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Evaluation of Nickel Ion Release and Corrosion Potential of Different Orthodontic Arch Wires Before and After Application of Some Environmental Natural Extracts (An In Vitro Study)

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ABSTRACT

Objective: *To evaluate the corrosion inhibitory effect of some natural environmental extracts on some orthodontic arch wires and assess their nickel ion-release and corrosion potential.*

Materials and Method: *245 wire specimens made of (St.St, NiTi, CuNiTi, Thermal NiTi and TMA as the control group) were used. The specimens were immersed in four different media (saliva, saliva with the extracts such as 'pomegranate, pro-polis and hibiscus'). The quantity of Ni ions was measured in three-time interval (7, 14, and 21) days using atomic absorption spectrophotometer. Wires were also tested for corrosion breakdown potential.*

Results: *The results showed significant variation in nickel ion release, breakdown potential between saliva and the corrosion inhibitors containing media. Ni ion release decreased to 0 by using the natural inhibitors. Hibiscus and pro-polis inhibited ion release with a very little concentration of 0.1%, while pomegranate needed more concentration to inhibit ion release (1%). The corrosion resistance of the examined wires was increased after using the inhibitors in comparison with the control specimens.*

Conclusion: *The inhibitors decreased the corrosion of orthodontic arch wires thus prevented their Ni ion release.*

Keywords: *Nickel, corrosion, pomegranate, pro polis, hibiscus, inhibitors.*

I. INTRODUCTION

Tooth movement and the changes associated to it are the result of an applied force system and its tissue response. Orthodontic wires which communicate through bracket, generate biomechanical forces to move teeth and are considered the master of orthodontic practice.(1) Among alloys used for orthodontic therapy especially orthodontic wires, four alloys are common: stainless steel (St.St.), nickel-titanium (NiTi, CuNiTi), cobalt-chromium and β titanium alloys (TMA). (2) Stainless steel (St.St.) alloys contain approximately 18% chromium and 8% nickel while nickel-titanium alloys have 50% nickel of its content.

Orthodontic appliances are subjected to physical and corrosive damage in the mouth, which act in combination to degrade physical properties and increase the failure potential.(3)The retro-gradation of the orthodontic arch wires corrosion has two sequels, the first is the loss of physical properties and the second is the nickel ions release. Unfortunately, nickel ions release from orthodontic alloys has been proved to be cytotoxic and cause allergic reactions.(2)Nickel metal, nickel carbonate, nickel oxide and nickel sulfide are definitely human carcinogens.(4)

For corrosion prevention, the primary strategy is to separate the metal surface from the corrosive agents. This can be achieved by adding 'corrosion inhibitors'. Corrosion inhibitor can be defined as a substance that can effectively reduce corrosion rate of a metal exposed to the environment in which it is added in a small concentration.

Large numbers of organic compounds were studied and are being studied to investigate their corrosion inhibition potential. All these studies, (5, 6) stated that organic compounds especially those with N, O and S showed significant inhibition efficiency, but, unfortunately most of these compounds are expensive and toxic to human beings.

It is needless to quantity the importance of cheap, safe inhibitors of corrosion. Green extracts have become important as an environmentally friendly acceptable, readily available and renewable source for wide range of inhibitors. They are the rich sources of ingredients, which have very high inhibition efficiency.[7]

Therefore, the aim of this study was to evaluate the corrosion inhibitory effect of some natural extracts on the orthodontic arch wires and to investigate the nickel ion release and corrosion potential.

II. MATERIALS AND METHODS

Materials

A total number of 245 metallic rectangular orthodontic arch wire specimens were divided into five groups and used in this study. (Table 1).

The wires were produced by different manufactures (Ormco Corp., Glendora, Calif, USA-Ortho organizers-American orthodontics) respectively**.**

Table 1: Materials used in the study**.**

Groups (arch wires)	*Number of specimens*	*Alloy*	*Manufacturer*	*Size (in inches)*
Group I	25	β-titanium (TMA)	Ormco corp.	**0.016x 0.022**
Group II	25	Stainless steel	Ortho-organizers	**0.016x 0.022**
Group III	25	Ni-Ti	Ortho-organizers	**0.016x 0.022**
Group IV	25	copper Ni –Ti	American orthodontics	**0.016x 0.022**
Group V	25	Thermal Ni-Ti	Ortho-organizers	**0.016x 0.022**

Samples were tested in the following media

Simulating artificial saliva, which consisted of (Sodium chloride 0.4 g, potassium chloride 1.21 g, sodium hypophosphate 0.78 g, sodium sulfide 0.005 g, urea 1g, distilled-deionized water 1000 ml at 36.5^{o} C and a PH of 5.6-7.

