

Corrosion behavior of SS 18/8 orthodontic wire in artificial saliva in the presence of an aqueous extract of *Spillanthes acmella* leaves

A.S. Begum,¹  S.S. Syed Abuthahir,¹  M.V. Mohamed,¹ 
S. Rajendran^{2,3*}  and A. Al-Hashem⁴

¹*P.G & Research Department of Chemistry, Jamal Mohamed College, Affiliated to Bharathidasan University, Tiruchirappalli-620013, India*

²*St. Antony's College of Arts and Sciences for Women (Affiliated to Mother Teresa Women's University, Kadaikanal), Dindigul-624 005, Tamil Nadu, India*

³*Adjunct Professor, Centre for Nanoscience and Technology, Pondicherry University, Chinna Kalapet, Kalapet, Puducherry 605014, India*

⁴*Petroleum Research Centre, Kuwait Institute for Scientific Research, 8WP4+7J9, Kuwait City, Kuwait*

*E-mail: susairajendran@gmail.com

Abstract

The corrosion resistance of SS 18/8 orthodontic wire in artificial saliva has been evaluated in the absence and presence of an aqueous extract of *Spillanthus acemella* plant leaves. Electrochemical studies such as polarization technique and AC impedance spectra have been employed. The protective film has been analyzed by FTIR and AFM. Polarization technique reveals that in the presence of extract, the corrosion potential of SS 18/8 orthodontic wire is shifted to cathodic side. The *LPR* value increases and corrosion current decreases. This implies that in the presence of extract the corrosion resistance of SS 18/8 orthodontic wire increases. Similar observation is made in the AC impedance spectra results also. There is increase in charge transfer resistance value, increase in impedance value and decrease in double layer capacitance value. FTIR spectral study reveals that the increase in corrosion resistance is due to adsorption of the active principles present in the extract of the leaves onto the metal surface. The AFM parameters of the AFM images [the average roughness (R_a) value, root mean square (RMS) roughness (R_q) and maximum peak to valley ($P-V$) height] were derived. These values increased when SS 18/8 orthodontic wire was immersed in artificial saliva whereas these values decreased when SS 18/8 orthodontic wire was immersed in artificial saliva+extract system. The outcome of the study is that the patients those who use these metal alloys for orthodontic purpose can chew and place the leaves of *Spillanthes acmella* in the teeth cavities to get relive from pain of toothache without any hesitation and fear.

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Keywords: *Spillanthes acmella*, artificial saliva, SS 18/8 orthodontic wire, corrosion resistance, electrochemical studies, natural products.

Introduction

In the modern world the materials used in the biomedical field plays a major role in the production of different artificial body parts [1]. The prosthetic devices are usually made by metallic, polymeric and ceramic products. The main requirement for any external substances to fix in the human body is hundred percentage biocompatible and do not create any side reaction in the body. Corrosion inhibition is one of an important required properties for dental materials. Oral intake is one of the major reasons for the process of corrosion in orthodontic wire. Saliva is produced by the salivary glands. It is very important for a healthy mouth. It moistens and breaks down food, washes away food particles from the teeth and gums, and helps people with swallowing. It is very obvious that different tests involving dental materials should be done in saliva [2, 3]. The artificial saliva is mostly used in corrosion inhibition studies of dental metal and alloys.

In dentistry, the replacement of single or a row of teeth or in the production of dental prostheses such as metal plates for dentures, crowns and bridges, essentially in patients requiring hypoallergenic materials [4] is essential.

Corrosion, the graded degradation of materials by electrochemical attack, is of concern particularly when orthodontic appliances are placed in the hostile electrolytic environment provided by the human mouth. The main factors such as temperature, saliva, plaque, pH and proteins, physical and chemical properties of solid and liquid type food, tablets and oral conditions may influence the process of corrosion [5–7]. *Spillanthes acmella* is a well-known anti-toothache plant belonging to family Asteraceae and is used in traditional medicine for many purposes. The different plant parts of *S. acmella* like flowers heads, leaves, roots, stem and other aerial parts have been used in various health care systems [8]. In India, juice of inflorescence of *S. acmella* is used to treat mouth ulcers [9] due its active principle compound spilanthol. It has been described to possess number of biological effects such as anti-pyretic, anti-diuretic, anti-pyretic, anti-oxidant, anti-inflammatory, anti-cancer and anti-toothache [10].

Hence, there is need to investigate the corrosion aspects orthodontic wire made up SS 18/8 used in dentistry in the presence of natural medicine (used for chewing or placing in tooth cavities to relieve pain) for toothache in oral environments by surface analytical studies such as FTIR, AFM and also by electro chemical studies such as AC impedance spectra and polarization studies [11].

