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An Evaluation of Corrosion on Commercially Available Orthodontic Brackets Using UV-VIS Spectrophotometre and Small Angle Xray Scattering: An In Vitro Study

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Abstract: The aim of this study was to compare three types of commercially available brackets for corrosion susceptibility after immersing them in artificial saliva. A total of 360 brackets were used. The brackets were divided in to four groups (Control Group X and Experimental Groups A, B and C). The experimental groups were divided on the basis of duration of immersion in artificial saliva of pH 6.75 ± 0.15. Each group was divided in to corresponding subgroups. The bracket surfaces were analysed using UV –Visible Spectrophotometer and Small Angle Xray Scattering (SAXSS). ANOVA (Analysis of One Way Variance) was used for group comparisons. For post hoc comparisons Newman-Keuls multiple posthoc procedure was applied. Leone and Nu Edge Cr- Co brackets showed progressive signs of corrosion and leaching of Fe and Mg ions after immersion in artificial saliva. Discovery SL brackets showed no signs of corrosion. Leone and Cr-Co Nu Edge are susceptible to corrosion activity in comparison to Discovery SL brackets.
Keywords: Artificial Saliva, UV –Visible Spectrophotometer, Small Angle Xray Scattering (SAXSS)

INTRODUCTION

Corrosion is a complex chemical process that occurs from either loss of metal ions directly into solution or progressive dissolution of surface film, usually an oxide or a sulphide.[1] Corrosion can be manifested in many forms such as Fretting corrosion [1], Galvanic [2], Stress and Microbiologically influenced corrosion [2], Pit and crevice [3] corrosion .Corrosion of orthodontic appliances [1] in the oral environment has concerned clinicians for some time as it effects the maximum efficiency of the orthodontic appliances by altering there morphology and bio-compatiblity .

This concern has focused around 2 principal issues: First, whether corrosion products which are absorbed into the body produce localized or systemic effects; and seconds the effects of corrosion on the physical properties and / or clinical performance of orthodontic appliances. Orthodontic brackets used in the correction of malocclusion need to be of a suitable material which does not corrode and release metallic ions when it is bonded in the oral cavity [4, 5]. Various metallic materials are used for fabrication of fixed orthodontic appliances such as Fe-Cr-Ni–based stainless steel (SS), Co Cr, Ti. Noble metals which are virtually inert; eg, gold and platinum are rarely used in orthodontic appliances while SS which is the most commonly used, has the highest propensity for corrosion. In order to evaluate the susceptibility of materials to corrosion laboratory studies have been conducted by exposing orthodontic brackets in an artificial medium for extended periods which would have the equivalent effect of being in the oral cavity. The artificial mediums that have been used in the past include 0.9% sodium chloride solution [6-9], 1% NaCl solution [10], 0.2% chlorhexidine gluconate, 5.25% NaOCl, chlorinated soda with KOH, and 17% EDTA irrigating solutions [10], Fusayama-Meyer artificial saliva [11] and Artificial saliva (pH 6.5) [12] with various NaF concentrations (0%, 0.01%, 0.1%, 0.25%, and 0.5%) [13]. Scientific studies in the past have operated such equipment including SEM [14, 15] and Atomic force microscopy [16] to check the morphology of wires and brackets before and after immersing in artificial saliva. UV-Vis Spectrophotometer [17] was carried out to measure the % Reflectance of surface against the surface homogeneity of brackets after immersing in artificial saliva. Linear polarization test [18], a fast electrochemical technique, was used to

evaluate the corrosion resistance, in terms of polarization resistance (R(p)) of brackets. Atomic absorption spectroscopy [15-17], and Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) [17, 19] have also been used to quantify the leaching of metallic ions in artificial saliva in order to investigate the clinical implications of metallic ions such as Ni.

