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To cite this article: Hayfaa Abdulameer Abbas *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **571** 012074

Study the Effect of Cyperus Rotundus Extracted as Mouthwash on the Corrosion of Dental Amalgam

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Abstract:
Many extracted material were used as mouthwash to settle different kinds of oral fungi and bacteria. In this study an extractive of Cyperus rotundus plant E.C.R. which is very active as a mouthwash, was used in concentration range (10-25) ppm in artificial saliva at pH=6.4 and temperature range (288-318) K, to study it's inhibitory effect on the corrosion of dental amalgam. Potentiostatic and cyclic polarization were used to follow this study. The presence of E.C.R. lead to shift the corrosion potential to the active direction and decreased the corrosion rate to about 98.87%, the inhibition action of E.C.R. resulting from the adsorption of C.R.E. layer between amalgam and saliva as FTIR spectrums shows, where the variety of FTIR adsorption of many group were noticed for the free E.C.R. and adsorbed E.C.R. layer. Thermodynamic of adsorption, corrosion kinetic parameters were also investigated.
Keywords*:* Corrosion, Inhibitors, Cyperus Rotundus, Extraction, Dental amalgam, Saliva.

1. Introduction

Corrosion is effect on personal safety and industrial applications. Serious environmental contaminations by heavy metals and different undesirable materials have a source of Corrosion. Dental amalgam Corrosion is one of a source of dangerous pollution with mercury in human body [1]. The oral environment dental exposed to a variety of chemical challenges cased a Failure due to either corrosion and/or fracture [2]. Alloys or Metals as biomaterials have been widely used to providing requirement of mechanical strength and corrosion resistance. [3]. Dental amalgam is formed by the reaction of mercury with metallic alloy, alloy powder containing (40–70%), (15–30%) and (10–30%), sliver, tin, and copper, respectively and sometimes a small percentage of zinc [4-6]. Oral Environment is suitable for the formation of corrosion products. The mouth is wet and it is continually exposed to temperature inconstancy [7]. The wide ranges of pH are released during the decomposition of foodstuffs, restorations providing a localized condition favoring an accelerated reaction between oral media and metal or alloy [8].

A substance with a very low concentration is added as inhibitor to reduce the corrosion of a metal surface that exposed to a corrosive environment by blocking site species or adsorption site blocking due to the adsorption properties of inhibitor (9-10).The more effective inhibitors are heterocyclic compounds having nitrogen, oxygen and sulphur atom in their structure [11]. A various plant have been extracted to use as corrosion inhibitors [12] The corrosion inhibition abilities for these compounds varies widely and depending on the yield percentage part of the plant and its location. There is a growing in natural products using which are non-toxic compounds, called green inhibitors, as corrosion inhibitors. In this study the Cyperus rotundus Extract is used as inhibitor for the corrosion of amalgam alloy in artificial saliva.

The name of the plant Cyperus Rotundus has two parts the word cyperus is derived from the Greek kyperos, and rotundus is from Latin, meaning round is commonly known as, Java grass, nut grass, purple nut sedge, red nut sedge [13]. The tuber part of Cyperus Rotundus is one of the oldest known medicinal plants, widely used to treat stomach ailments, wounds, boils and blisters and for a number of biological and pharmacological activities including anti-Candida, anti-inflammatory, antibacterial, antioxidant [14]

IOP Conf. Series: Materials Science and Engineering **571** (2019) 012074 doi:10.1088/1757-899X/571/1/012074

The extract of Cyperus Rotundus contained of various phytoconstituents like steroids, phenolic compounds, flavonoids, glycosides, saponins, triterpenoids, alkaloids, anthroquinones, tannins, quinines coumarins and reducing sugars (15).

2. Materials and Methods

Table 1 show the chemical composition of alloy of the Amalgam that used for corrosion test prepared by amalgamator process, and polished mechanically ,then mounted by using Pyrex-polymer that isolate all side except one, washed with distilled water and hold the specimens for electrical connection as working electrode .