Experimental

Specimens and solutions

The corrosion resistance of SS 18/8 orthodontic wire in artificial saliva (AS) in the presence of an aqueous extract of leaves of *Spillanthes acmella* was evaluated by electrochemical studies. The major chemical composition [12] of the electrode used in present study are iron (73.75%), chromium (18%), nickel (8%) and carbon (0.25%). The orthodontic wire was

encapsulated in Teflon. The orthodontic wire specimens were used as the working electrode in potentiodynamic polarization and AC impedance spectral studies.

The surface of the electrode was polished to a mirror finish and degreased with trichloroethylene. Fusayama artificial saliva solution (KCl – 0.4; NaCl – 0.4; CaCl₂·2H₂O – 0.906; NaH₂PO₄·2H₂O – 0.690; Na₂S·9H₂O – 0.005; urea – 1.0 gram/liter) constituents closely resemble those of natural saliva [13]. The orthodontic wire specimens were immersed in Fusayama artificial saliva solution. During the study, the temperature of artificial saliva solution was maintained at 36±0.1°C.

The leaves of *Spilanthes acmella* are shown in Figure 1. Spilanthalol is a major active principle compound [14].



Figure 1. *Spilanthes acmella* (Tamil Name: Palvali Poondu).

Preparation of leaves extract

Fresh leaves were collected from near Kolli Hills (Namakkal district, Tamil Nadu State India). The collected leaves were dried in shadow.

The aqueous extract of *Spilanthes acmella* was prepared by Soxhlet extraction (Soxhlet Medium Extractor with 250 mL flask equipped with a heating mantle and maintained at a temperature of 60°C. 100 g of powdered leaves of *Spilanthes acmella* was uniformly packed into thimble and extracted with double distilled water. The process of extraction continued until the solvent in siphon tube of the extractor becomes colorless. After the process of extraction, the extract was kept overnight for cooling and made up to 1000 mL with the same double distilled water to get 10% (w/v) leaves extract.

Qualitative phytochemical test

The aqueous extract of leaves of *Spilanthes acmella* was tested for various components by their specific tests *viz.* Mayer's test, Dragendroff's test, Wagner's test for alkaloids; Raymond's test, Legal's test, Bromine water test for glycosides; Gelatin test, Ferric chloride test, Vanillin hydrochloride test for tannins and phenolic compounds test, Saponification test for fats and fixed oils and Foam test for saponins.

Preparation of the inhibitor solution

Preparation of the artificial saliva containing 10% of inhibitor solution

10 mL of aqueous extract of *Spillanthes acmella* was added in 90 mL of made up artificial saliva solution which yields artificial saliva containing 10% of inhibitor solution.

Electrochemical studies

Potentiodynamic polarization study

The polarization studies were carried out in a CHI Electrochemical analyzer (model 660A). A three electrode cell assembly was used. SS 18/8 alloy specimens used as a working electrode. A saturated calomel electrode (SCE) was the reference electrode and platinum was the counter electrode. Three electrode cell assembly is shown in Figure 2. A time interval of 5 to 10 minutes was given for the system to attain a steady state open circuit potential. The working electrode and platinum electrode were immersed in artificial saliva in the absence and presence of aqueous extract of *Spillanthes acmella*. Saturated calomel electrode was connected with the test solution through a salt bridge. From the polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes (b_a and b_c) and linear polarization resistance (LPR), were calculated. During the polarization study, the scan rate (V/s) was 0.01. Hold time at E_f (s) was zero and quiet time (s) was two. (In E_f (s), E_f means final potential and “s” refers to seconds. Quiet time (s) refers to quiescent time before potential scan) [15–19].

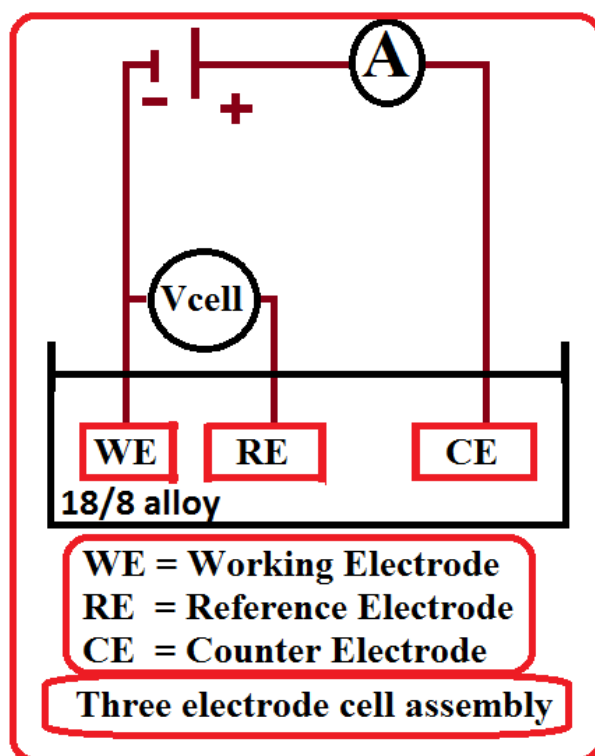


Figure 2. Three electrode cell assembly.