The aim of this study was to evaluate the corrosion of commercially available three orthodontic bracket systems through scientific measurements such as, SAXSS and UV-Vis Spectrophotometer for qualitative and quantitative analysis so as to establish that which is the best available orthodontic bracket for the clinician to use in his/her daily practice, to understand what is the best combination of materials which has minimal leaching and alteration in surface morphology which should serve as a "benchmark " in bracket fabrication and production .

MATERIALS AND METHODS

In the present study 360 commercially available brackets were used. The brackets were of three types (120 each) as in described in Table 1.

Table-1: Bracket Types and Composition

Bracket Type	AISI/DIN Number	Composition
Leone S.p.a, Freinze, Italy (SS Bracket)	304L	FeCrNi = Fe: 65.5 , Cr: 18.5 , Ni : 12.3 , Mo: 2.4 by % weight
Discovery SL DentaurumGmbh, Germany (SS Bracket)	1.4404 (equivalent AISI 316L)	C < 0.03 Si < 1 Mn< 2 P< 0.045 S< 0.015 Cr 16.5-18.5 Mo 2 - 2.5 Ni 10 - 13 others N < 0.11 by % weight
Nu Edge Cr- Co brackets TP Orthodontics Inc, USA (CoCr Bracket)		CoCr = Co: 60.3, Cr: 31.6, Mo: 8, Fe: 1.2.

The orthodontic brackets belonging to three different categories were divided in to 4 groups. Control Group X (90 Brackets) and Three Experimental groups A, B & C (90 brackets each). (A, B and C) were kept in controlled conditions in an incubator and then analysed by Scanning Electron microscopy, Small Angle X-ray Scattering and UV-Vis Spectrophotometer. The duration of the study was for the experimental groups A, B and C being evaluated at 30, 60 and 90 days respectively.

Brackets of Control Group X were analysed by Small Angle X Ray Scattering and UV spectrophotometer for the surface integrity of the brackets.

Brackets of Group A, B & C were autoclaved and stored glass test tubes (Borosil ®). Artificial saliva was prepared with a pH 6.75 ± 0.15 and composed of (0.4 g NaCl, 0.4 g KCl, 0.80 g $CaCl_2$ $2H_2O$, 0.01 g Na_2S_5H2O, 1.0 g CO (NH2)2 (Urea), 1 L distilled water). The artificial saliva was prepared at Spectro Labs, Okhla. , New Delhi.

The Artificial saliva was stored in 1L glass beaker (Borosil ®).The brackets of groups A, B & C were immersed for a period of 30, 60 and 90 days, and maintained at 37 degrees Celsius in an incubator (Khera Instruments Pvt. Ltd, Delhi).After the immersion period, the brackets were removed from the tubes with and washed in triple distilled water , dried and stored in airtight tubes marked as per bracket system as also the artificial saliva of each tube with corrosive products, at 4 degree Celsius until analysis. Small Angle X- Ray Scattering, model (SAXSess mc^2, with sealed-tube X-ray generator, Anton Paar GmbH, USA) of SMITA Labs Pvt Ltd , IIT Delhi was used to examine the surface integrity of the brackets of Groups X ,A , B and C .The UV –Vis Spectrophotometer(UV- Visible Spectrophotometer Lambda 35 Perkin Elmer, Waltham, Massachusetts, USA) of SMITA Labs Pvt Ltd , IIT Delhi was used for analysing the bracket samples to measure the surface reflectance of the bracket surface.

To further analyse the result Analysis of One Way Variance (ANOVA) and Newman-Keuls multiple posthoc procedure was carried out for test of significance for all the results obtained. The statistical analysis was carried out using SPSS 21 software IBM.

RESULTS

X ray scattering (SAXSS)

The graphs which are produced by the SAXSS machine show the relation between the scattering angle and the intensity of the scattered beam , wherein the intensity is a measure of the energy flow per unit of time and per unit of area of the scattered wave front of X rays. The graphs indicating the comparison between the Control Group X and Experimental Groups A, B and C are shown in Fig-1 and 2 for the various samples.

