The Effect of Artificial Saliva on The Surface Roughness of Different Esthetic Archwires (An *in Vitro* Study)

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ABSTRACT

Background:The demand for esthetic orthodontic appliances is increasing so that the esthetic orthodontic archwires were introduced. This *in vitro* study was designed to evaluate the surface roughness of fiber-reinforced polymer composite (FRPC) archwires compared to coated nickel-titanium (NiTi) archwires immersed in artificial saliva.

Materials and Methods:Three types of esthetic orthodontic archwires were used: FRPC (Dentaurum), Teflon coated NiTi (Dentaurum) and epoxy coated NiTi (Orthotechnology). They were round (0.018 inch) in cross section and cut into pieces of 15 mm in length. Forty pieces from each type were divided into four groups; one group was left dry condition and the other three groups were immersed in artificial saliva (pH=6.75 ± 0.015) at 37°C for 1, 14 and 28 days intervals. The AFM was used to evaluate surface analysis of all samples. ANOVA, Kruskal-Wallis, LSD and Mann-Whitney U tests were used to identify and localize the source of differences among the groups.

Results:At each immersion period, FRPC wires exhibited the highest Ra among the study groups, except at 28 days immersion period where the Teflon coatings were the roughest. On the other hand, the least rough surfaces were the epoxy coatings when compared to analogous esthetic archwires, except at 1 day immersion period where the Teflon coatings had the least roughness. However, statistically non-significant differences were found between Teflon and epoxy at the dry condition and the 1 day immersion.

Conclusions:The epoxy coated archwires were the best and the most appropriate esthetic orthodontic alignment archwires in term of the least surface roughness initially and over the course of study period.

Keywords:Esthetic archwires, fiber-reinforced polymer composite wires, surface roughness, AFM. (J Bagh Coll Dentistry 2017; 29(3):106-112)

INTRODUCTION

With the advent of increasing number of adults seeking orthodontic treatment, the development of orthodontic appliances with ample emphasis on esthetics coupled with optimal performance has become an essential goal or rather necessity of the day⁽¹⁾. It has been partially solved by the introduction of esthetic brackets made of ceramic or composite ⁽²⁾. However, most archwires are still made of efficientunesthetic metal alloys such as stainless steel and nickel-titanium. An esthetic archwire is highly desirable to complement esthetic brackets in clinical orthodontics ^(3,4).

Coating metallic archwires with plastic resin materials were the main solution to provide esthetic characteristics to wires with metallic or silver coloredappearance ⁽⁵⁾. Patients prefer that wires are not apparent or opaque, therefore alternatives could be archwires with transparent or translucent features ⁽⁶⁾. Moreover, esthetic coatings of alloy archwires are not clinically durable and tends to tear over a period of time ^(5,7). Through composite technology, an esthetic wire has been developed from continuous fibers and polymer matrix (tube shrinkage technique), giving rise to the fiber- reinforced polymer

composite (FRPC) archwire which showed promise in its application as an esthetic aligning archwire ^(1,8). The translucent nature of the polymer matrix confers itsesthetic property, whilstthe fiber content gives the material flexibility, overcoming the inherent problem of composite brittleness⁽⁹⁾.

Among the material's characteristics that alter the behavior of the archwires, the surface roughness plays an important role. It is an essential factor in determining the esthetics and color stability of archwires, hygiene, biocompatibility, effectiveness of archwire-guided tooth movement, surface contact and friction, and thereby, the quality of orthodontic treatment⁽¹⁰⁻¹³⁾. Intra-orally placed materials (i.e. wires, brackets) exhibit a pattern of continuous reaction with the environmental factors present in the oral cavity⁽¹⁴⁾. Orthodontic materials are in contact with a variety of substances that impose potent effects on their reactive status and surface integrity such as saliva⁽¹⁵⁾.

Looking at the surface roughness before and after immersion in artificial saliva using atomic force microscopy (AFM) may give more insights to these FRPC archwires and their application in orthodontics compared to their counterparts.

MATERIALSAND METHODS Preparation of Artificial Saliva

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The components of artificial saliva (400 mg/LNaCl, 400 mg/L KCl, 960 mg/L CaCl₂.2H₂O, 690 mg/L NaH₂PO₄.2H₂O, 5 mg/L Na₂S.9H₂O, 1000 mg/L Urea)⁽¹⁶⁻¹⁹⁾were measured via an electronic balance, and stirred with a glass rod until all the components dissolved in water (500 ml Deionized water and 500ml Distilled water). The pH of artificial saliva was adjusted to 6.75 ± 0.015 using apH meter (Jenway, model 3320, Cyprus)corresponding to the human salivary pH ^(20,21).