AC impedance spectra

AC impedance spectra have been used to investigate the formation of a protective film during corrosion protection process [20–32].

In the present study, the same instrument and cell set-up used for polarization study was used to record AC impedance spectra also. A time interval of 5 to 10 minutes was given for the system to attain a steady state open circuit potential. The real part (Z') and imaginary part ($-Z''$) of the cell impedance were measured in ohms at various frequencies.

Charge transfer resistance (R_t) and double layer capacitance (C_{dl}) were calculated.

$$R_t = (R_s + R_t) - R_s$$

where R_s = solution resistance. C_{dl} values were calculated using the relationship:

$$C_{dl} = \frac{1}{2} \pi f_{\max} R_t$$

Surface characterization studies

The SS 18/8 orthodontic wire specimens were immersed in blank, as well as inhibitor solution, for a period of 1 day. After 1 day, the specimens were taken out and dried. The nature of the film formed on the surface of the SS 18/8 metal specimens was analyzed by various surface analysis techniques such as FTIR and AFM.

Surface analysis by FTIR spectra

The film formed on the surface was scratched carefully and it was thoroughly mixed with KBr, made into pellets and the FTIR spectrum was recorded using Perkin–Elmer 1600 FTIR spectrophotometer with a resolving power of 4 cm^{-1} [33].

Surface morphology by AFM

The AFM images were recorded in an Agilent technologies 5500 series mode to examine the surface morphology by atomic force microscopy (AFM) [34].

Results and Discussion

FTIR spectroscopic analysis

The FTIR spectral patterns of the solid mass obtained after a few drops of Aqueous Extract of the *Spilanthes acmella L* dried on a glass plate is shown in Figure 3a. The FTIR spectrum of the protective film formed on the wire surface after immersion in the solution containing extract (10%)+artificial saliva system is shown in Figure 3b. The respective absorption bands are given in Table 1.

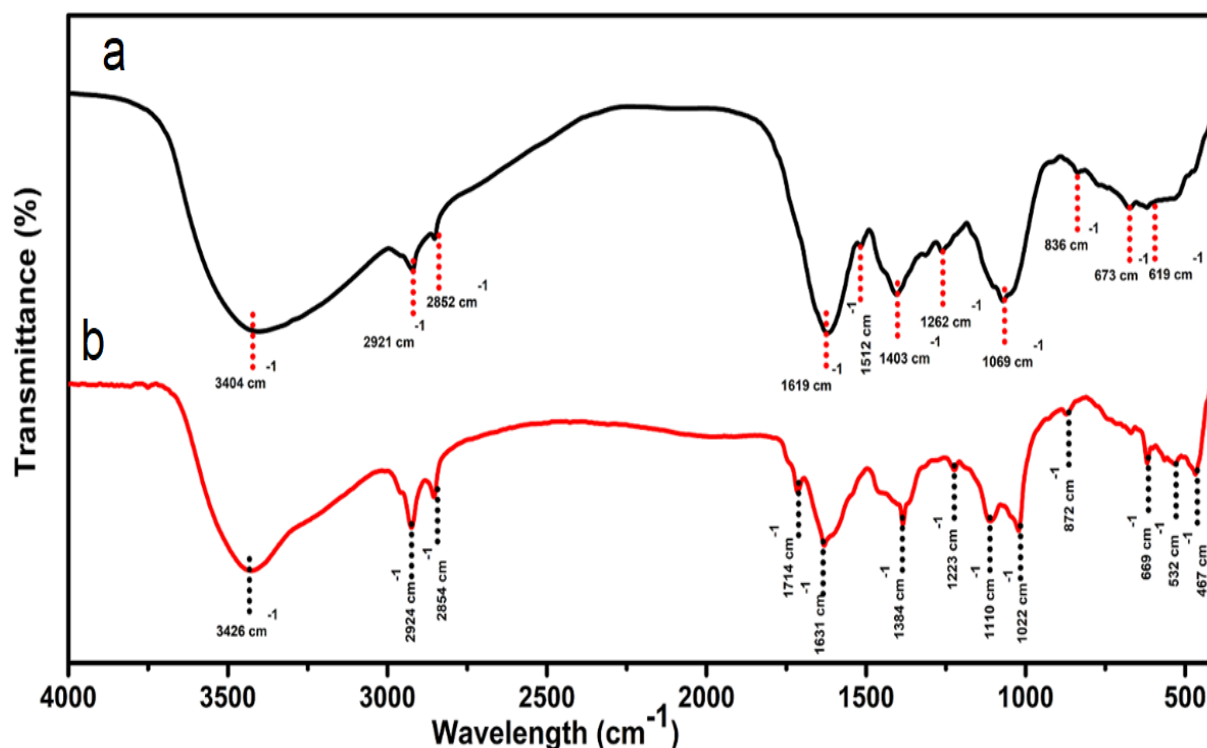


Figure 3. FTIR spectra (KBr) (a) solid mass obtained after a few drops of aqueous extract of the *Spillanthes acmella L* dried on a glass plate, (b) Scratched film after immersion in aqueous extract of the *Spillanthes acmella L* + AS medium.