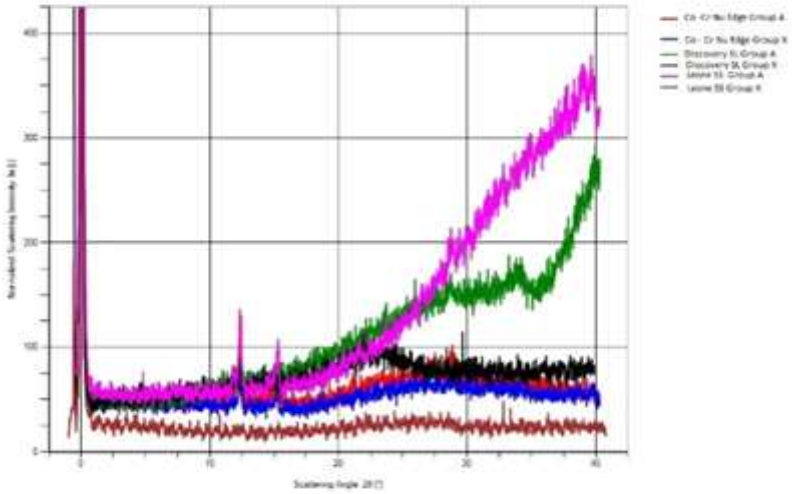

Fig-1: Overall Comparison of SAXSS for brackets in GroupX and Group A

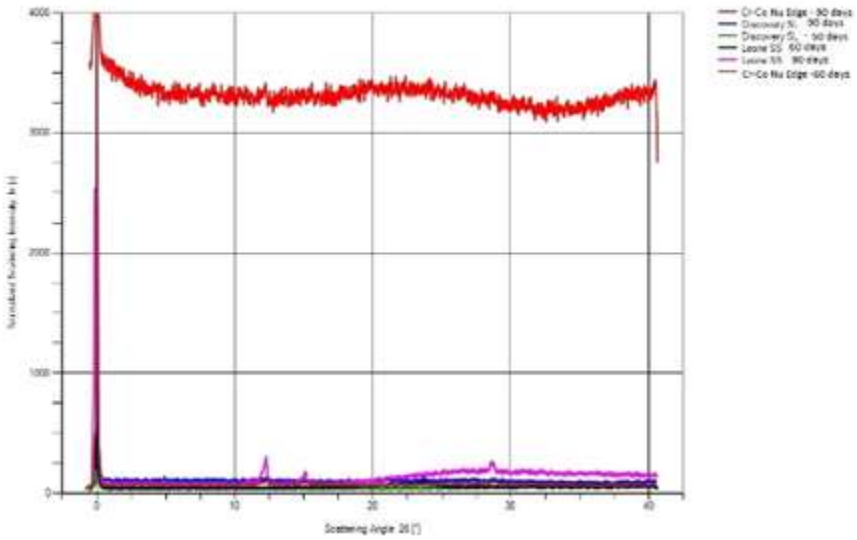

Fig-2: Overall Comparison of SAXSS for brackets in Group B and Group C

The statistical analysis gives us data of scattering intensity versus scattering angle from 2.5 to 38.75 degrees in steps of 1.25 degrees for brackets of Groups X, A, B and C . The results of above statistical calculations have been tabulated in Table 2. To further analyse the result Analysis of One Way Variance (ANOVA) and Newman-Keuls multiple posthoc procedure was carried out for test of significance which revealed *p<0.05 for scattering intensity in all the groups and for all types of brackets