Natural extracts

- **Pro-polis extract**

Collected from bee hive and placed with the proper amount of alcohol in a sealed container and stored in warm dark place. After dissolvation, it was filtered using a coffee filter.

- **Punica-granatum extract**

The pomegranate extract powder was obtained by heating the pomegranate peel at 60˚C with methanol, after being concentrated, it had been spray dried.

- **Hibiscus extract**

A manual grinding of hibiscus calyces in a thermostatic water bath with slight orbital stirring was done, then the mixture was cooled down and filtered with a 0.45 ml filter, to obtain hibiscus extract.

All media were prepared in faculty of pharmacy, Mansoura University

Methods:

The specimens were divided into subgroups of five samples for each media in the investigation. All specimens were used in the “as received” condition from the manufacturer.

A 6 Cm of distal end of every arch wire were immersed in Polypropylene screw top bottle containing 100 ml saliva. The inhibitors with different concentrations of 1ml, 0.5ml, 0.1ml were prepared in saliva media and the wires were immersed in the bottles and saved in incubator at 37^{o}C . The collected media were tested at different intervals; 7 days, 14 days, and 21 days using atomic absorption spectrophotometer (Sens AA"GBC Scientific equipment") model (Sens AA Dual, Dandenong, Victoria, Australia, 2009), for Nickel ion release.

Atomic absorption spectrophotometer measures the quantities of chemical elements present in environmental samples by measuring the absorbed radiation. This is done by reading the spectra produced when the sample got excited by radiation.[8] Since the sensitiveness of the equipment was restricted up to 1 ppm, a **“standard addition method”** was used.

The collected wires from the first week were tested using Gamry Instrument PCI300/4 Potentiostat/ Galvanostat/ZRA for

a. Potentio-dynamic polarization method, which gives information about the corrosion rate, pitting susceptibility, passivity, as well as the cathodic behavior of an electrochemical system. The potentiodynamic current-potential curves were recorded by changing the electrode potential automatically from -1 to +1 V with scan rate of 1mVs-1 using an electrochemical measurement system. Saturated calomel electrode (SCE) and platinum foil were used as reference and counter electrodes, respectively. The working electrode was in the form of -6 mm- sample of the arch wire under investigation.

b. AC electrochemical impedance spectroscopy (EIS) method, which measures the response of an electrochemical system (cell) to an applied potential. The cell and the apparatus used in EIS technique is the same as used in -potentiodynamic polarization- method with the same reference, auxiliary and sample electrodes. After the determination of steady-state current at a given potential, since wave voltage AC 10 mV peak to peak, at frequency ranges between '100 KHz and 0.2 Hz 'were superimposed on the rest potential.

c. AC electrochemical frequency modulation (EFM) method which gives a good tool for measuring electrochemical corrosion. With the electrochemical modulation technique (EFM), a potential perturbation by two sine waves of different frequencies is applied to the system. Identical cell assembly as used in both polarization and impedance studies was used for EFM measurements.

Electrochemical experiments were performed in conventional three-electrode electrochemical cell with a capacity of 100 ml(9) as shown in Fig. 1.

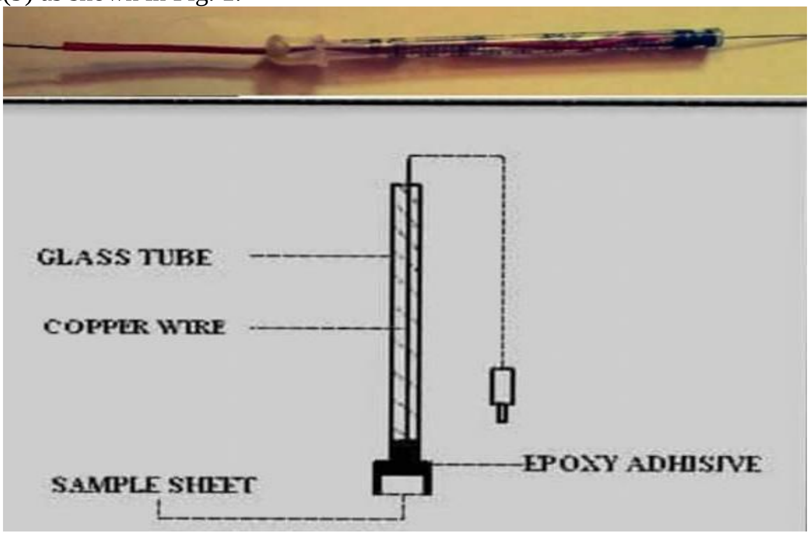

Fig. 1: Shows the electrode used for electrochemical measurements.