Preparation and Grouping of the Samples

The sampleswere consisted of three types of roundcross-section (0.018 inch) maxillary esthetic archwires: FRPC archwires (Translucent ideal arches pearl, Dentaurum, Germany), Teflon labially coated NiTi archwires (Rematitan® "LITE" White ideal arches, Dentaurum, Germany) and epoxy fully coated NiTi archwires (Tooth Tone® arches, Orthotechnology, Brazil). The straight portions were cut into pieces of 15 mm in length⁽²¹⁾. Total of 120 pieces, 40 pieces from each kind of archwires, weredivided in such away that 10 pieces from each type remained in a dry condition as a control group, while the other 30 pieces were immersed in artificial saliva for different immersion periods (1 day, 14 days and 28 days),ten pieces each.

Afterward, samples was placed in glass containers seperately and held from one of its ends using dental floss in such a way that avoid touching the wall. Artificial saliva was added so that the sample was immersed completely except for the epoxy ball. Thereafter, the glass container was capped perfectly by its lid and a piece of parafilm (**Figure1**).



Figure 1: Immersion of the sample in the artificial saliva.

After that, the samples were kept at 37^{0} C in an incubator (Fisher scientific, USA)for 1 day, 14 days and 28 days intervals.The artificial saliva was replaced regularly every 7 days with a fresh solution to avoid its saturation with the

degradation products^(20,21). After the intervals were elapsed the samples were washed with distilled water, left to dry on filter papers and then kept in petri dishes.

Preparation of Testing Specimens

Preparation of Slides and Fixing the Samples

In order to use AFM for analysis, it needs to use small slides instead of regular ones. The slides were cut into small sections (1x1 cm) using a diamond cutting pen. Each wire segment was then affixed on a new slide (**Figure 2**). For the labially coated samples, they were fixed with their labial surface facing upward⁽²²⁾.



Figure 2: Wire sample fixed on a small slide.

Cleaning the Samples

After each incubation the samples were immersed in distilled water with one drop of 2% sodium dodecyl sulfate solution, and ultrasonically cleaned at 20 watt for 3 minutes to remove the contaminated layer formed during handling. The samples were then rinsed with distilled water, allowed to dry in air and kept in closed petri dishes to be ready for the assessment.

Testing the Samples

The AFM was used to assess surface topography of the samples⁽²¹⁻²⁷⁾. For each specimen, three areas on the archwire have been scanned with a scanning area of 25 * 25 μ m: one in the center of the wire and the others on 2 mm away on both sides. Their mean value was used. Two numerical values in nm were determined in each scan (Ra and Ry) to elucidate its surface roughness^(3,4,25). Ra (Average Roughness) is the arithmetical mean of the absolute values of the scanned surface profile, while Ry (Maximum Peak-to-Valley Roughness Height) is the maximum height of a profile peak⁽²³⁾.

Tapping mode was used under ambient conditions^(8,24). The specimen was fixed to a piezo scanner with three translatory degrees of freedom. Subsequently, the three dimensional AFM view was shown on the monitor of the attached computer representing the surface of the specimen. Usingproprietary software supplied with the AFM, the images were processed.

Statistical analysis

Data were analyzed using a computer software

(SPSS -statistical package of social science-, version 19, Chicago, USA). The following statisticswere used:

<u>A. Descriptive Statistics:</u>including: the mean, median, standard deviation (S.D.),minimum (Min.) and maximum (Max.) values and statistical tables.

B.Inferential Statistics

Data were tested for its normality using the Shapiro-Wilks test.In addition,One-way Analysis of Variance (ANOVA), Kruskal-Wallis test, Least Significant Difference (LSD) and Mann-Whitney U test were carried out to see if there were any significant differences among the groups and to examine the source of these differences.

The probability (P) value of more than 0.05 was regarded as statistically non-significant and less than 0.05 was considered as significant.

Average Roughness (Ra)

At first, using Shapiro-Wilks test, it was found that Ra values were normally distributed.

Table (1) showed that at each immersion period, FRPC wires had the highest Ra among esthetic archwires, except that, at 28 days immersion period, the Teflon coated wires were the roughest. On the other hand, the lowest Ra found in epoxy coated wires except that at 1 day immersion period, the Teflon coated wires had the lowest. One-way (ANOVA) demonstrated a highly significant difference in Ra among the three types of wires at each immersion duration (P=0.000).

