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Continuous Flow Reactor

Aluminum in Acidic Solution

Highlights

Detection of p-Anisidine

Oils in Supercritical Methyl

Discovering Thoughts, Inventing Future

VOLUME 16 ISSUE 1 VERSION 1.0

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An Electrochemical Sensor for the Detection of P-Anisidine through Electrochemistry

By A. Zaroual, S. El Qouatli, A. Bellouchou, A. Guenbour, R. Najih & A. Chtaini
University Sultan Moulay Slimane

Abstract- In this study, a sensitive electrochemical voltammetry method for the analysis of p-anisidine (pA) using a carbon paste electrochemical (CPE) modified with a porous material, such as natural phosphate (NP), was proposed. p-anisidine strongly adsorbed on a electrode surface and prepared electrode NP-CPE provides easy methods for electrochemical quantitative electro-p-anisidine. Operational parameters have been optimized, and performance voltammetric stripping was investigated by cyclic voltammetry (CV). The current intensity peaks are very linear, with good sensitivity NP- CPE.

Keywords: modified electrodes; cyclic voltametry; natural phosphate; moringa oleifera p-anisidine; carée wave voltammetry.

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A. Zaroual [α], S. El Qouatli [σ], A. Bellouchou [ρ], A.Guenbour [ω], R. Najih [¥] & A.Chtaini [§]

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I. Introduction

Para-anisidine (p-anisidine), is the most toxic [1] of the three isomers of anisidine and causes damage to the blood when ingested orally, by inhalation or skin contact. If heated strongly it can release highly toxic fumes of oxides Trogen lev- [2-4]. Melting and boiling point 243°C P-anisidine (PA) is colorless crystal with point 57.2 it is an important intermediate for the synthesis of dyes, medicine and fragrances, pigments, and other chemical compounds [5-7].

P-Anisidine reacts with secondary oxidation products such as aldehydes and ketones in the fats and oils to form products which absorb at 350 nm of light wavelength; therefore, it is used as an official method for detection them by the American Oil Company Chemistsy [8-9]. It is particularly good at detecting unsaturated aldehydes, which are those most likely to generations at unacceptable flavors, making it especially useful in food quality testing [10]. Traditional preparation of p-Anisidine uses iron powder or sodium sulfide as reducing agent [11], which reproduces a large quantity of waste and results in the problem of serious environmental pollution. [12] In this article, we describe the electrochemical analysis of p-anisidine on a modified clay carbon paste electrode. The electrochemical characterization of adsorbed p-anisidine was assessed using cyclic voltammetric (CV) and square wave voltammetry (SQW).

II. Experimental

a) Reagents

Potassium nitrate was dissolved into Bidistilled deionized water (BDW) to form 1mg.L^{-1} stock solutions. Working standards for calibration were prepared by diluting the primary stock solution with BDW. Carbon paste was supplied from (Carbon, Lorraine, ref. 9900, French). All chemicals were of analytical grade and used without further purification.

b) Electrodes preparation

Firstly, the carbon-paste electrode was prepared according the following procedure [13]. The carbon-paste electrode was prepared by mixing the graphite powder with paraffin oil used as a binder.

The mixture was grinding in a mortar agate and then a portion of the resulting composite material was housed in PTFE cylinder. The geometric surface area of the working electrode was 0.1256cm^2. A bare of carbon vitreous inserted into carbon paste provided the electrical contact, and then the Phosphate natural film is electrodeposited onto carbon paste electrode. The deposit of Phosphate natural on carbon paste electrode surfaces was processed at 20 V. The current was maintained by a galvanostat with a function generator.

c) Apparatus

Electrochemical experiments were performed using a voltalab potentiostat (model PGSTAT 100, Eco Chemie B.V., Ultrecht, The Netherlands) driven by the general purpose electrochemical systems data processing software (voltalab master 4 software).

All the electrochemical experiments were performed in a standard one–compartment three-electrode cell. The reference electrode was SCE and the counter electrode was platinum. All electrode potentials were referred to this reference electrode. The working electrode was natural phosphate modified carbon paste electrode (NP-CPE).

III. Results and Discussion

a) Characterization of prepared electrodes

The surface structure of NP- CPE surface was observed using scanning electron microscopy (Fig. 1). The film layer of NP was formed on the surface of

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carbon paste electrode; it was not disintegrated or detached from the surface when immersed in the electrolytic solution (0.1M Na_2SO_4). The treatment described previously gives compact particle fractions between 100 and 400μm rich in phosphate. Natural phosphate treaty has the following chemical composition: CaO (54.12%), P_2O_5 (34.24%), F (3.37%), SiO_2 (2.42%), SO_3 (2.21%), CO_2 (1.13%), Na_2O (0.92 %), MgO (0.68%), Al_2O_3 (0.46%), Fe_2O_3 (0.36%), K_2O (0.04%) and order of several ppm metals.