III. RESULTS

Nickel ion release results:

In all specimens, the amount of released Ni reached the maximum on 7th day, thereafter, it diminished with time. The result showed that the maximum amount of Ni ion was released from St.St followed by NiTi alloys. Table 2

After application of the three inhibitors (punica granatum, pro polis, and hibiscus) for the same time interval (7, 14, and 21) days with 1% concentration, the results were:

For the first week, all the results were diminished to Zero.

For the second week, only hibiscus lose its effect.

For the third week, pomegranate gives positive results of Ni release. Table 3

At 0.5% and 0.1% inhibitor concentration, hibiscus and pro polis gave a zero results with all groups. In comparison, pomegranate gave positive results .Table 4

The results of one way ANOVA 'with respect to change in media only' showed significant difference among the tested groups with conc. (1%) for all media. A significant difference of ($p<0.001$) with (0.5%, 0.1%) pro-polis and hibiscus conc.

For pomegranate conc. (0.5%, 0.1%), one way ANOVA showed a non-significant difference ($p>0.0001$).

Two way ANOVA was used to determine the change in Ni release with respecting to wire and media changes, there was a significant difference among the studied groups ($p<0.036$, $p<0.032$, $p<0.011$) for conc. 1%, 0.5%, 0.1% respectively.

Three-way ANOVA was used to determine the interaction between the type of wire, media and time sequence for Ni release. There was a significant difference among the studied groups ($p<0.066$).

Table 2: Mean and standard deviation of nickel release in ***(µg)*** for wires in saliva at time zones of 'Day 7 vs. day 14, Day 7 vs. day 21, Day 14 vs. day 21. (Student`s pair *t* test).

Groups	*Time point*	*Mean ±SD (µg)*	*Time point compared Change*	*P value (significant)*
Group I TMA	Day 7	0	Day 7 versus day 14	-
	Day14	0	Day 7 versus day 21	-
	Day 21	0	Day 14 versus day 21	-
Group II St-St	Day 7	1.43 ± 0.70	Day 7 versus day 14	**<.0001**
	Day 14	4.61 ± 0.50	Day 7 versus day 21	**<.0001**
	Day 21	3.52 ± 0.52	Day 14 versus day 21	**<.0001**
Group III NiTi	Day 7	0 .65 ± 0.18	Day 7 versus day 14	**<.0001**
	Day 14	1.99 ± 0.50	Day 7 versus day 21	**<.0001**
	Day 21	1.55 ± 0.54	Day 14 versus day 21	**<.0001**
Group IV Copper NiTi	Day 7	1.30 ± 0.51	Day 7 versus day 14	**<.0001**
	Day 14	2.99 ± 0.54	Day 7 versus day 21	**<.0001**
	Day 21	1.66 ± 0.25	Day 14 versus day 21	**<.0001**
Group V Thermal NiTi	Day 7	1.42 ± 0 .56	Day 7 versus day 14	**<.0001**
	Day 14	3.11 ± 0.52	Day 7 versus day 21	**<.0001**
	Day 21	1.25± 0.30	Day 14 versus day 21	**<.0001**

Table 3: Mean and standard deviation of nickel release (µg) for wires in hibiscus and pomegranate.

variable	***Hibiscus*** *2nd week*		***Pomegranate*** *3rd week*	
	Mean	***SD***	***Mean***	***SD***
St.St	0.154	0.005	0.142	0.007
NiTi	0.143	0.095	0.112	0.013
CuNiTi	0.144	0.005	0.063	0.017
Thermal NiTi	0.132	0.010	0.102	0.009

Table 4: Mean and standard deviation of nickel release (µg) of wires in pomegranate extract with different concentrations.

variable	***pomegranate*** *0.5% conc.*		***Pomegranate*** *0.1%conc.*	
	Mean	***SD***	***Mean***	***SD***
St.St	0.148	0.019	0.142	0.011
NiTi	0.102	0.004	0.112	0.013
CuNiTi	0.144	0.006	0.063	0.017
Thermal NiTi	0.132	0.003	0.102	0.009

Corrosion results
(Electrochemical measurements)
AC electrochemical impedance spectroscopy (EIS) method

Mean values of Charge transfer resistance (R_{ct}), which is inversely proportional to the corrosion rate , showed that the highest mean value for R_{ct} in saliva was found for TMA in saliva (1.432 e^3 Ohm cm^{-2}). The lowest mean

of R_{ct} was found for SS wires (0.954 e^3 Ohm cm^{-2}).After inhibitors application there were a remarkable changes in the R_{ct}. Table 5
There was a significant increase in the corrosion resistance (R_{ct}) for all the tested wires.