Table 1. The chemical composition of amalgam.

	Ag	Sn	Cu
wt.%	56.7	28.6	14.7

Fusayama artificial saliva is the reference electrolyte that used in corrosion test [16], having a chemical composition closely to natural saliva. Table 2 shows this composition, and with pH equal to 6.2.

Table 2. The composition of artificial saliva.

	KCl	NaCl	$CaCl_2.H_2O$	$NaH_2PO_4.2H_2O$	$Na_2S.9H_2O$	urea
g/l	0.4	0.4	0.906	0.69	0.005	1

The tubers of Cyperus Rotundus, is obtained from Baghdad market. Clean and wash carefully, let to complete dry, fine powdered using electrical grinder.A different concentration range (0, 5, 10, 15 and 20) ppm was prepared in artificial saliva solution to examine using Soxhlet extractor. (A (50) grams of dry powder peel with 500 ml of ethanol, continuous extraction for 24 hours at 60°C, filtering and let too dry at room temperature) .

The behaviors of amalgam filling corrosion investigated by Mlab (Germany, 2000) potentiostate, controlled by MLabSci software. Corrosion cell with three electrodes, (the Calomel electrode was the reference electrode, amalgam filling was a working electrode and platinum electrode was an auxiliary electrode. To establish steady the working electrode was immersed in artificial saliva solution for 15 minutes and recorded the open circuit potential (E_{ocp}), then electrochemical measurements were range (±200) mV from open circuit potential, in aerated solutions at (288-318) K. The effect of temperature in the kinetic process for the corrosion of amalgam in artificial saliva in the presence and absence of inhibitor leads to get more information about electrochemical behavior for metallic materials in aggressive environment [17]. FTIR analysis was carried out using Fourier transform infrared spectrophotometer.

3. Results and Discussion

3.1. Corrosion behavior

The corrosion parameters at temperature range (288-318) K. obtained from the polarization curves were calculated, recorded and listed in Table (3). The values of cathodic Tafel (βc) , anodic Tafel (βa) slopes, corrosion potential (E_{Corr}), corrosion current density (i_{Corr}) and polarization resistance (R_p).Figure (1) shows the potentiodynamic polarization curves obtained for amalgam alloy in free artificial saliva and presence of different concentration of E.C.R. at 288K. It is clearly that the corrosion potential were shifted to more active direction, the polarization curves in different temperatures show same for those at 288K. The potential polarization was more active with 15 ppm E.C.R. reach to (-772 mV). The way in which a metal changes its potential upon immersion in solutions indicates the nature of reaction taking place at its surface and oxide film growth [18].

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Table 3. Corrosion kinetic parameters for amalgam in absence and presence of different E.C.R. concentration in artificial saliva at temperature range (288-318) K.

Temp./K		-OCP /mV	$-E_{corr}$/ mV	I_{corr}/μA $.cm^{-2}$	-ba/ mV $.dec^{-1}$	bc/ mV $.dec^{-1}$	θ	IE %	R_p
Without E.C.R.	288	452	462.2	5.17	146.2	157	-	-	6358.1
	298	435	443.1	6.6	198.1	179.8	-	-	6200.9
	308	422	372.8	6.59	173.9	276	-	-	7029.3
	318	426	384.4	7.2	124	219.6	-	-	4779.4
10ppm C.R.E	288	673	635.5	0.083	364.9	156.8	0.983	98.39	573755
	298	662	658.7	0.101	285.4	196	0.984	98.46	499561
	308	666	664.3	0.114	253.8	209.7	0.982	98.27	437362
	318	669	682.6	0.13	240.7	208.8	0.982	98.19	373456
15ppm E.C.R.	288	762	756.6	0.073	96	96.5	0.985	98.58	286253
	298	730	756.8	0.087	97	117.2	0.986	98.68	264890
	308	773	760.7	0.105	122	118.9	0.984	98.41	249012
	318	779	772.6	0.12	121.4	121.1	0.983	98.33	219369
20ppm E.C.R.	288	650	645.0	0.058	160.3	110.8	0.988	98.87	490480
	298	662	658.0	0.074	168.7	131.4	0.988	98.87	433429
	308	667	662.0	0.101	212.4	112.3	0.985	98.46	315817
	318	681	700.8	0.114	152.9	149.7	0.984	98.41	288112
25ppm E.C.R.	288	522	499.1	0.074	284.9	167.7	0.986	98.56	619420
	298	469	496.3	0.092	107.4	103.9	0.986	98.61	249251
	308	435	426.1	0.103	86.9	175.4	0.984	98.44	244973
	318	788	662.2	0.132	168.4	153.1	0.982	98.16	263796

