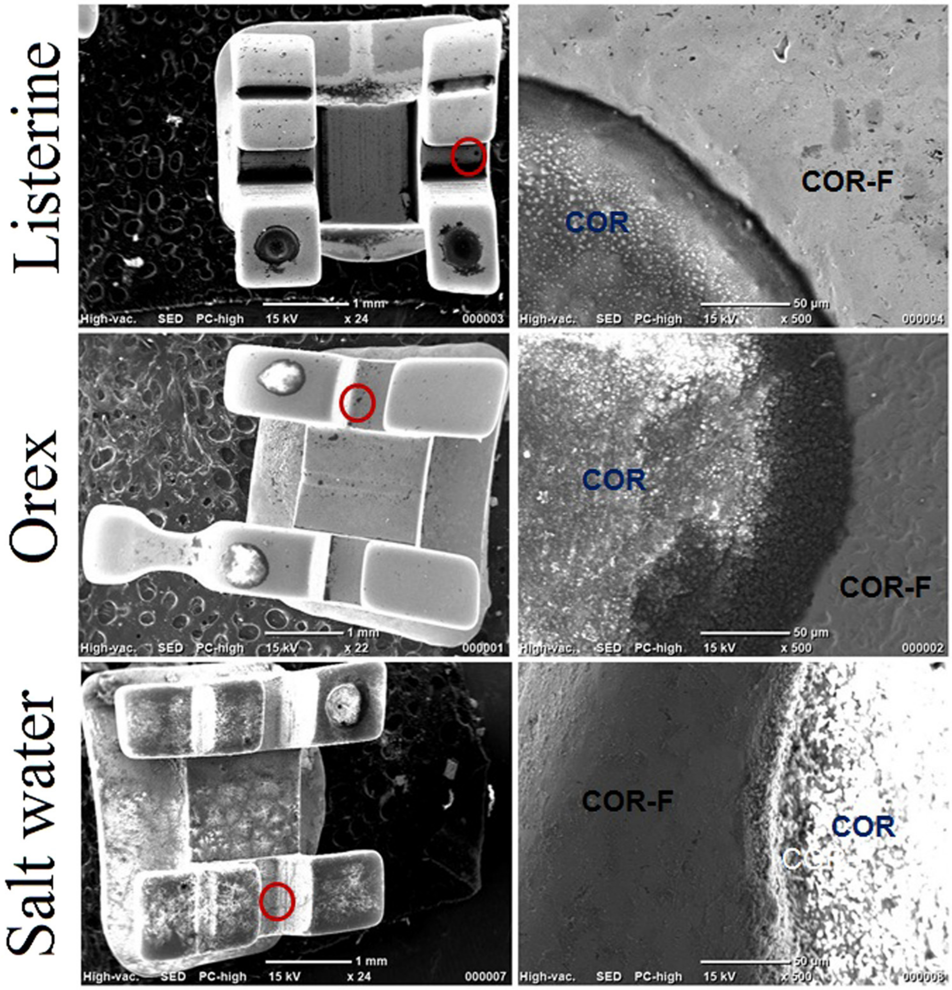

Figure 4. SEM micrographs demonstrating the noticeable COR and adjacent COR-F spots of the Mini Diamond™ brackets (magnification ×500). The red circle indicates the bracket slot area used for imaging of COR-F and COR spots.

	Oral rinses	Cr	Mn	Fe	Co	Ni
Victory	COR-F	16.6 ± 1.41[a]	2.6 ± 0.20[a]	64.3 ± 1.01[a]	5.8 ± 0.58[a]	4.6 ± 0.68[a]
	Listerine™	2.3 ± 0.08[b]	0.9 ± 0.06[b]	8.7 ± 0.14[b]	0.1 ± 0.07[b]	0.08 ± 0.04[b]
	Orex™	2.8 ± 0.09[b]	0.4 ± 0.09[c]	1.2 ± 0.01[c]	0.04 ± 0.04[b]	0.9 ± 0.008[b]
	Salt water	11.7 ± 0.11[c]	0.08 ± 0.04[c]	18.6 ± 0.58[d]	0.50 ± 0.02[b]	1.3 ± 0.05[b]
Mini Diamond	COR-F	16.3 ± 0.53[a]	3.3 ± 0.15[a]	66.1 ± 0.82[a]	5.8 ± 0.37[a]	4.8 ± 0.79[a]
	Listerine™	0.2 ± 0.03[b]	0.09 ± 0.04[b]	1.3 ± 0.12[b]	0.04 ± 0.02[b]	0.03 ± 0.01[b]
	Orex™	2.4 ± 0.07[c]	0.1 ± 0.05[b]	2.7 ± 0.17[b]	0.4 ± 0.04[b]	1.8 ± 0.12[b]
	Salt water	4.5 ± 0.02[d]	0.1 ± 0.04[b]	30.4 ± 1.34[c]	0.5 ± 0.11[b]	1.8 ± 0.09[b]

Different lower-case letters between the oral rinse groups within the bracket system imply a significant difference for the listed metal ion ($P < 0.01$).