Table 1. FTIR Spectral data for the aqueous leaf extract of *Spillanthes acmella L* and the scratched film from SS 18/8 orthodontic wire after immersion in AS with the 10 mL aqueous extract of *Spillanthes acmella L*.

IR bands of aqueous extract of <i>Spillanthes acmella L</i> .	IR Bands of film from the surface of the SS 18/8 orthodontic wire	Frequency assignment
3404	3426	–OH stretch (phenolic)
2921, 2852	2924, 2854	C–H– stretch(aliphatic)
–	1714	–C=O stretch (carbonyl)
1619	1631	–C=O stretch (amide group)
1262	1223	–C–O– stretch (aromatic)
1512	–	–C–C stretch aromatic ring
1069	1022	C–N stretching (amine group)
1262	1223	–C–O–stretch (sec. alcohol)

The FTIR spectral data of the aqueous extract of *Spillanthes acmella* leaves shows the broad band at 3404 cm^{-1} is mainly due to the presence of stretching vibration of O–H. The bands at 2921 and 2852 cm^{-1} represent the stretching vibration of aliphatic C–H. The absorption peaks at 1619 cm^{-1} is assigned to –C=O stretch and 1512 cm^{-1} are attributed to

the presence of $\text{C}-\text{C}$ stretch in aromatic ring. The absorption noted at 1262 cm^{-1} is assigned to the presence of $\text{C}-\text{O}$ stretch (sec alcohol). The band observed at 1069 cm^{-1} are due to the $\text{C}-\text{N}$ stretching. The shifts in IR peak 3404 cm^{-1} to 3426 cm^{-1} shows the presence of $\text{O}-\text{H}$ groups. The peak 2921 cm^{-1} shifts to 2924 cm^{-1} and 2852 cm^{-1} to 2854 cm^{-1} indicating the $\text{C}-\text{H}$ stretching. The peaks shifted from 1619, 1403, 1262 to 1631, 1384 and 1223 cm^{-1} in adsorbed layer. It shows that the $\text{C}=\text{O}$, $\text{C}-\text{O}$ groups may be involved in the formation of barrier layer. The new peaks at 467 and 532 cm^{-1} are observed for the scratched film from the surface of the SS 18/8 orthodontic wire after immersion in artificial saliva (AS) with the addition aqueous extract of *Spilanthes acmella* leaves (10 mL) and it shows the formation of protective complex present in the adsorbed layer on the surface of mild steel.

The obtained FTIR data show the presence of the reported phyto-components in the aqueous leaf extract of *Spilanthes acmella* [35–39]. The plant has been identified for its secondary metabolites like spilanthol, scopoletin, myrecene, alpha amyryn *etc.* Among all the secondary metabolites, the bioactive principle chemical component is spilanthol. It is an alkamide type compound which is present in all aerial parts of the plant (Figure 4).