Corrosion kinetic parameters obtained from table (3) appear the corrosion current density, increased with increasing the temperature in absence and presence of different C.R.E concentrations, while the corrosion current density reduced after add different concentration of E.C.R., and it reach to maximum IE% 98.87% with 20 ppm E.C.R. as shown in Figure (2). In order to discuss the effect of increasing temperature on the corrosion of amalgam alloy in the artificial saliva solution the variation of IE% with increasing temperature were draw Figure (3) The increase of temperature was found to increase the corrosion of the alloy via increasing the cathodic , anodic and currents i_{Corr}, while decreased the values of Rp. This is because the increase of temperature increases the activation of the surface of the alloy, which reflects on its fast dissolution and thus the increase of its corrosion [19].

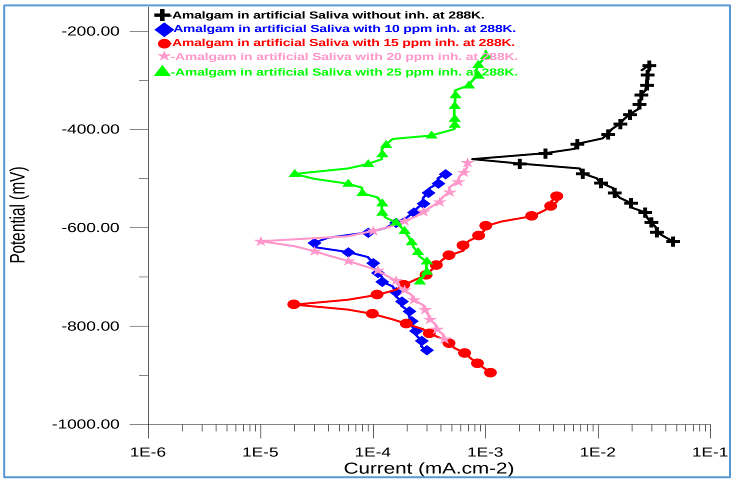

Figure 1: The polarization curve of amalgam in artificial saliva without inhibitor and with different inhibitor concentrations at 288K.

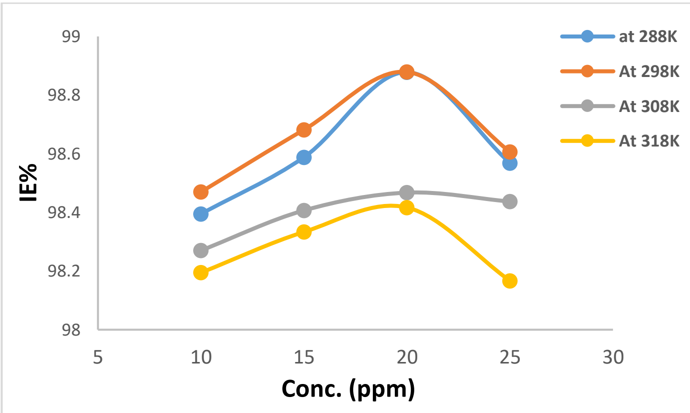

Figure 2. The relation between inhibitor efficiency (%IE) and concentration of E. C.R. in artificial saliva at different temperature.