Table 4. Chemical element concentrations of the COR-F and COR spots of the brackets obtained via EDS.

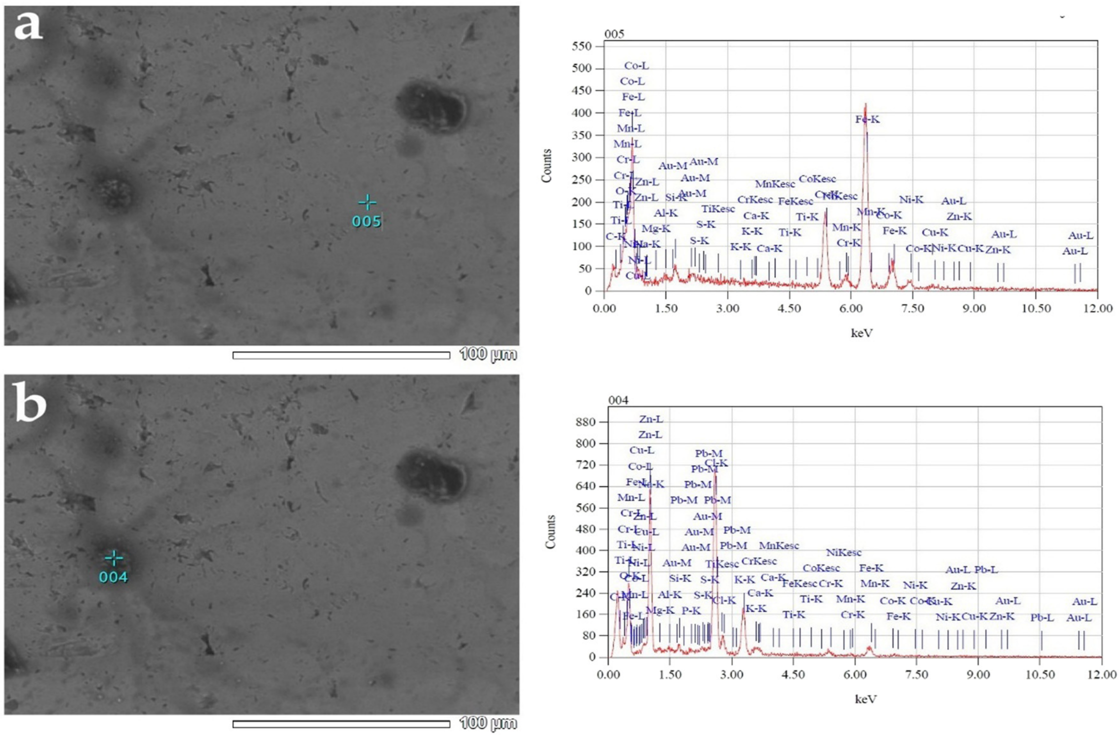

Figure 5. Chemical composition of COR-F (a) and COR (b) spots of the brackets as analyzed by EDS.

and oral bacteria count. They concluded that salt water rinsing can be an adjuvant to regular mechanical plaque treatment to prevent oral diseases.

The VHN of Twin Diamond and Victory brackets was 300.55–312.76 at baseline. Although both the brackets were manufactured by the MIM method, the difference in the hardness between the brackets was statistically significant, which could be related to the minor differences in the elemental composition of the brackets. It was earlier reported that the VHN of the orthodontic brackets manufactured by the MIM method varied between 154 and 287 VHN [17], which is consistent with the values obtained in our study. After immersion, the brackets immersed in Listerine and Orex oral rinse

Metal ions	Oral rinses	Victory			Mini Diamond			*P*-value
		Mean diff.	SE	Loss (%)	Mean diff.	SE	Loss (%)	
Fe^{2+}	Listerine™	45.6	0.5	71.0	64.8	0.4	98.0	$P < 0.0001$*
	Orex™	53.0	0.5	82.5	63.5	0.4	96.0	$P < 0.0001$*
	Salt water	35.7	0.6	55.6	35.7	0.9	54.0	$P = 0.976$
Ni^{2+}	Listerine™	4.5	0.3	98.2	4.8	0.4	99.4	$P = 0.418$
	Orex™	3.7	0.3	80.5	3.1	0.4	63.4	$P = 0.528$
	Salt water	3.3	0.3	72.4	3.0	0.4	62.6	$P = 0.131$
Cr^{2+}	Listerine™	14.3	0.8	86.2	16.1	0.3	99.0	$P = 0.0238$*
	Orex™	13.9	0.8	83.4	14.1	0.3	86.9	$P = 0.619$
	Salt water	4.9	0.8	29.3	11.7	0.3	72.2	$P = 0.0002$*

*A statistically significant difference in the mean difference of the respective metal ions between the bracket types for the oral rinses groups (paired *t*-test; $P < 0.05$).