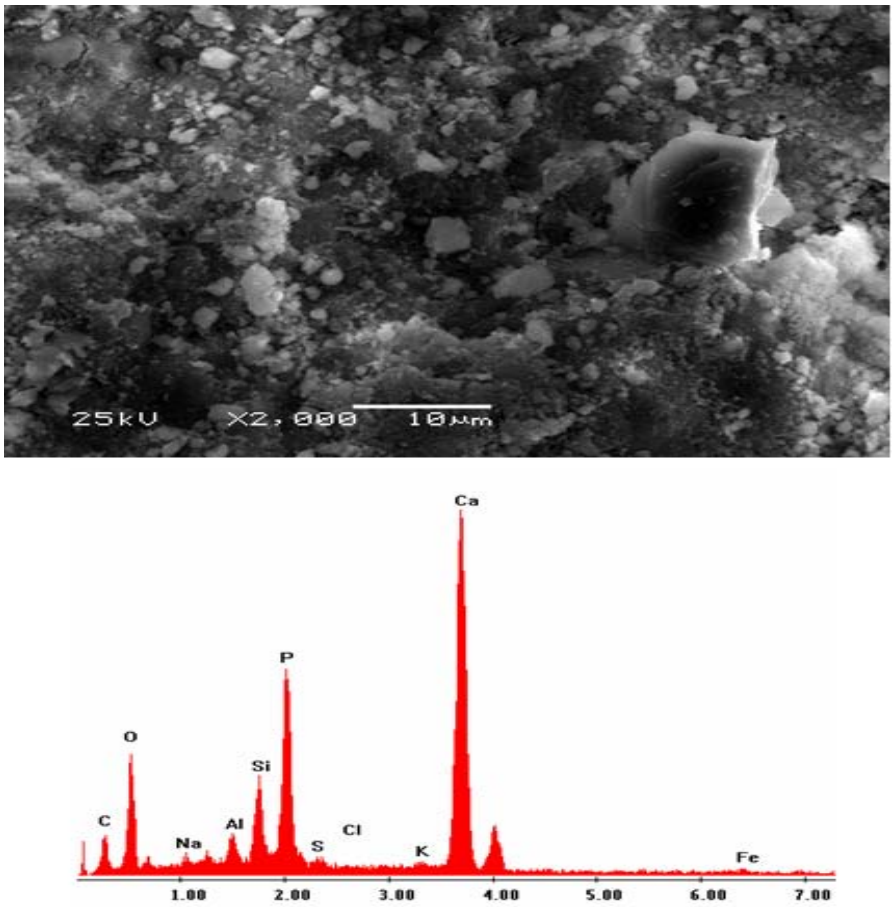

Figure 1 : Scanning electron micrograph of NP-CPE

b) Voltammetric characteristic of p-anisidine

The determination of p-anisidine at the NP-CPE was performed by using cyclic (CV) and square wave voltammetry (SQW). The results are presented in Figures 2 and 3 show the CV and the SQW measurements, respectively. Two oxidation peaks were observed at NP-CPE towards the positive sweep direction, the first one around −0. 2 V and the second at approximately 0.15 V versus SCE, scanning in the negative sense brings up a cathodic peak at -0.4V. Fig. 3 shows the square wave voltammograms obtained in 0.1 mol L^{-1} Na_2SO_4 for unmodified and modified carbon paste electrode. When the NP-CPE was dipped into the accumulation medium containing p-anisidine followed by square wave voltammetry two well defined peaks appeared.

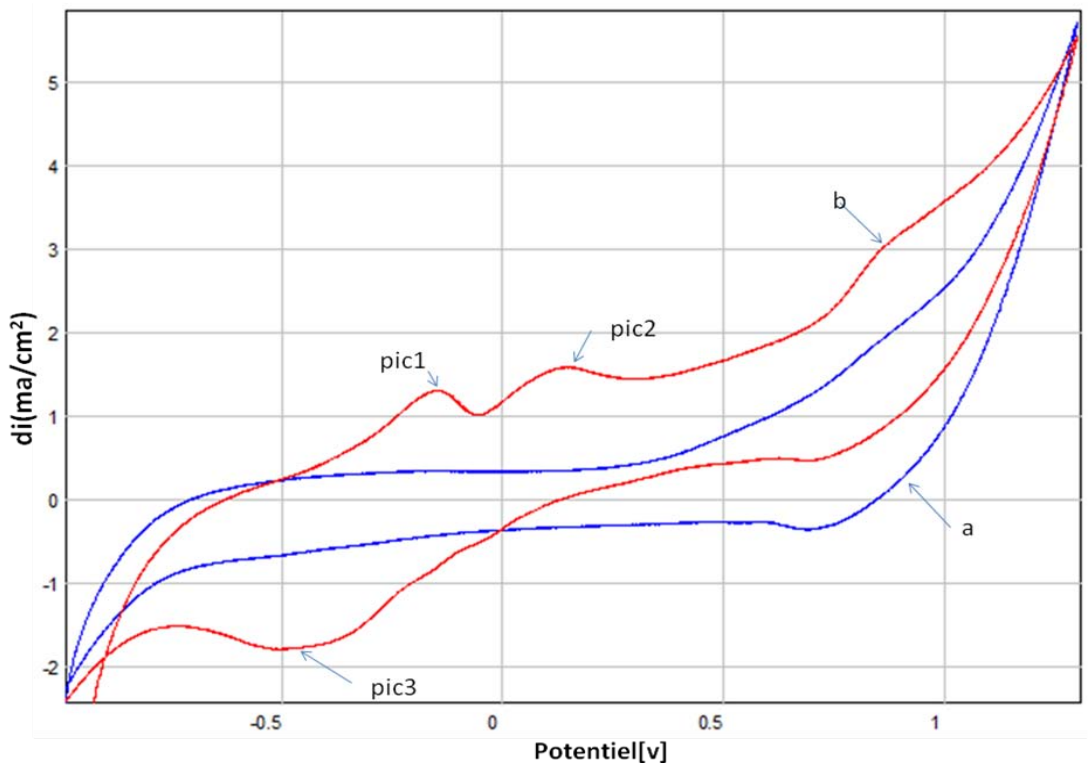

Figure 2 : Cyclic voltammetry recorded at NP-CPE in 0.1 M Na_2SO_4 solution
a - without p-anisidine b - with p-anisidine , pH = 7, accumulated time = 10min