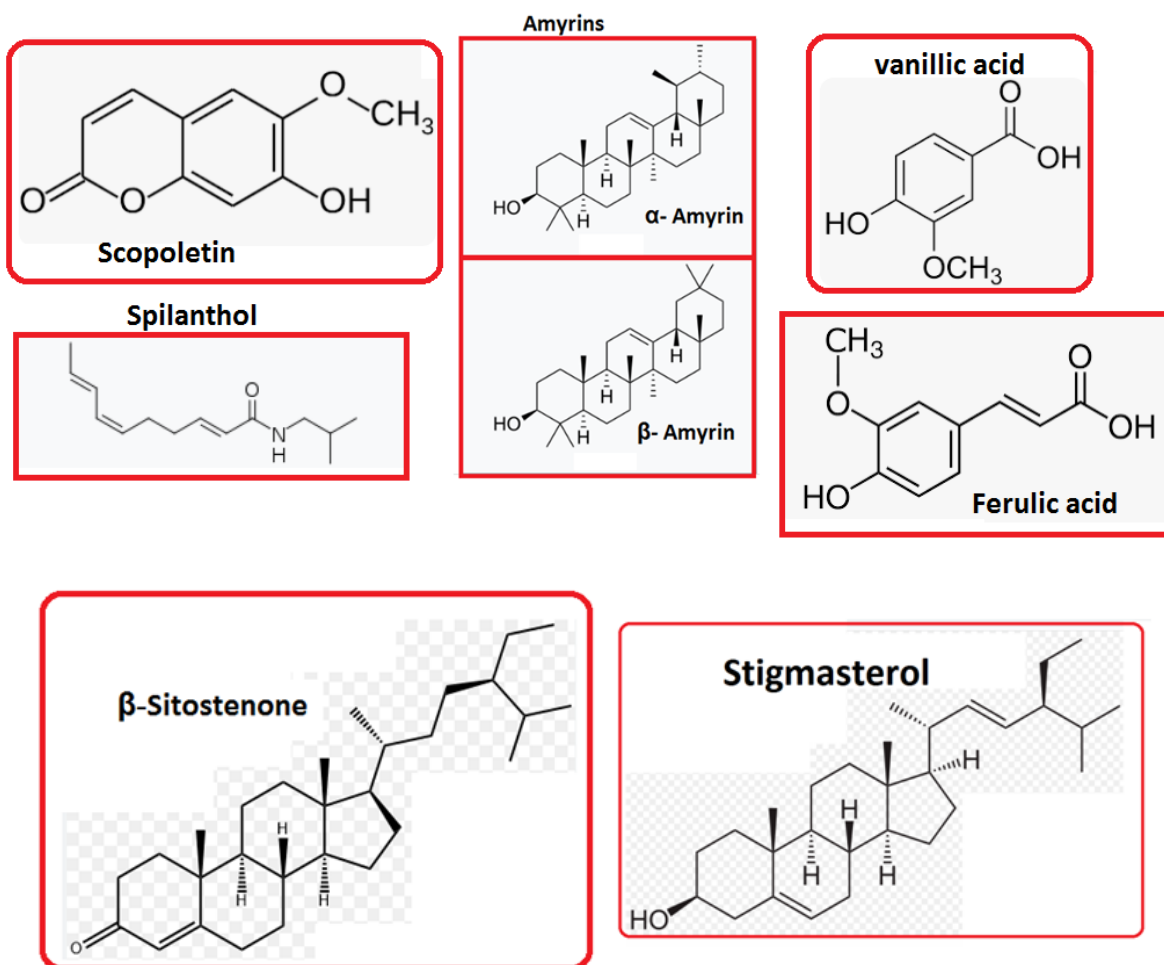


Figure 4. Phytochemical constituents of aqueous extract of *Spilanthes acmella* leaves.

Electrochemical analysis

Potentiodynamic polarization studies

Electrochemical polarization studies have been used to confirm the formation of protective film formed on the metal surface during corrosion inhibition process. If a protective film is formed on the metal surface, the corrosion current value (I_{corr}) decreases and LPR value (linear polarization resistance value) increases. The potentiodynamic polarization curves of SS 18/8 immersed in artificial saliva (AS) in the absence and presence of 10 ml of aqueous extract of *Spillanthes acmella* leaves, obtained from polarization study are shown in Figure 5. The corrosion parameters, namely, corrosion potential (E_{corr} mV vs SCE), Tafel slopes (b_c mV/decade; b_a mV/decade), linear polarization resistance (LPR Ohm·cm²), and corrosion current (I_{corr} A/cm²) values are given in Table 2.

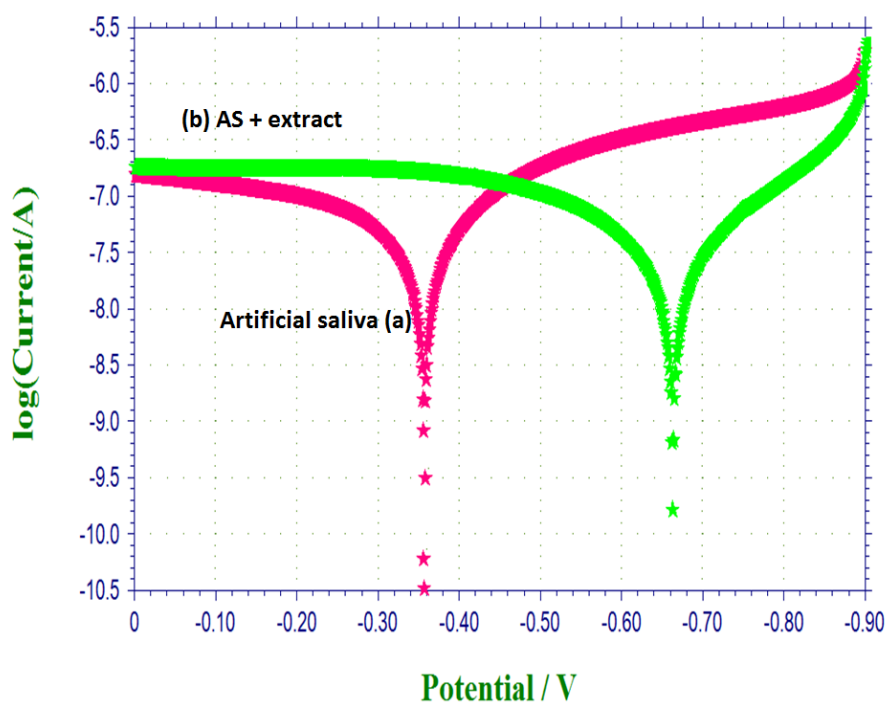


Figure 5. Potentiodynamic polarization curves of SS 18/8 orthodontic wire immersed in artificial saliva (AS) in the absence and in the presence of 10 ml of aqueous extract of *Spillanthes acmella* leaves.