Table-2: Summary of Results of SAXSS

Sl No	**Leone**				**Discovery**				**Cr- Co**			
Groups	**Gp X**	**Gp A**	**Gp B**	**Gp C**	**Gp X**	**Gp A**	**Gp B**	**Gp C**	**Gp X**	**Gp A**	**Gp B**	**Gp C**
No of steps of Scattering angle	30	30	30	30	30	30	30	30	30	30	30	30
Mean Value of Scattering Intensity*	27.63	135.90	57.97	151.40	81.53	122.37	53.87	115.93	59.00	71.20	80.83	3347.90
SD of Scattering Intensity	6.31	90.50	10.87	57.78	15.68	58.18	9.64	13.33	10.56	17.14	15.93	61.66
Coefficient of Variation%	22.82	66.60	18.75	38.17	19.23	47.54	17.89	11.50	17.89	24.07	19.71	1.84

*Less difference between Mean Value of Scattering Intensity of Experimental Groups of the three brackets imply less corrosion

UV-VIS spectrophotometer

The results of UV –Vis Spectrophotometer are represented graphically showing varying values of reflectance for wavelengths ranging from 200-800 nm. The graphs generated by the equipment for various bracket from Control Groups X and Experimental Groups A, B & C are shown in Fig-3 & 4.

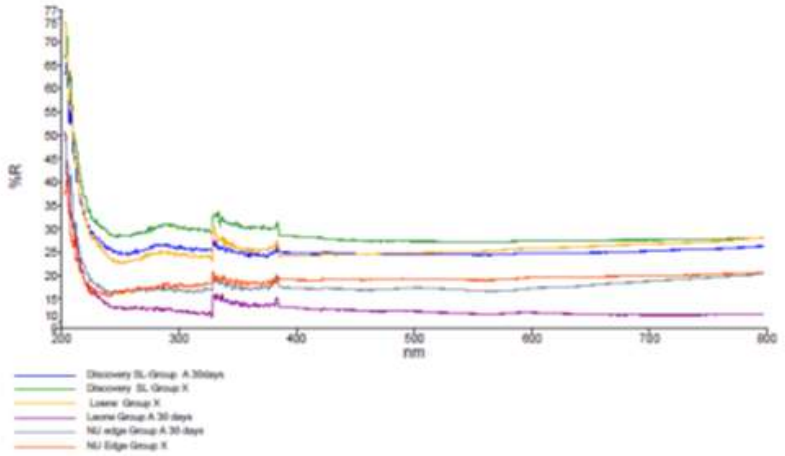

Fig-3: Comparison of UV-VIS Spectrophotometer for brackets in Group X & Group A

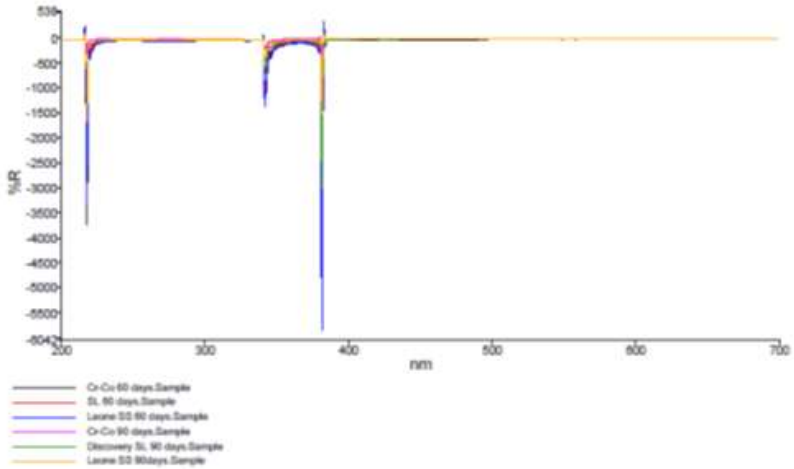

Fig-4: Comparison of UV-VIS Spectrophotometer for brackets in Group B and Group C

The statistical analysis gives data of % R versus wavelength from 200 to 800 nm in steps of 20 nm for brackets of Groups X, A, B and C. The results of above statistical calculations have been tabulated in Table 3. To further analyse the result Analysis of One Way Variance (ANOVA) and Newman-Keuls multiple posthoc procedure was carried out for test of significance which revealed *p<0.05 for % Reflectance in all types of brackets across Control and Experimental Groups.