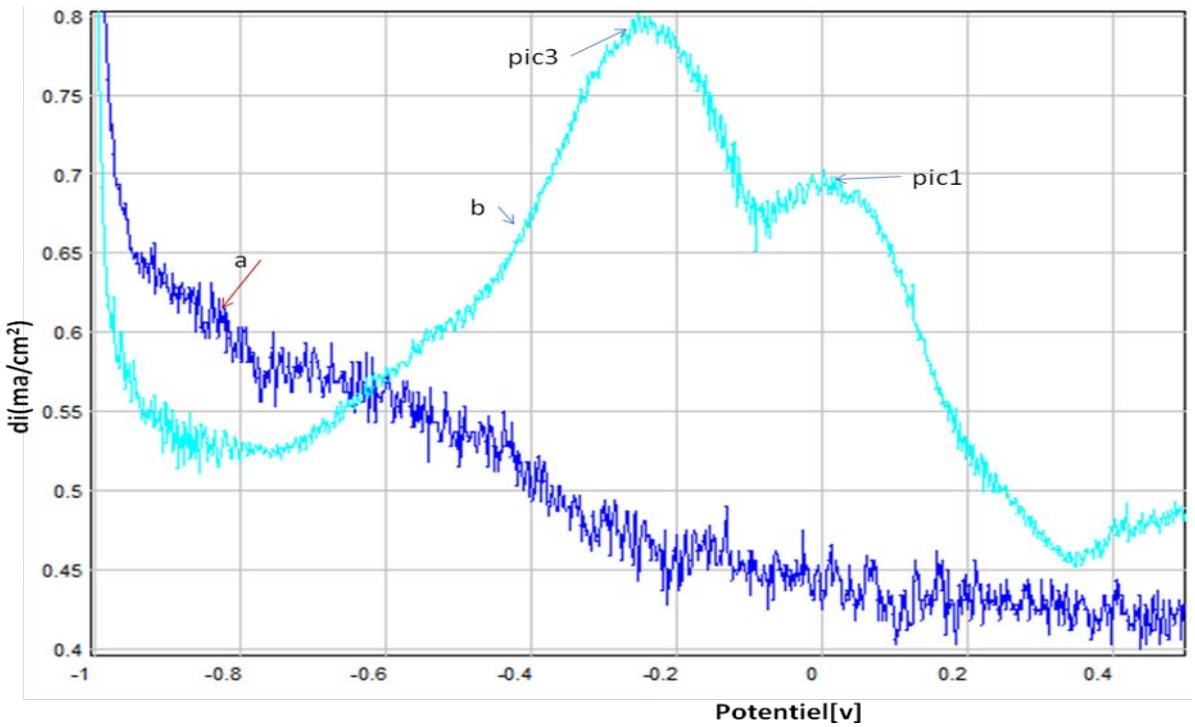

Figure 3 : Square wave voltammetry recorded at NP-CPE in 0.1 M Na_2SO_4 solution
a - without p-anisidine, b - with p-anisidine, pH = 7, accumulated time = 10min

c) Calibration graph

Under these selected conditions, the peak current increased linearly with the p-anisidine concentration in solution using a preconcentration time of 10 min (Figs. 4 and 5). The linear dynamic range was comprised between 0.8 and 2.4 mM in terms of the relationship between p-anisidine concentration and the oxidation peak current (Fig. 6). . The relationship can be described in the following linear regression equation in the mentioned concentration range:

$IP1 = 0{,}105[P\text{-}A] + 0{,}438 \quad R^2 = 0{,}983$

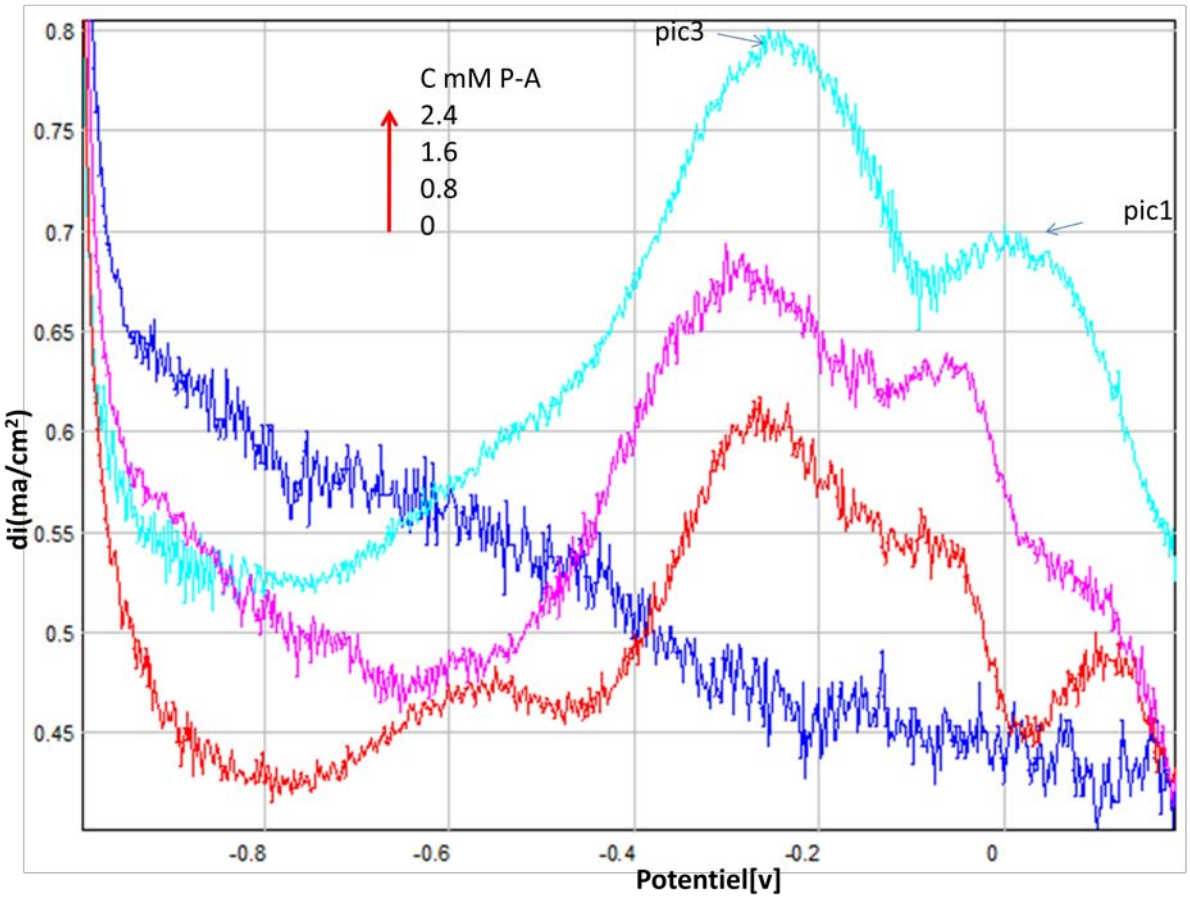

Figure 4 : Square wave voltammograms at different concentration of p-anisidine recorded at NP-CPE in 0.1M Na_2SO_4,