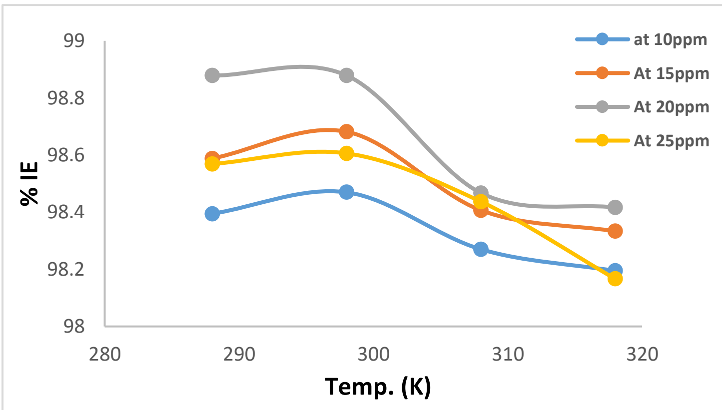

Figure 3. The inhibitor efficiency (%IE) and different temperature at various E.C.R. concentrations

3.2. Cyclic Polarization Curves:

The alloys and materials suggested for use as a biomaterial which must exhibit excellent resistance to localized corrosion, cyclic polarization measurements were performed in order to determine the tendency of the alloys to undergo pitting or crevice corrosion when immersed in the corrosive solutions. A plot of potential versus log of the current density reveals the tendency of the surface material to undergo localized attack. Figure (4) show the cyclic polarization curves for the corrosion of amalgam in absence and presence of E.C.R. the reverse scan shifted to lower current densities, as well as reduce the hysterias loop when add 20ppm E.C.R. So use of E.C.R. reducing the pitting corrosion in amalgam alloy. This type of cyclic polarization curve is known to resist localized corrosion [20].

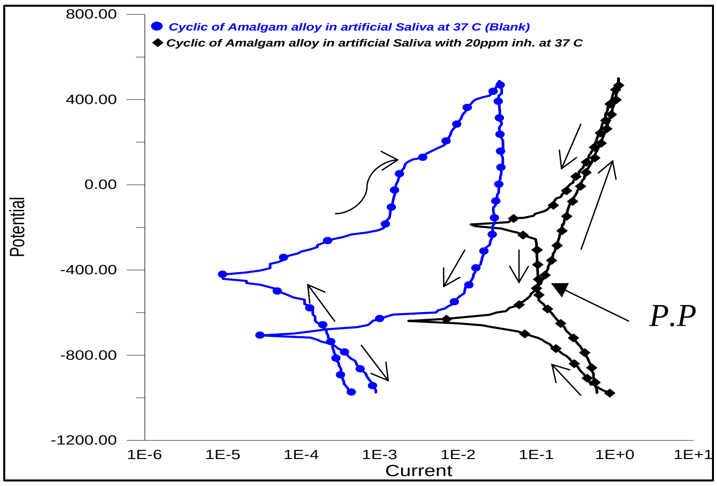

Figure 4 .Cyclic polarization curves recorded for amalgam alloy in artificial saliva in absence and presence 20ppm E.C.R. at 35˚C.

3.3. Kinetic parameters for the corrosion inhibition process

According to Arrhenius equation, the activation energy for corrosion reaction can be calculated, expressed as follows:

$$i_{corr} = A\ e^{-Ea/RT} \ldots\ldots\ldots (1)$$

Where i_{corr} is the corrosion rate (corrosion current density), A is the pre-exponential factor, R is the gas constant, T is the absolute temperature and Ea is the activation energy.

Figure (5) presented the linear regression, between $\log i_{corr}$ and 1/T. The calculated activation energies (Ea) and pre-exponential factors (A) are listed in Table (4).). The value of evident activation energy Ea of hydrogen evolution reaction for artificial saliva without E.C.R. (7.63 kJ.mol-1) agrees well with literature data on Ea for amalgam alloy in artificial saliva [21]. In the presence of E.C.R. the process of metal dissolution is characterized by activation energy which is more than that in the artificial saliva without E.C.R. The Values were reached to (17.85 kJ.mol^{-1}) in presence 20 ppm E.C.R. proved that the presence of E.C.R. in artificial saliva would result in structural change of the double layer at the metal/water interface and reduced the rate of electrochemical reaction. Adding E.C.R. lead to decease values which related to decrease the number of corrosion sites or pitting corrosion sites [22].