Table 5: Mean results of R_{ct} (Ohm cm^{-2}) of the studied wires in different media.

Eispot(R_p)	***TMA***	***St.St.***	***NiTi***	***CuNiTi***	***Thermal NiTi***
Saliva	$1.432e^3$	$0.954e^3$	$1.100e^3$	$1.122e^3$	$1.307e^3$
Pome.	$2.980e^3$	$3.531e^3$	$2.709e^3$	$3.402e^3$	$2.707e^3$
Propolis	$3.507e^3$	$4.895e^3$	$3.210e^3$	$3.516e^3$	$3.813e^3$
hibiscus	$1.622e^3$	$1.202e^3$	$1.451e^3$	$1.555e^3$	$1.468e^3$

AC electrochemical frequency modulation (EFM) method (I corrosion)
Mean values for Corrosion current density (I_{corr}), which is inversely proportional with corrosion resistance (R_{ct}) and directly proportional with potentiodynamic corrosion current density showed that the highest mean value for $i_{corr.}$ In saliva was found for TMA wires (44.330 µA cm^{-2}), while the lowest mean was for CuNiTi wires (7.367 µA cm^{-2}). After application of inhibitors, there is a remarkable change in the corrosion values. The results showed corrosion rates diminishing with the inhibitors. Table 6

Table 6: Mean results of I_{corr} (µA cm^{-2}) of the studied wires in different media.

EFM (i_{corr}.)	***TMA***	***St.St***	***NiTi***	***CuNiTi***	***Thermal NiTi***
Saliva	44.330	36.647	22.797	7.367	12.650
Pomegranate	39.790	35.690	20.410	3.896	5.839
Pro polis	19.500	32.620	9.170	2.300	11.420
Hibiscus	7.923	26.990	7.619	3.694	1.418

Potentio-dynamic polarization curves (PDP Measurements):

Fig.2, 3, 4 show the cathodic and anodic polarization curves of the different wires in the tested media (with and without inhibitor) the figures shows anodic shift of all results.

Potentio-dynamic curves showed obvious decrease in' $I_{corr.}$' from saliva to the inhibitor thus proving its efficacy. Table 7, 8, 9.

Three way ANOVA was used to determine the interaction between the type of wires, media and method used for corrosion testing, there was asignificant difference among the studied grous ($p<0.074$).

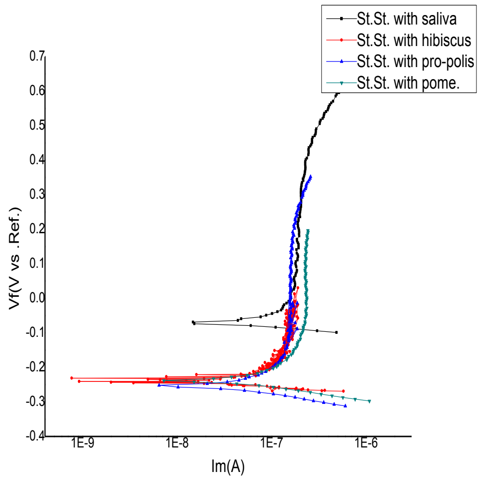

Fig. 2. Shows potentio-dynamic curves of St.St

Table 7.potentio-dynamic of St.St. in µC.

INTERGRATE-Section (100.0 p to 10.00 u)	*Results(c)*
Curve (St.St. +saliva POTDYN.DTA):	32.87 µC
Curve (St.St. +pome. POTDYN.DTA):	17.28 µC
Curve (St.St. +pro-polis POTDYN.DTA):	19.22 µC
Curve (St.St. +hibiscus POTDYN.DTA):	18.28 µC

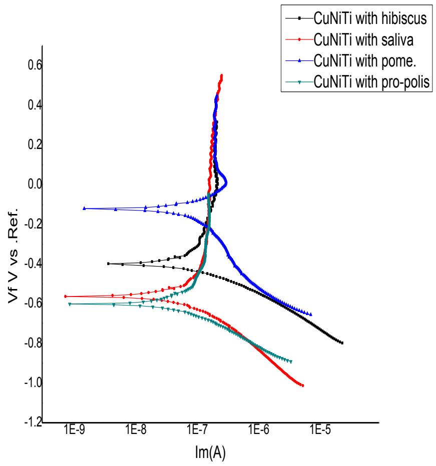

Fig 3. Shows potentio-dynamic curves of CuNiTi wire.