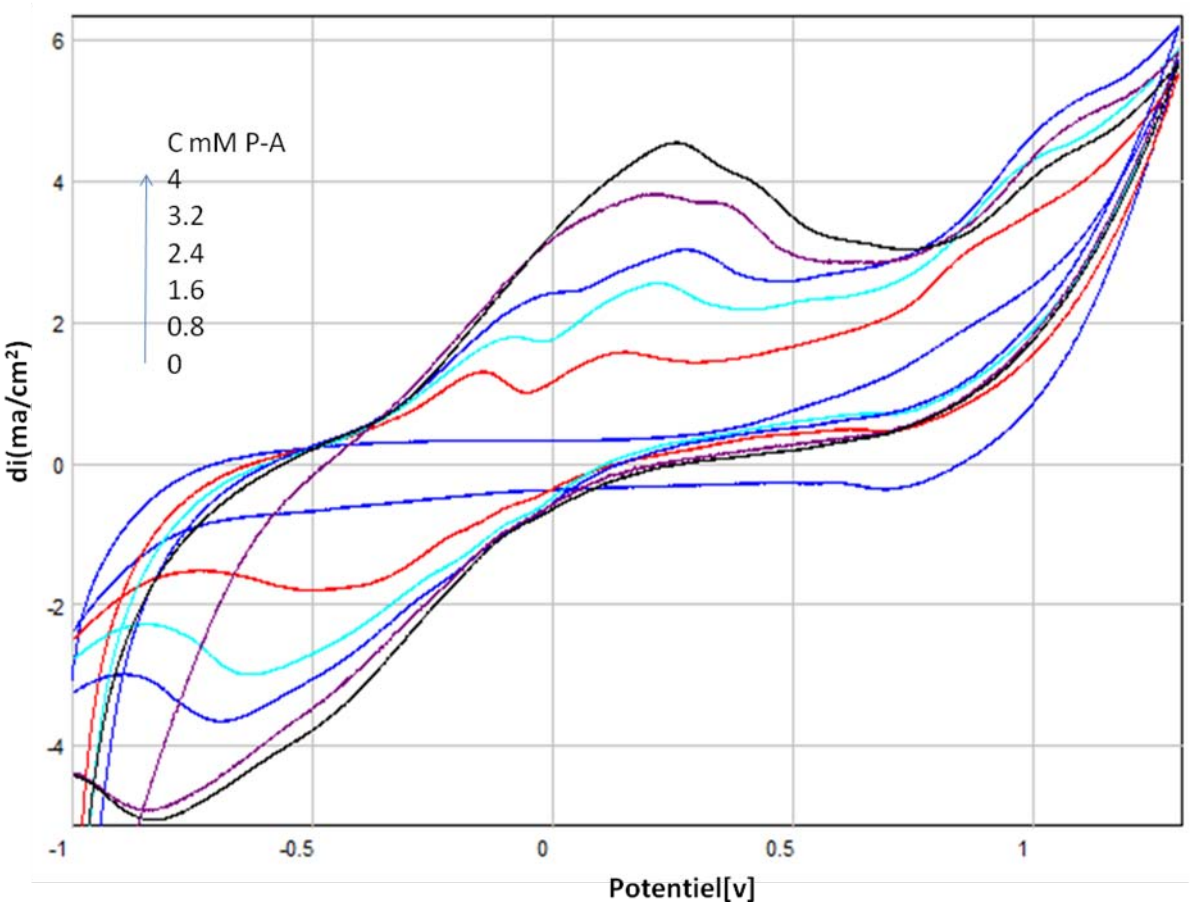

Figure 5 : CV's recorded at NP-CPE, in different concentration of p-anisidine, the scan rate and 100 mV / s