Table 4. The thermodynamic parameters for the corrosion of activation complex (Activation energy (Ea), pre exponential factor (A))

C_{inh}	ΔG^* /kJ.mol^{-1}				ΔH^*/kJ .mol^{-1}	$-\Delta S^*$/ kJ.mol^{-1} .K^{-1}	Ea/kJ. mol^{-1}	A Molecules. cm^{-2} .S^{-1}
	288	298	308	318				
without	66.40	68.53	70.66	72.78	5.115	0.2128	7.63	7.9E+25
10ppm	76.38	78.73	81.08	83.43	8.684	0.2350	11.19	5.4E+24
15ppm	76.72	79.03	81.34	83.64	10.285	0.2307	12.80	8.6E+24
20ppm	77.25	79.41	81.56	83.71	15.34	0.2149	17.85	6.1E+25
25ppm	76.69	78.96	81.22	83.48	11.54	0.2262	14.06	1.6E+25

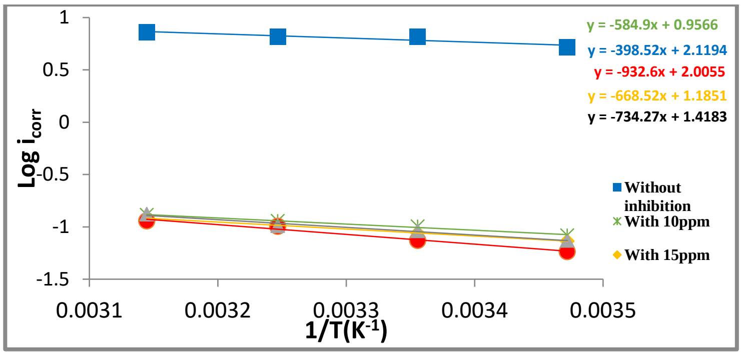

Figure 5. A Plot of $\log_{icorr}$ versus 1/T for the corrosion of amalgam in free artificial saliva and presence containing various E.C.R. Concentrations.

From the result of effect of temperature, thermodynamic parameters for activation such as ΔG^*, ΔH^* and ΔS^* were calculated table (4). The plot of $\log i_{corr}$/T Vs 1/T. ΔH^*, ΔS^* were obtained from the slope and intercept respectively figure (6), dependent to Arrhenius equation [23]. Where the value of enthalpy of activation increased with add C.R.E in saliva solution, were reached to 15.34 KJ.mol^{-1} with 20 ppm. The values of entropy of activation are negative reflect that the inhibitor molecules, lead to more ordering transition-state. ΔG^* values were positive and showed slightly increase with increasing temperature, which means that inhibitor cause to reduce the thermodynamic feasibility of corrosion transition-state. [24]

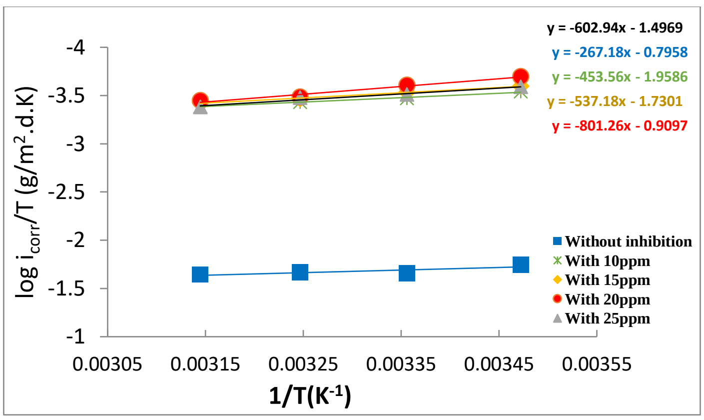

Figure 6. Plot of 1/T Versus log CR/T for the corrosion of amalgam in free artificial saliva solution and presence of different Concentrations of inhibitor.