The data revealed that there were nonsignificant differences in Ra between Teflon coated and epoxy coated wires at the dry condition and the 1day immersion, whilst a highly significant difference was found between each pair of wire's types at other durations(**Table 2**).

RESULTS

Table 1: Mean and S.D. values of the average roughness (Ra) in nm of different esthetic
archwire types.

Condition	Wire types	Descriptive statistics				ANOVA test (d.f.= 29)	
		Mean	S.D.	Min.	Max.	F-test	p-value
Derry	FRPC	147.14	13.06	135.94	168.63		
condition	Teflon coated	111.45	15.82	92.38	129.45	16.947	**0.000
condition	Epoxy coated	109.18	19.54	90.53	143.12		
1 day	FRPC	182.991	9.89	172.10	199.43		
1 day	Teflon coated	104.79	9.91	90.55	115.23	264.187	**0.000
minersion	Epoxy coated	107.62	5.18	100.07	112.23		
14 dava	FRPC	157.91	7.40	150.70	168.61		
immorsion	Teflon coated	121.46	6.53	112.35	128.95	289.496	**0.000
minersion	Epoxy coated	89.22	5.00	83.01	96.43		
28 days immersion	FRPC	126.93	12.26	107.76	139.33		
	Teflon coated	172.24	27.68	140.81	219.13	47.086	**0.000
	Epoxy coated	95.41	5.83	87.99	104.21		

(**) means highly significant ($P \le 0.01$).

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Condition	Wire	types	Mean Difference	p-value
Dray	FRPC	Teflon coated	35.69	**0.000
condition	TRIC	Epoxy coated	37.95	**0.000
condition	Teflon coated	Epoxy coated	2.26	0.759
1 day immersion	FDDC	Teflon coated	78.13	**0.000
	FKFC	Epoxy coated	75.30	**0.000
	Teflon coated	Epoxy coated	-2.83	0.469
14 days immersion	EDDC	Teflon coated	36.45	**0.000
	ГКГС	Epoxy coated	68.69	**0.000
	Teflon coated	Epoxy coated	32.24	**0.000
28 days immersion	EDDC	Teflon coated	-45.31	**0.000
	FKPC	Epoxy coated	31.52	**0.000
	Teflon coated	Epoxy coated	76.84	**0.000

(**) means highly significant ($P \le 0.01$).

Maximum Peak-to-Valley Roughness Height (Ry)

Shapiro-Wilks test revealed that Ry values were not normally distributed.

Table (3)showed that at each immersion period, FRPC wires had the highest Ry among esthetic archwires, except that, at 28 days immersion periods, the Teflon coated wires had the highest value. On the other hand, the lowest Ry found in epoxy coated wires except that for the control group, the Teflon coated wires had the lowest value. Kruskal-Wallis test demonstrated a non-significant difference in Ry among the three types of wires at 14 days immersion period and a significant difference between them at the 28 days immersion, whereas highly significant differences were found at the other two periods.

Mann-Whitney U test revealed that there were non-significant differences in Ry between Tefloncoated and epoxy coated wires at the dry condition, FRPC and Teflon coated wires at 1 day immersion period and FRPC and epoxy coated wires at 28 days immersion period. In contrast, Ry differed significantly between FRPC and epoxy coated wires at the dry condition, FRPC and Teflon coated wires at 28 days immersion period and Teflon coated and epoxy coated wires at 28 days immersion period, while highly significant differences were found between the others (**Table 4**).

 Table 3: Medianvalues of the maximum roughness height (Ry)in nm of different esthetic archwire type.

Condition	Wire types	Desc	criptive stat	Kruskal-Wallis test (d.f.= 2)		
		Median	Min.	Max.	X^2	p-value
Deres	FRPC	731.080	635.790	819.780		**0.001
Dry condition	Teflon coated	329.420	202.110	525.090	13.154	
	Epoxy coated	414.350	214.710	910.950		
1 day	FRPC	260.350	255.700	269.620		**0.000
I day immersion	Teflon coated	256.850	192.270	368.200	19.079	
	Epoxy coated	170.725	143.470	193.520		
14 days immersion	FRPC	247.260	203.340	256.100		
	Teflon coated	245.920	204.840	260.100	5.040	0.080
	Epoxy coated	218.880	99.489	245.930		
28 days immersion	FRPC	215.955	153.260	241.470		
	Teflon coated	241.040	206.530	435.800	6.823	*0.033
	Epoxy coated	174.410	135.340	278.160		

(*) means significant $(0.05 \ge P > 0.01)$.

(**) means highly significant ($P \le 0.01$).