Table 5. Mean difference and % loss of Fe^{2+}, Ni^{2+}, and Cr^{2+} ions from COR-F to COR spots of the brackets immersed in oral rinses.

demonstrated significantly lower hardness compared to salt water rinse, which did not differ significantly from the baseline values.

A noncontact profilometer was the test tool utilized in this investigation to quantify surface roughness. Compared to an atomic force microscopy, this device is quicker, nondestructive, and delivers a broader field without requiring sample preparation or a stylus profilometer [27]. The reported clinically acceptable roughness threshold in clinical environment is 0.2 µm (R_a) [27,32]. At baseline, the mean *R*a of Victory™ and Mini Diamond™ brackets was around 0.2 and 0.3 µm, respectively. On the contrary, the R_a of the brackets after immersion in oral rinses increased substantially above the threshold value regardless of the oral rinses and bracket types. The difference in R_a did not vary between Listerine™ and Orex™ oral rinses but significant difference was observed between the NaF-based oral rinses vs salt water rinse for both bracket systems. This shows that salt water rinses produced less surface changes compared to NaF-based oral rinses. The R_a of Mini Diamond™ brackets were significantly higher and different than Victory™ brackets. This confirms that the properties of the two brands of orthodontic brackets with the same formulations may vary. The primary process of fabricating orthodontic brackets using the MIM method involves a sintering temperature of 1,300–1,350°C to achieve full or almost full density and high mechanical characteristics in 17-4 PH SS [17]. However, the processing technique results in surface irregularities and favorable environment for corrosion. This probably explains an increase in R_a above the threshold limit [33].

Regarding biocompatibility, the most corrosion-resistant SS brackets are essential for clinical applications. Therefore, while selecting commercially available orthodontic brackets on the market, orthodontists should also consider the metal ion leaching of the different orthodontic brackets [10]. Previous studies have reported that the sintered 17-4 PH SS has a tendency to pit and corrode, especially when exposed to saltwater solution (>3%) and could impact the mechanical qualities and corrosion resistance of the final product [14,26]. Therefore, it was expected that saltwater oral rinse would produce more changes than the NaF-based oral rinses. On the contrary, NaF-based oral rinses demonstrated comparatively more ion leaching than the salt water oral rinses for both tested brackets. The comparison of the metal ion concentration between the oral rinses showed significant differences for Cu^{2+} and Zn^{2+} ions in Listerine™ and all ions except Ni^{2+} for salt water. Regarding SS, Cr creates a thin, adherent protective layer based on chromium trioxide (Cr_2O_3), which gives the substrate alloy its corrosion resistance, and this layer is formed with a minimum of 11% Cr [34]. On exposure to fluoride rinses, the chemical reaction between NaF and the passive protective oxide coatings (Cr_2O_3) on the SS surface causes the corrosive effects of fluoride products, as shown below [35,36].

$$Cr_2O_3 + 2NaF \rightarrow CrF_2 + Na_2O + CrO_2. \quad (1)$$

Although the brackets in salt water showed comparatively less ion leaching, only Victory™ brackets demonstrated significantly less ion leaching compared to Mini Diamond™ brackets in salt water, except for Ni^{2+} ions. It has been stated that the nature of the protective layer on the alloy surface depends on the compositional elements of the alloys [37]. However, the metal ion leaching may not always be related to the alloys' metal ratios [33].