3.4. Adsorption isotherm:

The adsorption behavior of the inhibitor compounds on the amalgam surface, isotherms are usually used to describe this process which depends upon the nature of the metal surface, electronic characteristics of the metal surface, adsorption of solvent, other ionic species, temperature of the corrosion reaction the charge,, and the electrochemical potential at the metal solution interface [25] . (θ) is The degree of surface coverage for different concentrations of C.R.E. it evaluated from polarization measurements data by using the following equation [26]

$$\theta = \frac{IE\%}{100} \quad \text{.........(2)}$$

Several adsorption isotherms were fitted for the experimental data and the Langmuir adsorption isotherm was found to be the best expression of the adsorption behavior of E.C.R. which obeys the following equations:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad \text{....... (3)}$$

Where K_{ads} is the adsorption/desorption equilibrium constant, C_{inh} is the inhibitor concentration, and (θ) is the surface coverage. The plot of C_{inh}/θ vs. C_{inh}. Figure (7) gave a straight line with an intercept of $1/K_{ads}$. The results obtained isotherm is presented in table (5), All the linear regression coefficients at different temperatures are more than 0.99, which indicates that the adsorption process can be expressed as Langmuir adsorption [27].

Table 5. Thermodynamic parameters for adsorption of the inhibitors on the surface of amalgam in saliva

Temp.	K_{ads}	ΔG_{ads}/ kJ.mol^{-1}	R^2	$-\Delta S_{ads}$/ J.K^{-1}.mol^{-1}	$-\Delta H_{ads}$/ kJ.mol^{-1}
288	35.84	-18.1902	0.999	20.637	12.2465
298	59.52	-20.0788	0.999	26.282	
308	38.61	-19.6439	0.999	24.017	
318	70.92	-21.8897	0.999	30.324	

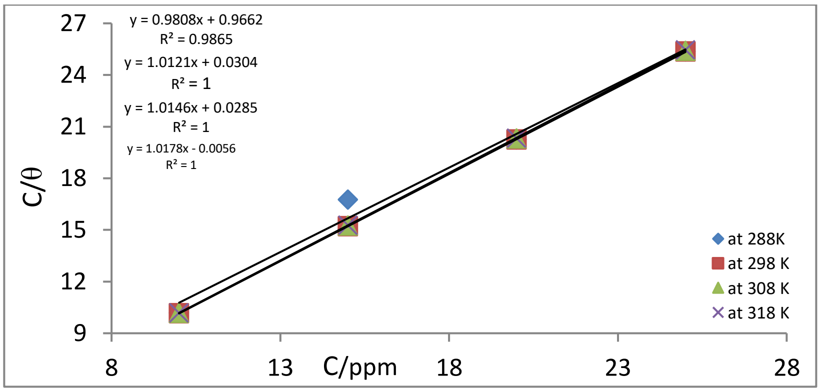

Figure 7. Langmuir isotherm plot for the adsorption of E.C.R. on the surface of amalgam.

Thermodynamic function for the adsorption process (ΔH_{ads}, ΔS_{ads} .ΔG_{ads}) can be calculated by using the known formulas [28].

$$K_{ads} = \frac{1}{55.5}\exp[\frac{-\Delta G_{ads}}{RT}] \quad \text{........ (4)}$$

This equation can also be expressed as:

$$\Delta G_{ads} = -2.303RT\log(55.5K_{ads}) \quad \text{........ (5)}$$

The integrated of vant Hoff equation can be used to calculate the enthalpy of adsorption ΔH_{ads} and entropy of adsorption ΔS_{ads} and explained the thermodynamic model for inhibition effect of E.C.R. on amalgam surface from Plotted $\log K_{ads}$ against 1/T (figure 8) the straight line with slope equal to ($-\Delta H_{ads}/2.303R$) and intercept equal to ($\Delta S_{ads}/2.303R + \log 1/55.5$). These values (the heat of adsorption and entropy of adsorption) are listed in table (5)

$$\log K_{ads} = \frac{-\Delta H_{ads}}{2.303RT} + \frac{\Delta S_{ads}}{2.303R} + \log\frac{1}{55.5} \ldots\ldots\ (6)$$