Table-3: Summary of Results of UV –VIS Spectrophotometer

Sl No				**Leone**			**Discovery**				**Cr- Co**	
Groups	**GpX**	**Gp A**	**Gp B**	**Gp C**	**Gp X**	**Gp A**	**Gp B**	**Gp C**	**Gp X**	**Gp A**	**Gp B**	**Gp C**
No of samples	30	30	30	30	30	30	30	30	30	30	30	30
Mean Value of %R *	25.95	11.76	48.6	25.05	27.45	24.95	20.53	24.95	19.58	17.53	12.11	25.05
SD of %R	1.10	0.35	5.74	0.23	0.33	0.52	18.10	0.23	0.48	1.24	9.18	0.23
Coefficient of Variation % R	4.26	2.96	11.78	0.92	1.20	2.10	88.16	0.92	2.45	7.08	75.81	0.92

* Less difference between Mean Value of %R of Experimental Groups of the three brackets imply less corrosion

DISCUSSION

In order to evaluate the progress of corrosive processes on the surface of brackets different groups of brackets were retained in the artificial saliva medium for 30,60 and 90 days. Similar studies [12-14, 20, 21] have used this technique to measure the leaching of ions such as Fe, Ni and Cr from bracket surfaces. The quantitative results of this In Vitro study revealed that unused saliva clearly indicated absence of any metallic ions and thereby indicating its purity for experimental purposes. The scientific measurements that were carried out to examine the surface homogeneity of brackets included the Small Xray Scattering (SAXSS) and UV – Vis Spectrophotometer for brackets before immersing in artificial saliva and after having kept them immersed in artificial saliva for a fixed time period.

The assessment revealed that Leone SS and Nu Edge Cr –Co brackets have increased corrosion when kept durations of time in artificial saliva. The extent of corrosion also depended on type of steel or its manufacturing processes used which lead to imperfections in the surface and thereby enabled the corrosive process. As regards to Discovery SL brackets, it was noticed there wasn't much change in surface topography with different duration time in artificial saliva medium. This could be attributed to either a higher grade of Stainless Steel used for the bracket by the manufacturer or a higher quality in manufacturing which has definitely resulted in a resolute product. The results of this method were similar to earlier studies [7, 12, 20, 21, 22] carried out which correlated the grade of steel to the rate of corrosion.

The measurement has been based on Small Angle X- ray Scattering (SAXSS) which has been used in scientific studies for probing surface morphology and characterization of pore structure in metal organic framework [23, 24]. However no current literature in orthodontics could be traced and there is paucity of data regarding it application in in vitro testing for orthodontic appliances. The results of SAXSS given in show all three types of brackets Leone, Discovery & Nu Edge brackets have a significance in their readings as the duration has increased which indicates more surface irregularities with time which in turn indicates progressive corrosion. The Leone SS and Cr- Co Nu edge showed higher significance as compared to Discovery SL reconfirming the earlier experimental results that the corrosion was more.

The results of UV – Vis Spectrophotometer showed that only two bracket types (Leone and Nu Edge) had higher significance in their readings in Groups A & C as compared to Group B. There was no significant scores Discovery SL brackets as the % Reflectance values were very high. This was similar to studies in literature which correlated the high significance of % reflectance to corrosion [12].

CONCLUSIONS

1. Quantitative evaluation by SAXSS concluded high significance in all Leone and Nu Edge types of brackets for presence of corrosion.
2. Similarly quantitative evaluation by UV –Vis Spectrophotometer indicated presence of high surface irregularity due to corrosion in Leone SS and Nu Edge Cr –Co Brackets.
3. The In vitro study conclusively brought out the susceptibility of commercially available Leone and Cr-Co Nu Edge to corrosion activity.
4. This in vitro study also gave insight into the working of SAXSS which can be used for testing and evaluating the surface homogeneity for orthodontic biomaterials.

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