When SS 18/8 is immersed in artificial saliva (AS), the corrosion potential is -357 mV vs SCE. When 10 mL of aqueous extract of *Spillanthes acmella* leaves is added to the above system the corrosion potential is shifted to -663 mV vs SCE.

This indicates that the cathodic reaction is controlled predominantly. In the presence of extract, the LPR value increases from 9.57×10^5 Ohm·cm² to 1.37×10^6 Ohm·cm². The corrosion current decreases from 5.12×10^{-8} A/cm² to 3.00×10^{-8} A/cm². All these observations lead to the conclusion that in presence of 10 mL of aqueous extract of *Spillanthes acmella* leaves the corrosion resistance of SS 18/8 increases [40–43]. Hence

polarization study leads to the conclusion that people having orthodontic wires made of SS 18/8 need not hesitate to take an aqueous extract of *Spillanthes acmella* leaves. The active phytoconstituents have not corroded the orthodontic wires made of SS 18/8; rather they have protected the orthodontic wires.

Table 2. Potentiodynamic polarization parameters of SS 18/8 orthodontic wire immersed in artificial saliva (AS) in the absence and the presence of 10 mL (wt/v%) of aqueous extract of *Spillanthes acmella* leaves.

System	E_{corr} (mV vs SCE)	b_c (mV/decade)	b_a (mV/decade)	I_{corr} (A/cm ²)	LPR (Ohm·cm ²)
AS	-357	184	290	5.12×10^{-8}	9.57×10^5
10 mL aqueous extract of <i>Spillanthes acmella</i> leaves+AS	-663	169	212	3.00×10^{-8}	1.37×10^6

Electrochemical impedance spectroscopy

AC impedance spectra (electrochemical impedance spectra) have been used to confirm the formation of protective film on the metal surface. If a protective film is formed on the metal surface, charge transfer resistance (R_t) and impedance values are increases; double layer capacitance value (C_{dl}) decreases and impedance value increases [44–50]. The AC impedance spectra of SS 18/8 immersed in artificial saliva (AS) in the absence and presence of 10 mL aqueous extract of *Spillanthes acmella* leaves, obtained from AC impedance spectra are shown in Figure 6. The AC impedance parameters namely charge transfer resistance (R_t), double layer capacitance (C_{dl}) and impedance values derived from Nyquist plots are given in Table 3. It is observed that when a 10 ml aqueous extract of *Spillanthes acmella* leaves is added to artificial saliva, the charge transfer resistance (R_t) increases from $22.09 \times 10^3 \Omega \cdot \text{cm}^2$ to $34.98 \times 10^3 \Omega \cdot \text{cm}^2$. The C_{dl} value decreases from $51.26 \times 10^{-2} \text{ F/cm}^2$ to $47.03 \times 10^{-2} \text{ F/cm}^2$. The impedance value increases from 4.75 to 4.99.

Table 3. Electrochemical impedance parameters of SS 18/8 orthodontic wire immersed in artificial saliva (AS) with and without the presence of 10 mL aqueous extract of *Spillanthes acmella* leaves.

System	R_t (Ohm·cm ²)	C_{dl} (F/cm ²)	Impedance, log(Z/Ω)
AS	22.09×10^3	51.26×10^{-2}	4.75
10 ml aqueous extract of <i>Spillanthes acmella</i> leaves+AS	34.98×10^3	47.03×10^{-2}	4.99