,

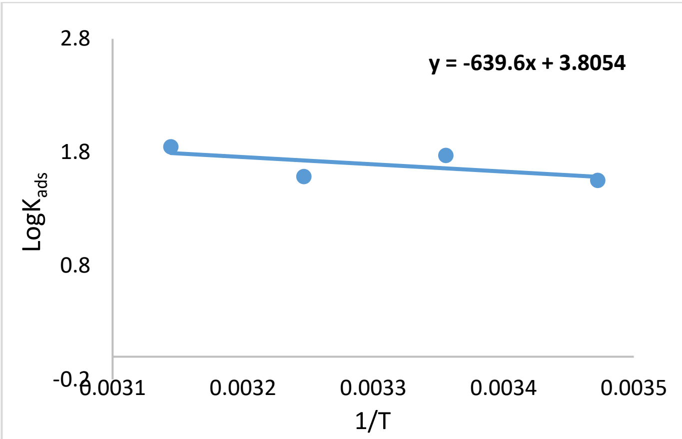

Figure 8. The relation between $\log K_{ads}$ against 1/T.

3.5. FTIR & Inhibition Mechanism

The adsorption process will be dependent on factors such as the extract composition and type of corrosive medium ions .The major compounds that isolated from the extracts of Cyperus rotundus are identified as, 5-hydroxymethyl furfural) (1), methyl ferulate (2), ferulaldehyde (3), and N-*trans*-feruloyl tyramine (4) (Figure 9).

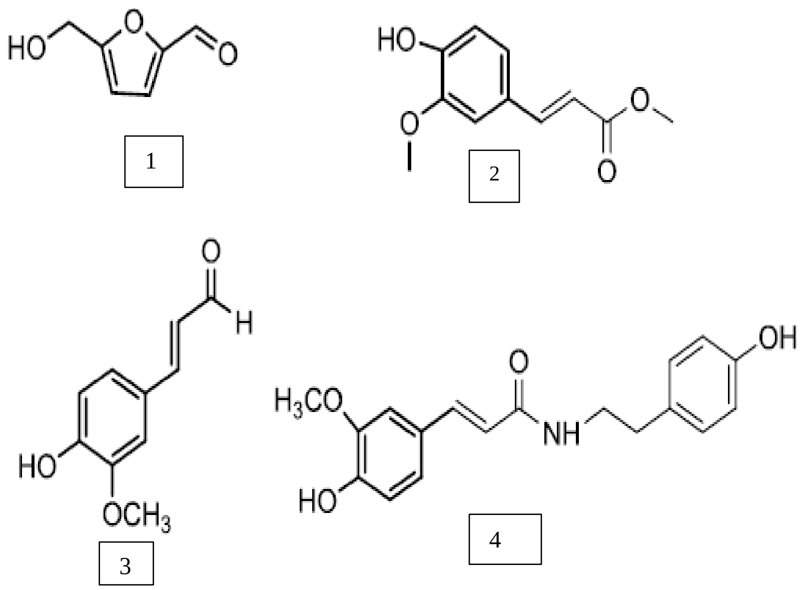

Figure 9. Shows structures of most important phytochemicals found in C.R. Extract. [29]

The inhibition effect is refer to structures of the extracted compounds from C.R. that contain many oxegen atoms in there functional groups, π-electrons and heterocyclic rings .These compound may be adsorbed on the amalgam surface by displacement of water molecules from the amalgam surface and the electrons of the oxygen atom and silver, or by adsorbed on the amalgam surface via the basis of acceptor- donor interactions between vacant d-orbitals of silver and p-electrons of the heterocycle in chemisorption mechansium .