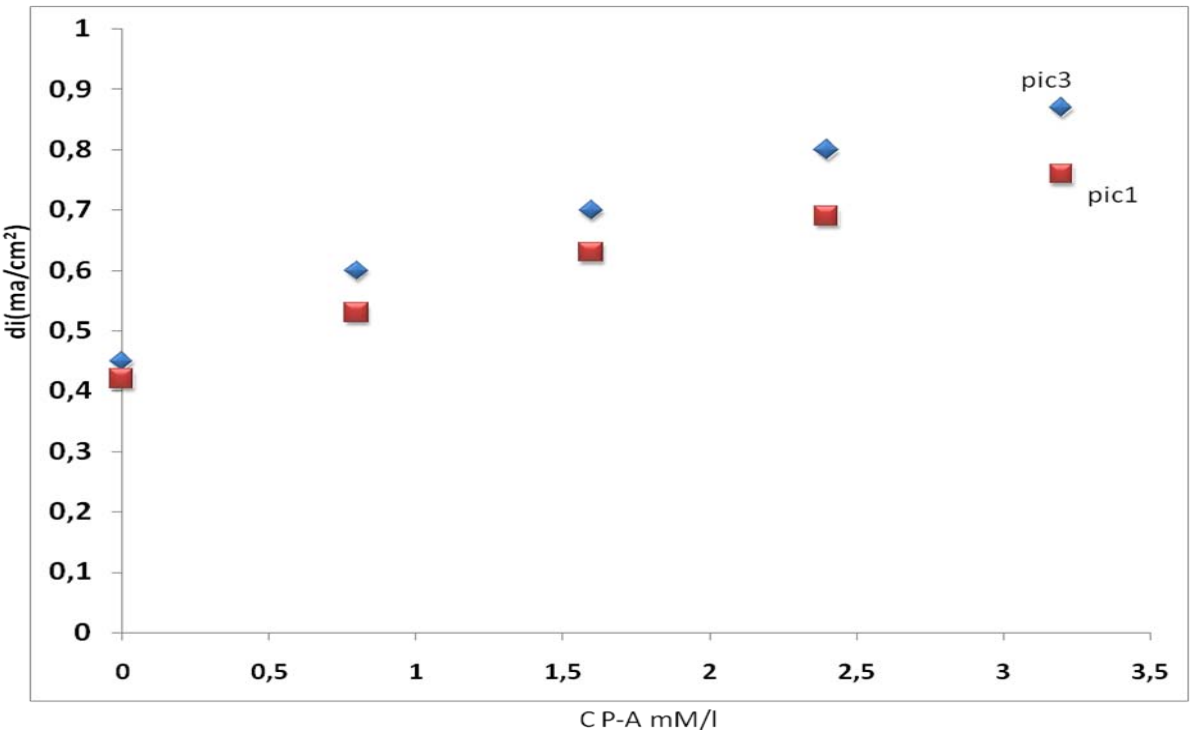

Figure 5 : Plot of peaks area versus added concentration of p-A

IV. Conclusion

A new chemically modified carbon paste electrode has been developed with natural phosphte for the determination of p-anisidine at trace levels by square wave and cyclic voltammetry. The electrode offers attractive properties such as simplicity of electrode preparation. Also, there is no leaching of the electrode because of the low solubility of the NP in aqueous solution.

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Expired Cidamex Drug as Corrosion Inhibitor for Aluminum in Acidic Solution

By M.M.Motawea, H.S.Gadow & A. S. Fouda
Delta Higher Institute for Engineering and Technology

Abstract- The inhibitive effect of expired Cidamex drug on aluminum in 1M HCl solution was studied using weight loss, hydrogen evolution, potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM) techniques. The results indicate that Cidamex is good inhibitor and inhibition efficiency improved with concentration and reached 99.6% at 300 ppm. Thermodynamic activation parameters that govern the process were deduced from the temperature dependence. Potentiodynamic polarization curves indicated that this drug behaves as mixed-type inhibitor. This drug was adsorbed on aluminium surface follows Langmuir adsorption isotherm. The results obtained from all investigated techniques are in good agreement.

Keywords: *expired cidamex drug, corrosion inhibitors, inhibition efficiency, aluminum, HCl.*

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Keywords: expired cidamex drug, corrosion inhibitors, inhibition efficiency, aluminum, HCl.

I. Introduction

Aluminum and its alloys are widely used materials for their excellent electrical and thermal conductivities in many applications and recently in the manufacture of integrated circuits [1, 2]. So, the study of its corrosion inhibition is of great importance. The most widely used pickling acid is the hydrochloric acid, so this medium induced a great deal of research on aluminum [3-5]. A number of organic compounds are known to be applicable as corrosion inhibitors for aluminum in acidic environments. Such compounds typically contain nitrogen, oxygen or sulphur in a conjugated system and function via adsorption of the molecules on the metal surface, creating a barrier to corrodent attack [6-9]. The adsorption bond strength is dependent on the composition of metal, inhibitor structure and concentration as well as temperature [10]. Because of the fact that most of the chemical compounds that prevent the corrosion of metals and alloys are toxic, and thus pose threat both for human health and environment, their usage is limited. For this reason, several authors reported the use of natural products as corrosion inhibitors [11]; also, some authors used drugs as green corrosion inhibitors for various metals and alloys [12-20]. The use of environmental friendly pharmaceutical compound (expired Cidamex) as corrosion inhibitor for aluminum has not been reported before. Most of the pharmaceutical active substances are far more expensive than the organic inhibitors currently implemented. Therefore, our study was focused on the usage of expired drugs or unused drugs because of patient's non-compliance that contain in their composition active substances with inhibitory properties. This will solve two problems: a) Limitation of environmental pollution with pharmaceutically active compounds and b) reduction of the disposal costs of expired drugs.

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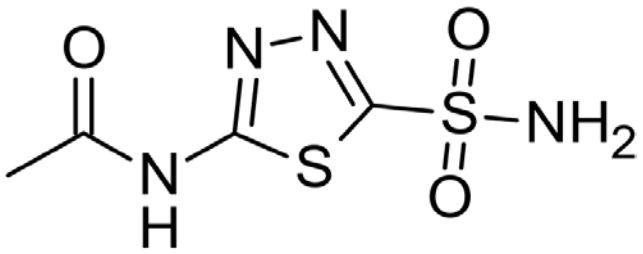
II. Experimental

a) Materials and Solutions

The investigated compound (expired Cidamex) shown in Figure1, this drug was obtained from CID Giza Co. for Pharmaceuticals, Egypt., Analytical grade HCl (37%) was used as corrosive solution. Double distilled water used throughout experiments for the preparation of solutions.



Formula = $C_4H_6N_4O_3S_2$, Mol. Mass = 222.245

Figure 1 : Structure of expired Cidamex Drug *[N-*(5-sulfamoyl-1,3,4-thiadiazol-2-yl)] acetamide

b) Methods and Techniques

i. Weight loss method

The weight experiments carried out using specimens of aluminum having dimensions (2 x 2 x 0.05 cm) and with composition more than 99.9%. The test pieces of aluminum samples were weight up to fourth decimal place using digital electronic balance. The aluminum specimens were polished by a series of emery paper (grade 320-1200 grit size) and then washed with double distilled water and acetone. After weighing, the specimens were totally suspended in

beakers containing test solutions using glass hooks at temperature (25 and 45^{0}C) in thermostat water bath. Each piece taken out of the test solution, rinsed with double distilled water, dried between two filter papers and weighed again. The difference in weights for an exposed period was taken as weight loss. The experiments carried out at various concentrations of Cidamex. Triplicate samples were used to check reproducibility of results.

ii. *Hydrogen evolution and corrosion rates*

The gas-volumetric technique provides a rapid and sensitive method of monitoring any perturbation by an inhibitor regarding gas evolution at the metal-corrodent inter phase. The corrosion of aluminum in acid solution is characterized by rapid effervescence resulting from hydrogen gas evolution. The corrosion rates of aluminum in the absence and presence of drug assessed by measuring the volume of H_2 gas evolved during the corrosion reaction. An ideal device for hydrogen evolution collection is easy to set up and operate. Figure 2 schematically illustrates a simple set-up that used for the hydrogen evolution rate measurements in this study. This is actually a classic set-up for detection of the negative difference effect. The aluminium specimen put in a beaker containing the test solution. A funnel placed over the specimen, which ensured the collection of all the hydrogen from the specimen surface as well as from any undermined metal particles at the bottom of the beaker. A burette was mounted over the funnel, and was initially full of the test solution. The hydrogen collected by the funnel went into the burette and gradually displaced the test solution in the burette. In this way, the volume of the evolved hydrogen easily measured by reading the position of the test solution level in the burette.