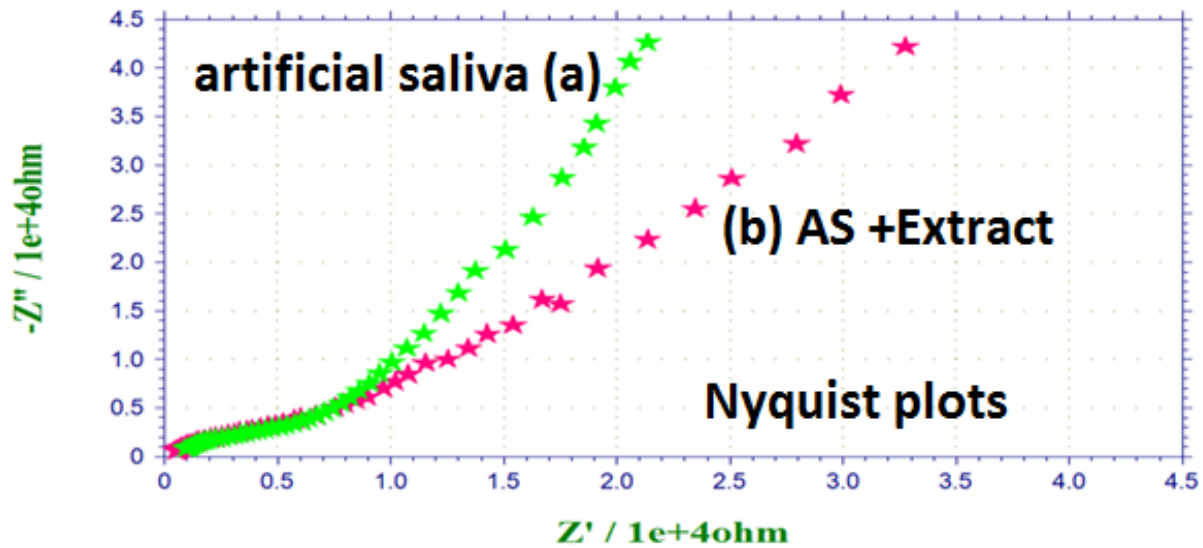


Figure 6. Electrochemical impedance spectra (Nyquist Plots) of SS 18/8 immersed in artificial saliva (AS) in the absence and in the presence of 10 mL aqueous extract of *Spillanthes acmella* L.

Analysis of Atomic Force Microscopy study

The AFM images are useful in predicting the relative smoothness of various surfaces [51–55].

The AFM images of the polished metal surface, polished metal surface immersed in artificial saliva and the protective film formed on metal surface in the presence of artificial saliva+extract are shown in Figures 7–9. The AFM parameters, namely, Average roughness (R_a), nm, Root mean square (RMS) roughness (R_q), nm, and Maximum peak to valley ($P-V$) height, nm are given in Table 4.

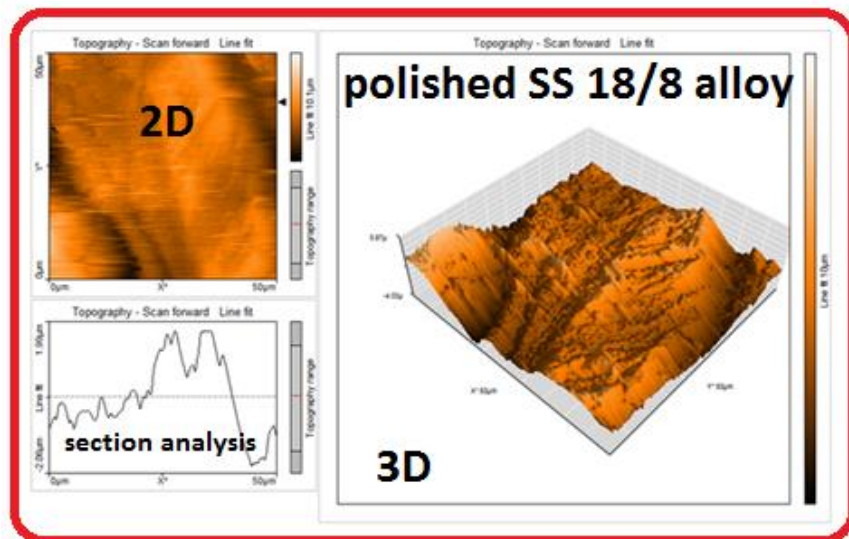


Figure 7. AFM images of the polished SS 18/8.

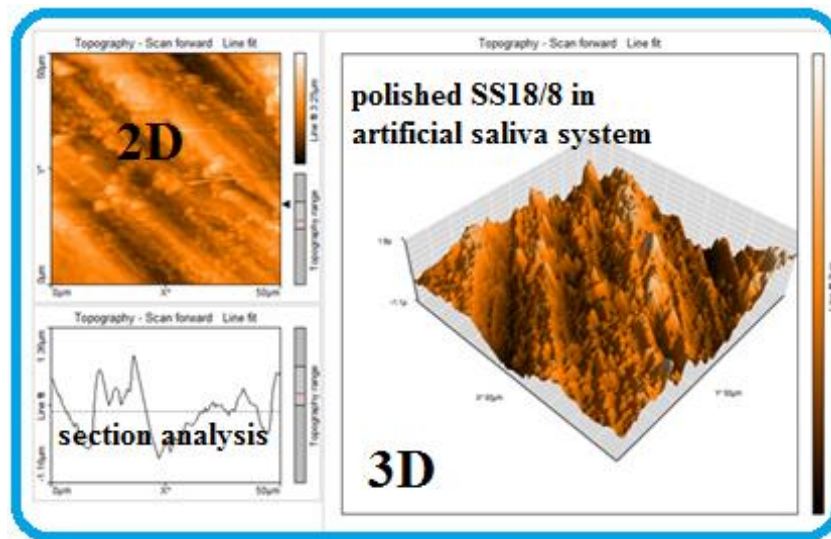


Figure 8. AFM images of the polished SS 18/8 in artificial saliva system.