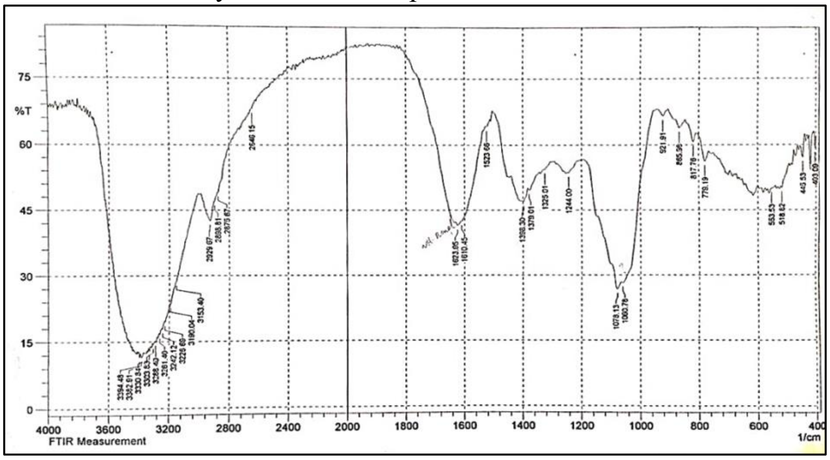

Figure 10. FT-IR spectra of pure Galangal Extract (C.R.E) .

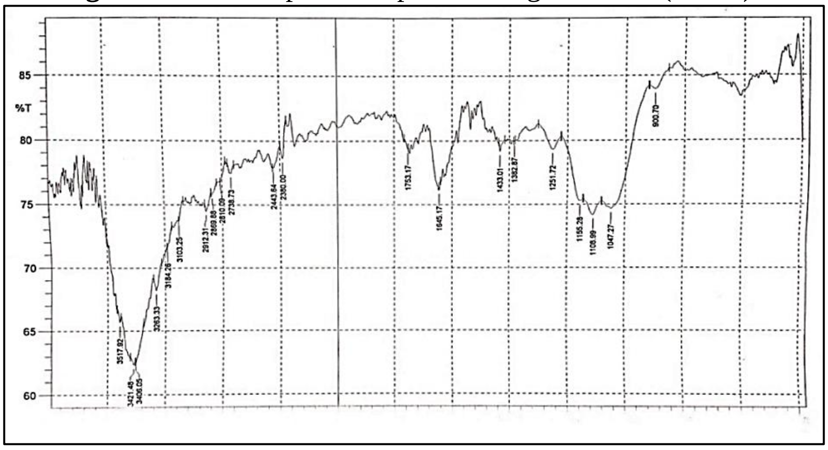

Figure 11. FT-IR spectra of pure and adsorbed film.

The FT-IR spectra in (Figuer 19 and Figuer 11) compare between FT-IR spectra for isolated pure E.C.R. and it thin film on the amalgam alloy surface, the absorbance frequency of functional groups have been shifted to less or high value due to the force of binding between the interface of amalgam surface and the inhibitor, Table 6. Also the figures appear that the vibration of all functional group that affected by inhibition process have a less intensity in transition bands.

Table 6. Wave number of FT-IR functional groups adsorption for pure (E.C.R.) and (E.C.R.) as adsorbed inhibitor.

Pure (E.C.R.)	Corrosion product	Group
Wave number (cm^{-1})	Wave number (cm^{-1})	Assignment
3394.48	3406.05	O-H
2929.67	2912.31	C-H
1623.95	1645.17	C=C
1244.00	1251.72	C-O-C for ether
1078.13	1108.99	C-O for alcohol

IOP Conf. Series: Materials Science and Engineering **571** (2019) 012074 doi:10.1088/1757-899X/571/1/012074

Conclusions

The use of E.C.R. as a mouthwash lead to decrease the corrosion of dental amalgam and act as an inhibitor in artificial saliva, and prevent the amalgam from pitting corrosion.

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