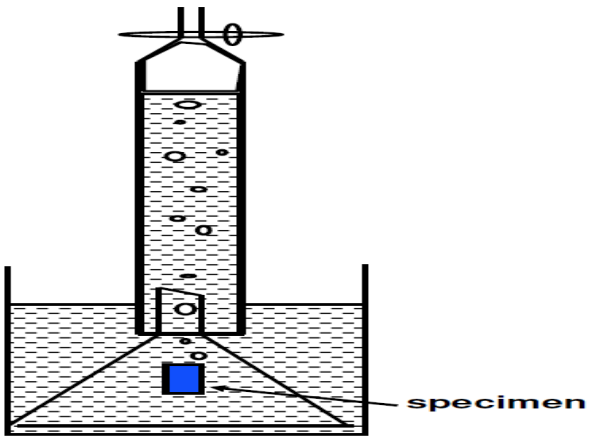


Figure 2 : Schematic illustration of the set-up for measurement of the volume of hydrogen evolved

iii. *Potentiodynamic polarization measurements*

For potentiodynamic polarization studies of aluminum, a cylindrical rod embedded in araldite with an exposed surface area (1 cm^2) used and the experiments were carried out at 25^{0}C. It was abraded with different grades of emery papers up to 1200 grit size. After that, the electrode washed with acetone, rinsed different times with distilled water and dried. The potentiodynamic measurements performed in a conventional three electrodes glass cell, which consists of aluminum as working electrode, platinum counter electrode and a saturated calomel electrode(SCE) as the reference electrode. All measurements carried out in aerated solution of 1 M HCl in the absence and presence of different concentrations of Cidamex. Potential curves were recorded by changing the electrode potential automatically from -0.8 to 0.5 V at a scan rate of 1 mVs^{-1}. The Tafel plots of the anodic and cathodic curves extrapolated to obtain the corrosion potential (E_{corr}) and corrosion current density ($i_{corr.}$).

iv. *Electrochemical impedance spectroscopy measurements*

Electrochemical impedance spectroscopy (EIS) is a powerful technique for the characterization of electrochemical systems and mechanistic information. For this reason this technique is being applied to an increasing extent to understand corrosion process in solution, to study rate determination, inhibitor performance, coating performance and passive layer characteristics [21-23]. Electrochemical impedance spectroscopy (EIS) measurements were performed at an open circuit potential 30 minutes of immersion in the test solution with amplitude of 5 mV. The cover frequency range was of 10^5 Hz to 0.1 Hz. The experiments always repeated at least three times to check reproducibility of the results. Impedance diagrams are given in Nyquist representations. The electrical equivalent circuit Figure 3 was used to fit EIS data which consists from R_s is the solution resistance, R_{ct} is the charge transfer resistance and C_{dl} is the double layer capacitance.

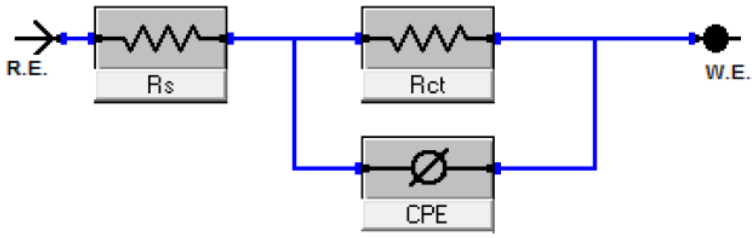


Figure 3 : Equivalent circuit proposed to fit the EIS experimental data

v. *Electrochemical frequency modulation (EFM) measurements*

The electrochemical frequency modulation has many features [24-28]. EFM is a non- destructive technique, rapid test, gives directly value of the corrosion current without a prior of knowledge of Tafel constants and has a great strength due to casually factors, which serve an internal check on the validity of the EFM measurement.

All electrochemical measurements were performed using Gamry Instrument (PCI 300/4) Potentiostat / Galvanostat /ZRA. This includes a Gamry framework system based on the ESA 400. Gamry applications include DC105 software for potentiodynamic polarization measurements, EIS300 software for EIS and EFM140 software for EFM measurements along with a computer for collecting data. Echem Analyst 6.03 software was used for plotting, graphing, and fitting data.

III. Results

a) Weight loss studies

The weight loss recorded to the nearest 0.0001g was given by equation (1):

$$\Delta w = w_1 - w_2 \quad (1)$$

Where w_1 and w_2 are the weights of metal before and after exposure to the corrosive solution, respectively.

Corrosion rates calculated using the following expression [29]:

$$\text{Corrosion rate} = \frac{\Delta W}{AT}\ \text{mg cm}^{-2}\,\text{min}^{-1} \quad (2)$$

Where Δw is the weight loss in mg, A is the area of the specimen in sq-cm and "T" is the exposure time in min.

The percentage inhibition efficiency (% IE) of different concentration of expired Cidamex calculated from the corrosion rate values by using the following equation:

$$\%\ IE = \theta \times 100 = [(W_1 - W_2)/W_1] \times 100 \quad (3)$$

where w_2 and w_1 are the weight losses (mg) for aluminum sample in the presence and absence of the inhibitor and θ is the degree of surface coverage of the inhibitor. The % inhibition efficiency (% IE) and the degree of surface coverage (θ) were tabulated .Figure 4 shows the plots of corrosion rate against different concentrations of drug. while Fig. 5 represents the effect of concentration of the drug on the % IE at different temperatures (25 and 45°C).

The experimental data of weight loss (Δw), percentage of inhibition efficiency (% IE), corrosion rate (C.R.) and degree of surface coverage (θ) for aluminum in 1 M HCl and in the presence of various concentrations of expired Cidamex at different temperatures are shown inTable1.