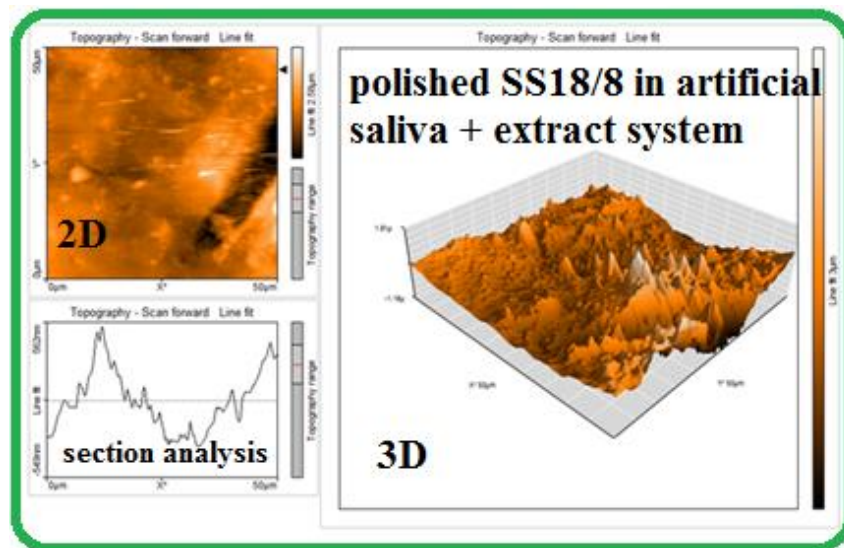


Figure 9. AFM images of the polished SS 18/8 in artificial saliva+extract system.

Table 4. AFM parameters of various metal surfaces.

System	Average roughness (R_a), nm	Root mean square (RMS) roughness (R_q), nm	Maximum peak to valley ($P-V$) height, nm
Polished SS 18/8 orthodontic wire	3.40×10^2	4.39×10^2	1.76×10^3
SS 18/8 orthodontic wire in artificial saliva	8.28×10^2	1.08×10^3	4.51×10^3
SS 18/8 orthodontic wire in artificial saliva+extract	2.94×10^2	3.73×10^2	1.90×10^3

It is observed from Table 4 that when SS 18/8 orthodontic wire in artificial saliva, the Average roughness (R_a) value is very high, when compared with the value for Polished SS 18/8 orthodontic wire. This indicates that the film becomes rough when SS 18/8 orthodontic wire is immersed in Artificial Saliva. Interestingly when SS 18/8 orthodontic wire is immersed in Artificial Saliva+extract system the roughness decreases and becomes smoother. That is the protective nature increases in the presence of extract.

Similar observations are made in the case of other two parameters, namely, root mean square (RMS) roughness (R_q) and maximum peak to valley ($P-V$) height.

Implication

It implies that the corrosion resistance of SS 18/8 orthodontic wire increases when it comes in contact with Artificial Saliva+extract system.

This is due to the presence of the phytochemical constituents [56–60] present in the *Spillanthus acemella* plant leaves aqueous extract.

Conclusion

The corrosion resistance of SS 18/8 orthodontic wire in artificial saliva has been evaluated in the absence and presence of an aqueous extract of *Spillanthus acemella* plant leaves. Electrochemical studies such as polarization technique and AC impedance spectra have been employed. The protective film has been analysed by FTIR and AFM. Polarization technique reveals that in the presence of extract, the corrosion potential of SS 18/8 orthodontic wire is shifted to cathodic side. The LPR value increases and corrosion current decreases. This implies that in the presence of extract the corrosion resistance of SS 18/8 orthodontic wire increases. Similar observation is made in the AC impedance spectra results also. There is increase in charge transfer resistance value, increase in impedance value and decrease in double layer capacitance value. FTIR spectral study reveals that the increase in corrosion resistance is due to adsorption of the active principles present in the extract of the leaves onto the metal surface. The AFM parameters of the AFM images [the Average roughness (R_a) value, Root mean square (RMS) roughness (R_q) and Maximum peak to valley ($P-V$) height] were derived. These values increased when SS 18/8 orthodontic wire was immersed in artificial saliva whereas these values decreased when SS 18/8 orthodontic wire was immersed in artificial saliva+extract system. The outcome of the study is that the patients those who use these metal alloys for orthodontic purpose can chew and place the leaves of *Spillanthus acemella* in the teeth cavities to get relive from pain of toothache without any hesitation and fear.

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