Table 4: Differences in Ry of different types of esthetic wires at eachimmersion period.

Condition	Wire	types	Mann-Whitney U test	p-value
Deu	FPDC	Teflon coated	0	**0.000
condition	TKIC	Epoxy coated	23	*0.041
	Teflon coated	Epoxy coated	36	0.290
1 day immersion	EDDC	Teflon coated	44	0.650
	ГКГС	Epoxy coated	0	**0.000
	Teflon coated	Epoxy coated	1	**0.000
28 days immersion	EDDC	Teflon coated	24	*0.049
	FKPC	Epoxy coated	42	0.545
	Teflon coated	Epoxy coated	18	*0.016

(*) means significant $(0.05 \ge P > 0.01)$.

(**) means highly significant ($P \le 0.01$).

DISCUSSION

At the dry condition, FRPC wires were the roughest (had the highest Ra) which was presumably due to the concurrent high (Ry) values in relation to other two types this might be due to the surface characteristics of the composite material and/or the manufacturing process. The pre-existed surface defects are believed to be the preferred degradation sites and accelerate it due to the higher residual stressesbesides harboring stagnant solution. Thesemight contribute todislodgement of fillers from composite materials in artificial saliva which is in agreement with *Ferracane and Condon*⁽²⁸⁾ and/or leaving partly exposed filler particleswhich is in agreement with Larsen and Munksgaard⁽²⁹⁾ that might explain the higher roughness of FRPC wires than the other wires in artificial saliva thereafter. This outcome is also congruent with the findings noted by Al-Najafy⁽³⁰⁾, Al-Jumailiand *al.*⁽⁸⁾. *Tawfek*⁽³¹⁾and*Chng* et On the contrary, Inami et al. (13) found that the surfaces of the as-received FRPC and metallic wires (except β -Ti) appeared almost smooth with slightly higher Ra in the FRPC ones. The cause of this conflicting finding might be due to the differentmanufacturers of FRPC archwires used in both studies such as Biomers.

Teflon coated NiTi archwires had the highest roughness at 28 days that were probably explained by changes in the elemental composition of their surfaces and the occurrence of additional elements due to interactions with saliva that altered the morphology, which is in agreement with Zegan et $al.^{(32)}$. This result is also in concordance with the one month clinically retrieval and AFM study done by Rongo et al.⁽²⁶⁾. However, in these two in vivo studies, two factors might contribute to this effect; intra-oral exposure of Teflon coated archwires and the archwire-bracket friction and thereby direct comparisons with the present results are difficult due to the differences in the study designs.Conversely, the current result contradicts *Mohsin*⁽²¹⁾ study which was a similar AFM study reported non-significant differences in roughness between Teflon and epoxy NiTi coated wires at 28 days immersion period in artificial saliva. Thecause of this disagreement might be the different manufacturers of the Teflon coated wires used and the slightly different protocol used as measuring roughness without ultrasonic cleaning and immersion of 10 samples in the same container that might alter the roughness values.

On the other hand, the least rough surfaces (Ra) were the epoxy coatings compared to the analogous archwires, except that at 1 day immersion periods where the Teflon coatings had the lowest Ra. However, statistically non-significant differences were found between Teflon and epoxy at the dry condition and the 1 day immersion. This might be attributed to the concurrent low (Ry) values of the two coatings at the dry condition in relation to the FRPC wires, being non-significantly different between Teflon and epoxy coatings which coincides with that

reported by **Rongo et al.**⁽²⁶⁾. This might be ascribed to the method of applying the coating, which could require some surface treatment and/or heat treatment, and/or due to the properties and composition of the coating material, and these specific information are not readily available and are companies secretes. Furthermore, these little pre-existed surface defects inflicted during manufacturing process are believed to prefer less and slow the degradation that might explain the lower roughness of epoxy coated wires than the other wires in artificial saliva thereafter. In addition, the epoxy resin was primarily recognized for its excellent adhesion and a broad range of physical properties, such as chemical resistance and dimensional stability. Meanwhile, due to the strength of the carbon-fluorine bonds, PTFE (Teflon) is nonreactive and hydrophobic. All that is in agreement with *Kravitz*⁽³³⁾. These data are also consistent with the results of other previous AFM study done by D'Antò et al. (23) and *Mohsin*⁽²¹⁾.In addition, the results from the current study agreed with that reported by Krishnan et al.⁽²⁵⁾who found that the as-received epoxy coated NiTi wires demonstrated the significantly lowest roughness values. However, they found higher roughness values of Teflon coated NiTi wires in relation to other study groups (surface modified and conventional NiTi wires). Similarly, Krishnan et al. (34) found that Teflon had more breakdown potential in Ringer's solution than the epoxy type that complies with the current result. However, the present result disagreed with their findings, which showed a significant higher roughness of epoxy than Teflon coatings in asreceived state. These inconsistencies might arise from the different manufacturing processes of the NiTi-based archwires and/or from the different protocols used.On the other hand, the present result disagreed with Rongo et al.⁽²⁶⁾ who found that the epoxy coated NiTi wires had a significantly lower roughness than Teflon in the as-received state.