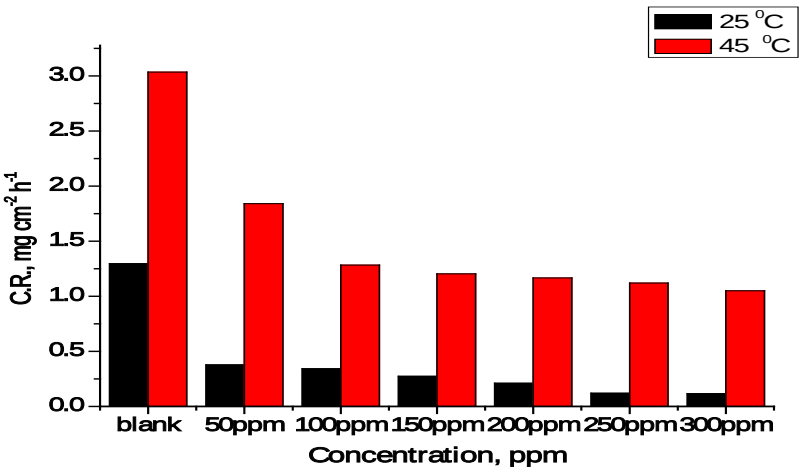


Figure 4 : Corrosion rates of various concentrations of expired Cidamex on aluminum in 1 M HCl at 25 and 45^{0}C

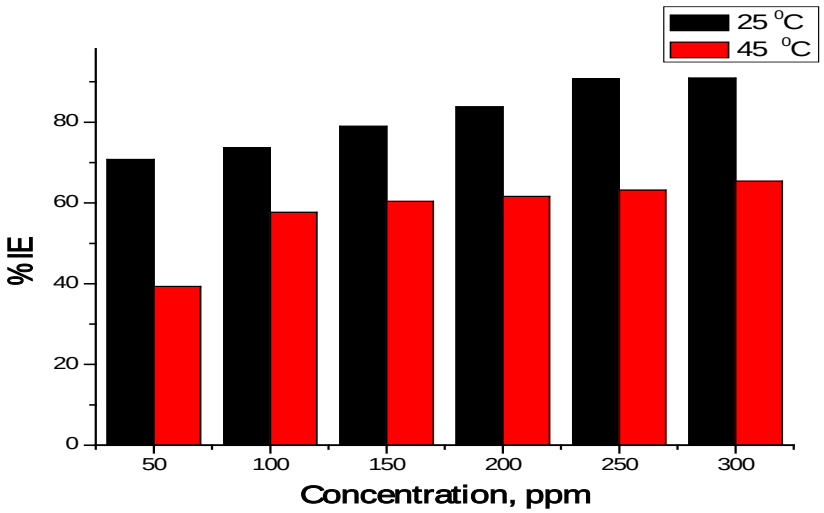


Figure 5 : The variation of inhibition efficiency with expired Cidamex concentration of aluminum in 1 M HCl solution

Table 1 : Data from weight loss of Al in 1 M HCl for various concentration of expired Cidamex after 1.5 h at 298 and 318 K

	298 K				318 K			
	ΔW mg cm^{-2}	θ	% IE	C.R., mg cm^{-2}h^{-1}	ΔW mg cm^{-2}	θ	%IE	C.R., mg cm^{-2}h^{-1}
Blank	1.950	-----	----	1.297	4.553	----	----	3.035
50	0.568	0.708	70.8	0.378	2.764	0.393	39.3	1.84
100	0.513	0.737	73.7	0.342	1.925	0.577	57.7	1.283
150	0.410	0.790	79.0	0.274	1.805	0.604	60.4	1.203
200	0.316	0.838	83.8	0.211	1.750	0.616	61.6	1.167
250	0.180	0.908	90.8	0.120	1.675	0.632	63.2	1.120
300	0.178	0.909	90.9	0.116	1.575	0.654	65.4	1.050

The results show that the inhibitor influenced on reducing the dissolution of aluminum in 1M HCl solution at all concentrations used. The inhibition efficiency increased with increasing the concentrations while at the same time the corrosion rates significantly decreased.

b) Adsorption isotherms

The adsorption of organic molecules provides information about the interaction between the adsorbed molecules themselves as well as their interaction with metal surface. When the fraction of the surface covered is determined as function of the concentration at constant temperature, adsorption isotherm evaluated at equilibrium conditions. There are a number of mathematical expressions having thus developed to take into consideration of non-ideal effects. The most used isotherms are Frumkin, De Boer, Parsons, Temkin,

Flory-Huggins and Bockris-Swinkless [30-33]. The degree of surface coverage (θ) for different concentrations of expired Cidamex in 1 M HCl was calculated from weight loss measurements Table 1 and was tested graphically for fitting a suitable adsorption isotherm. Figure 6 confirms that the inhibition processes due to adsorption of the inhibitor on the Al surface. This is because a straight line is obtained when log (C/θ) is plotted against log C and the linear correlation coefficient of the fitted data is close to unity. This indicates that the adsorption of expired Cidamex molecules obeys the Langmuir adsorption isotherm [34] which expressed as:

$$\frac{C}{\theta} = \frac{1}{K} + C \qquad (4)$$

Where C is the expired Cidamex concentration and K_{ads} is the equilibrium constant for the adsorption/ desorption process of the inhibitor molecules on the metal surface.

The relationship between the equilibrium constant, K_{ads}, of adsorption and the free energy of adsorption, ΔG^0_{ads} is given by the following expression [35].

$$K_{ads} = 1/55.5 \exp [-\Delta G^o_{ads}/RT] \qquad (5)$$

Values of free energy of adsorption calculated from equation (5) using K_{ads} values obtained from Langmuir adsorption isotherm is presented in Table 2. The values are negative and less than - 40 kJ mol^{-1}. This implies that the adsorption of the inhibitor on aluminum surface is spontaneous and confirms the physical adsorption isotherm mechanism [36].