Table 8.potentio-dynamic of CuNiTi in µC.

INTERGRATE-Section (100.0 p to 10.00 u)	*Results(c)*
Curve (CuNiTi +saliva POTDYN.DTA):	34.99 µC
Curve (CuNiTi +pome. POTDYN.DTA):	4.390 µC
Curve (CuNiTi +pro-polis POTDYN.DTA):	10.11 µC
Curve (CuNiTi +hibiscus POTDYN.DTA):	11.07 µC

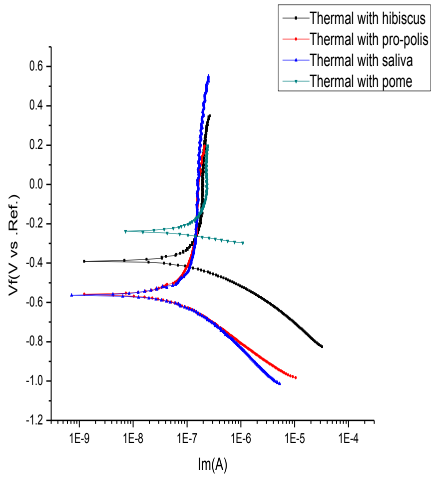

Fig 4. Shows potentio-dynamic curves of Thermal NiTi wire.

INTERGRATE-Section (100.0 p to 10.00 u)	*Results(c)*
Curve (Thermal NiTi +saliva POTDYN.DTA):	34.90µC
Curve (Thermal NiTi +pome. POTDYN.DTA):	17.28µC
Curve (Thermal NiTi +pro-polis POTDYN.DTA):	21.00µC
Curve (Thermal NiTi +hibiscus POTDYN.DTA):	26.93µC

Table 9.potentio-dynamic of Thermal NiTi in µC.

IV. DISCUSSION

The present study considered the effect of some natural inhibitory extracts on the corrosion of orthodontic arch wires and evaluated the nickel ion release. All the orthodontic arch wires used in this study contained nickel except the β-titanium wires that was used as the control group. The nickel ion measurement was done at three different time intervals (7, 14, and 21) days to study the Ni ion release and detect the effect of the inhibitors over prolonged period. Various corrosion tests were done to detect corrosion rate, pitting susceptibility and passivity after the addition of inhibitors.

The oral cavity is a complete corrosion cell, with many factors that enhance the biodegradation of orthodontic appliances. Saliva acts as an electrolyte solution for electrons and ions conduction, and the various chemicals introduced to the oral cavity through diet are all capable of inducing corrosion.[10] The corrosion of an alloy is of fundamental importance to its biocompatibility, because the release of metallic elements is a primary reason for adverse biologic effects. Thence, corrosion and ion release are the two side of the same coin.

Several authors[11, 12] have reported corrosion of orthodontic appliances in vitro. Nickel ions release from the orthodontic appliance has gained more interest lately because of the critical situations of cytotoxic responses. Dunlap *et al*.[13] in their study have reported a case of severe allergic reactions after insertion of NiTi arch wire in a Ni sensitive patient. Schriver et al. [14] reported a case of allergic reaction to an intermaxillary stainless steel wire in a nickel-sensitive patient. This hazardous nickel effects encouraged the "European Union legislation "to compel a statute limiting of nickel release from manufactured materials.[15]

In the last years, Umoren et al., [16] evaluated plant extracts as 'corrosion inhibitors', e.g. Phyllanthus amarus, Pachylobus edulis Raphia hookeri and Ipomoea involcrata. Up till now, little information is available for natural, safe and cheap inhibitors for orthodontic patients.

Pomegranate, pro polis, and hibiscus are natural materials with organic constituents that can be used safely in our daily life. The inhibition efficiency of plant origin inhibitors has been reported by Oguzie, et al,[17] to be dependent on the degree of the organic constituents' coverage on the metal surface.