The pH of the oral rinses was 5.1, 7.2, and 6.8 for Listerine™, Orex™, and salt water rinses, respectively, and it was anticipated that the oral rinses' pH would affect the metal ion leaching. However, the Orex™ rinse with a pH higher than the other two oral rinses demonstrated more metal ion leaching. This confirms that the corrosion process is multi-factorial and does not depend on individual factors. Moreover, the current study showed that metal ion leaching also occurred when the brackets were exposed to salt water. The most significant corrosion products of SS are Fe, Cr, and Ni ions, which have a potentially harmful effect on the body. However, Cr^{2+} and Ni^{2+} ions are profoundly significant because they have been reported to trigger allergic, toxic, and even carcinogenic reactions [10]. The permissible daily allowance of Fe, Cr, and Ni is 8, 50–200, and 25–35 mg, respectively [12,13,33]. This study's calculated daily release of Fe^{2+}, Cr^{2+}, and Ni^{2+} ions was less than the permissible allowance, regardless of the bracket types and mouth rinses, implying a lack of toxicity. However, a slow but continual release of corroded metal ions from the brackets would likely accumulate in the gingival fibroblast cells, where it could initiate inflammation or inflict DNA or cellular damage [12,33].

The 17-4 PH SS is commonly used for fabricating the so-called "mini brackets" because of its high tensile strength. The tested orthodontic brackets in this study, Victory™ and Mini Diamond™, were from two manufacturers but were fabricated using the MIM process using the 17-4 PH SS material. The brackets used had a high percentage of Fe (70%), which has a high potential to corrode [38]. The representative EDS spectra showed the presence of solid peaks of Fe followed by Cr, Co, and Ni at COR-F spots. The COR spots, however, irrespective of the oral rinses, showed declined Fe, Cr, and Ni peaks. The EDS analysis showed no significant difference in the reported elemental concentration at COR-F spots between the two bracket brands. The difference in elemental composition (Fe, Cr, and Ni) of COR spots after immersion in oral rinses showed significant differences for Listerine™ and Orex™ oral rinses for Fe and both Listerine™ and salt water oral rinses for Cr. However, there was no significant difference between the bracket systems for any of the oral rinses for Ni. This implies that electrochemical and biocompatibility properties can vary between SS alloys, even with the same processing method [39]. SEM images of the noticeable COR surface of the bracket presented with localized, superficial granulated spots, with formation of multiple pitting corrosion and release of metal scraps. The SEM analysis also demonstrated that the corrosion behavior of the surfaces differed between the bracket systems. Moreover, the SEM surface changes was not in agreement with the ion release from the COR spots. Furthermore, the SEM images obtained with high magnification (×500) limits the generalizability of the changes as the small imaging area may not be a representative of the entire bracket.

This laboratory study has few strengths and limitations. The strengths of this study are as follows: this is the first study to evaluate the effect of salt water on the surface characteristics and corrosion behavior of orthodontic brackets manufactured by the MIM method. The employed qualitative and quantitative analysis gives a clear and reliable understanding of the tested brackets' metal ion leaching and corroded surface. The outcome of the current study will eventually help clinicians understand the corrosion behavior of the commercially available brackets, make selection of the brackets, and recommend optimal oral hygiene practices for their orthodontic patients, ultimately improving oral health outcomes.

On the contrary, there are a few limitations. This study does not completely simulate the corrosive oral environment, *viz.* the brackets were immersed in the oral rinses for 28 days, which is not the actual situation in the oral environment for orthodontic treatment. The role of saliva in clearing the ion concentration was not considered. That said, the ion concentration might be somewhat lower than what we have reported. Furthermore, the interaction of the ions with cells and tissue in the oral environment was not studied, but belongs to our near future plans. Although the average comprehensive orthodontic treatment is up to 2 years, the orthodontic appliance may remain in the patient's mouth for several years, which may present with more corrosive behavior. This is why initial metal ion dissolution data are justified. Finally, the data obtained in this study cannot be necessarily compared with other studies as this is the first report on the effect of salt water on the surface characteristics and corrosion behavior of metallic orthodontic brackets.

In the near future, clinical trials should be performed to analyze the efficacy of salt water rinse on orthodontic brackets to corroborate the findings of this study. Also, it would be interesting to study the effect of salt water on the corrosion behavior of orthodontic brackets manufactured using other techniques and different alloys.

5. Conclusion

Based on the laboratory work, the following conclusions are made.

(a) The study outcome showed that salt water oral rinse demonstrated significantly fewer surface changes than NaF-based oral rinses.
(b) Surface roughness significantly varied between the bracket systems. The pre to post-immersion of brackets in oral rinses demonstrated that R_a significantly increased for Listerine™ and Orex™ rinses, but did not vary for the salt water rinse.
(c) The leaching of the metal ions was significant for Fe^{2+} and Cr^{2+} but did not vary considerably for Ni^{2+} between the tested oral rinses.
(d) Irrespective of the oral rinses and brand, the ICP-MS analysis after 28 days of bracket immersion showed that the amounts of Fe, Cr, and Ni were still below the permissible daily allowances.

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Author contributions

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Conflict of interest statement

Authors state no conflict of interest.

Data availability statement

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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