Generally, the disparities in roughness among the three types of wires might be attributed to the type of surface material, manufacturer, and manufacturing technique. Moreover, probable factors influencing the surface integrity in artificial saliva might be associated with the original surface roughness, deposition method (synthesis and fabrication process) used, material stability, and surface material-substrate adhesion strength that is in agreement with **Bourauel et al.**⁽¹¹⁾, **Daemset al.**⁽¹²⁾,**Ryu et al.**⁽²⁷⁾and**Zegan et al.**⁽³²⁾.

It can be concluded from this *in vitro* study that the surface roughness of esthetic archwires

immersed in artificial saliva has a material specific pattern. Further refinement in the manufacture of FRPC archwires would be necessary to fully realize their potential as esthetic archwires. Care should be taken of their extremely high initial roughness. In addition, improvements to coating techniques of Teflon coatings or using alternative wires must be explored. Epoxy coated archwires are the best esthetic archwires in term of the least surface roughness initially and over the course of orthodontic treatment for patients seeking esthetics during fixed appliance therapy.

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الخلاصة

المقدمة: إن الطلب على أجهزة تقويم الأسنان التجميلية في تزايد مستمر ولذلك تم إستحداث أسلاك تقويم الأسنان التجميلية. صممت هذه الدراسة التجريبية لتقييم خشونة أسطح الأسلاك المركبة البوليمرية المقواة بالألياف(FRPC) مقارنةً بأسلاك النيكل تيتانيوم المغلفة (NiTi) المغموسة في اللعاب الإصناعي.

المواد وطريقة العمل: تم إستخدام ثلاثة أنواع من أسلاك تقويم الأسنان التجميلية: الأسلاك المركبة البوليمرية المقواة بالألياف (Dentaurum)، النيكل تيتانيوم المغلفة بالتفلون (Dentaurum) والنيكل تيتانيوم المغلفة بالإيبوكسي (Orthotechnology). كانت جميعها مستديرة المقطع (0.018 إنج) وقطعت إلى قطع بطول 15 ملم. أربعون قطعة من كل نوع قسمت إلى أربعة مجاميع. تركت المجموعة الأولى في البيئة الجافة وغمستالمجاميع الثلاثة الأخرى في اللعاب الإصطناعي (معامل الحموضة 30.0 ± 0.55) تحت درجة حرارة 37 درجة سليزية ولمدة 1, و28 يوم. أستخدم مجهر القوى الذرية لدراسة أسطح جميع العينات. أستخدمت إختبارات ال (ANOVA)، (Kruskal-Wallis)، (LSD) (Mann-Whitney U) لتحديد وتمييز مصدر الإختلافات بين المجاميع.

ا**لنت أنج**:عند كمل فترة إنغماس،أظهرت الأسلاك المركبة البوليمرية المقواة بالألياف أعلى قيم (R_a)بين المجاميع المدروسة، ما عدا عند فترة الإنغماس 28 يوم حيث كانت أغلفة التفلون هي الأكثر خشونة. من جهة أخرى، الأسطح الأقل خشونة كانت أغلفة الإيبوكسي عندما قورنت مع الأسلاك التجميلية المناظرة، ما عدا عند فترة الإنغماس ليوم واحد حيث متلكت أغلفة التفلوناقل قيم الخشونة.غير أنه وجدت فروقات غير معنوية إحصائيا بين التفلون والإيبوكسي عند الحالة الجافة ومدة الإنغماس ليوم واحد.

الإستنتاح:إن الأسـلاك المغلفة بالإيبوكسي هـي أسـلاك تقـويم الأسـنان التجميليـة الأفضـل والأكثـر ملاءمـة كونهـا الأقـل خشـونةمن البدايـة وتسـتمر خـلال فترة الدراسة.

الكُلمات الرئيسية: الأسلاك التجميلية، الأسلاك المركبة البوليمرية المقواة بالألياف، خشونة السطح، مجهر القوى الذرية.