Pomegranate contains organic acids with (phenolic, hydroxyl, and carbonyl groups and antioxidant materials) [18]; some of these organic compounds have been used as metal corrosion inhibitor. On the other hand, organic molecules adsorption as 'phenolic compounds'(gallic acid and granatin B, may be due to the presence of an oxygen atom (a heteroatom), π electron of aromatic rings, and electron donating groups. Furthermore, according to literature organic acid component itself can form passive oxide film on metal surface. Corrosion inhibition action of pomegranate juice increases as its concentration increases in the corrosive solution [19], this corresponds with the findings of our results and explains the non-significant results with its lower concentration.

Mechanism of Hibiscus extract inhibition on metal surface was described by Oguzie, et al,[20] as 'a mixed type', where the molecular and the protonated organic species in the extract contribute to the inhibition.

The probable mechanism of pro polis inhibition can be explained by the structure of the constituents present in the pro polis extract and on the process of adsorption. The anodic inhibition may be due to the adsorption of principle phytochemical constituents present in the extract through oxygen atoms/or ring oxygen atom in phenolic acid, flavonoid' and /or oxygen atoms of –OH group in (Kaempferol), and tectochrysin, these constituents form a protective passive coating on the metal surface (passivation). [21]

Data analysis of the nickel ions release test showed that the great concentration started from the day 7 of the study then the amount began to decrease through the third week.The findings of the current study are in accordance with the study by **Kerosuo *et al*.** [22] and **Gjerdet *et al***.[23]. The Ni can be both a solubilized solution and insoluble precipitate. The results of the present study were also in relevance with the study done by **Park and Shearer**. [24]

Of the orthodontic wires tested in this study, the largest amount of Ni release per day was from St.St wires (4.61 ± 0.50 µg)that contain ferric in its composition(71% wt.) thus undergo more corrosion and less corrosion resistance[25] followed by thermal NiTi, CuNiTi and NiTi wires (3.11 ± 0.52 µg, 2.99 ± 0.54 µg, 1.99 ± 0.50 µg)respectively. An interesting finding is the fact that St.St. wires (typically containing 8% Ni) release more Ni than NiTi and CuNiTi arch wires (which have about 50% Ni),this may be due to the high concentration of Fe (71%) in St.St. alloys which have a great corrosion susceptibility. [25]

For the three inhibitors at 1% conc, the Ni ion release diminished to Zero in the first week and continued to the third week for pro polis. This was in agreement with **Fouda** and **Badr** who proved that pro polis is a proposed corrosion inhibitor for carbon steel in aqueous solutions.[21]

..There were a variation in hibiscus inhibitory effect from the first week in which it showed zero results to positive results in the second week. This variation in the inhibition efficiency of it with time of exposure may be due to oxidation and reduction rate variation with time, thereby impacting residual stresses on the absorbed molecules, on the metal surfaces, thus affecting their stability. The same for pomegranate in the third week, this was in agreement with **Chidiebere et al.** and **Ameer and Fekry,** who evaluated corrosion inhibition of Punica granatum and hibiscus on mild steel alloy in an acidic environment [26, 27]

For corrosion, the results are in accordance to those found by **Speck et al and Ryan et al** [28, 29] . For saliva, the results of EFM (i_{corr}) showed that there was significant difference between all the wires in their corrosion potential. The St.St and TMA wires had the highest tendency for corrosion followed by NiTi wires.

For inhibitors, the results showed a significant difference of the tested wires compared with saliva group, indicating a good corrosion inhibitory effect of these extracts. This was in agreement with **Ashassi-Sorkhabi et.al,** who concluded that the pomegranate peel extract acts as a nontoxic, cheap, and easily prepared inhibitor for corrosion of mild steel in hydrochloric acid media.[19] Also with many studies, [20, 30] that showed the inhibition ability of hibiscus extract on carbon steel. **Fouda et al, also** proved [21] the inhibitory effect of pro polis extract on carbon steel in aqueous solution.

V. CONCLUSION

This study was conducted to evaluate the effect of a cheap compatible, and available corrosion inhibitor materials on the Ni ion released from orthodontic arch wires.

- The used inhibitors prevent Ni ion release even with very little concentration; thereby they can be considered efficient against corrosion of orthodontic arch wires.
- A pomegranate and pro polis mouthwashes were introduced for anti-inflammatory and anti-plaque properties but not for anti-corrosive property which was proved in this research, so a recommendation might be done to introduce mouthwashes, tooth pastes or even gums containing pomegranate, pro-polis and hibiscus extract for orthodontic patients.
- Further studies should be done to investigate the inhibitory effect of the three media under more complicated situations of the oral cavity (highly acidic, different temperatures, bacteria, fluoride, etc.)

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