Table 2 : Langmuir adsorption parameters for adsorption of expired Cidamex on aluminum in 1 M HCl for 1.5h immersion period at different temperatures

Temp., °C	Langmuir isotherm		
	K, M^{-1}	R^2	$-\Delta G^o_{ads}$ kJ mol^{-1}
25	32.54	0.992	18.58
45	21.13	0.995	17.51

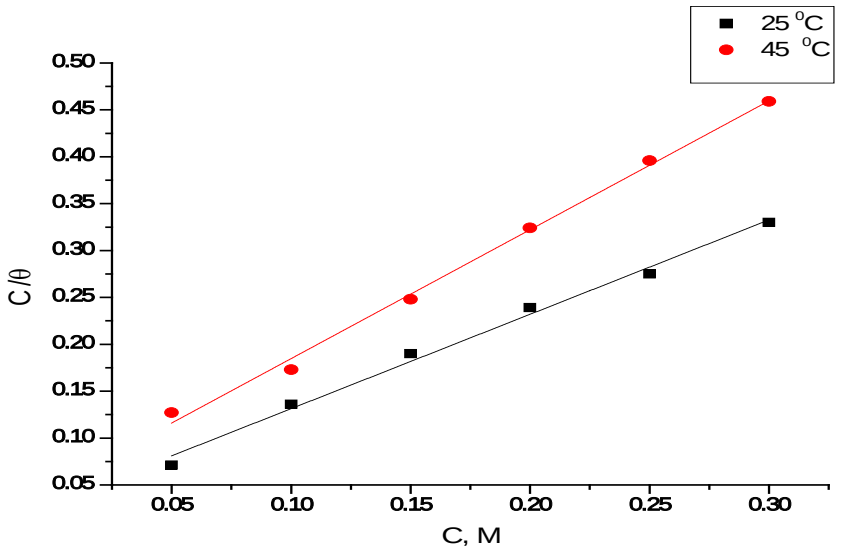


Figure 6 : Langmuir adsorption isotherm for expired Cidamex adsorption on aluminum in 1 M HCl at two different temperatures after 1.5h immersion

c) Effect of temperature

From our study, the protection efficiency decreases with an increase in temperature. This can be due to the decrease in the strength of adsorption process at higher temperature, suggesting that physical adsorption of the inhibitor on the sample surface. The apparent activation energies (E_a) for the corrosion, process in absence and presence of expired Cidamex can be evaluated from Arrhenius equation (6):

$$\text{Log (C.R.)}_2/\text{(C.R.)}_1 = E_a/2.303R\left(\frac{1}{T1} - \frac{1}{T2}\right) \qquad (6)$$

Whereas estimates of the heats of adsorption (Q_{ads}) can be obtained from the trend of surface coverage with temperature as follows [37]

$$Q_{ads} = 2.303R \left[\log\left(\frac{\theta 2}{1-\theta 2}\right) - \log\left(\frac{\theta 1}{1-\theta 1}\right)\right] x \frac{T1T2}{T2-T1} \qquad (7)$$

Table 3 : Calculated values of apparent activation energy (E^*_a) and heat of adsorption (Q_{ads}) of expired Cidamex on aluminum in 1 M HCl at different temperatures

Concentration, ppm	E_a, kJ mol^{-1}	$-Q_{ads}$, kJ mol^{-1}
1M HCl	33.50	------
50	62.36	52.03
100	52.09	28.37
150	58.29	35.57
200	67.39	46.13
250	88.00	68.90
300	86.80	65.60

Increased activation energy, E_a in inhibited solutions compared to the blank suggests that the expired Cidamex is physically adsorbed on the corroding metal surface while either unchanged or lower (E_a) in the presence of inhibitor suggest chemisorptions [38]. It seen in Table 3 that E_a values increased with inhibitor concentrations, showing that the expired Cidamex retards corrosion at ordinary temperature and its corrosion retarding efficiency considerably diminished at higher temperature [39]. The low and negative Q_{ads} values are indicative of less surface coverage with rise in temperature, supporting the earlier proposed mechanism of physisorption [40].

d) Hydrogen evolution method

The method of hydrogen evolution (via gasometric assembly) was determined as previously described elsewhere [41-43].

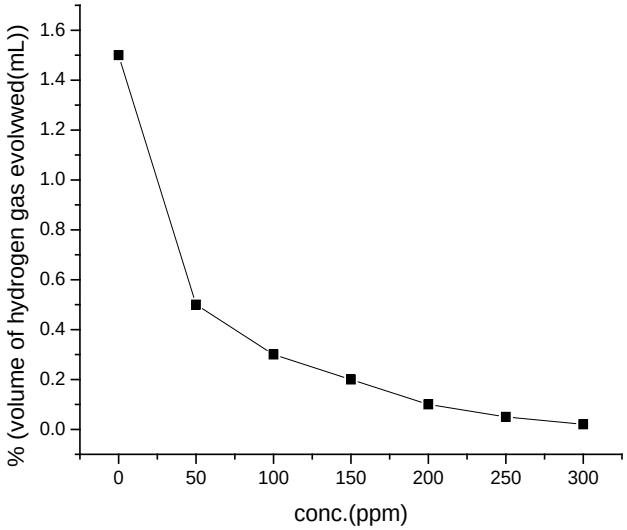


Figure 7 : Volume of hydrogen gas evolved during the corrosion of aluminum in 1M HCl in the absence and presence of different concentrations of Cidamex

Table 4 : Inhibition efficiency obtained from gasometric method for aluminum in 1M HCl at various concentrations of Cidamex

Conc., ppm	Volume of hydrogen gas evolved (ml)	% IE
blank	1.50	------
50	0.50	66.6
100	0.30	80.0
150	0.20	86.0
200	0.10	93.3
250	0.05	96.7
300	0.02	98.7

From Figure 7 and Table 4 when the concentration of expired Cidamex increases the hydrogen evolution decreases and inhibition efficiency increases, so expired Cidamex consider having an excellent ability to inhibit the corrosion of aluminum in the acid solution.

e) Potentiodynamic polarization measurements

Potentiodynamic polarization curves for aluminum in 1M HCl solution in the absence and presence of different concentration of expired Cidamex at 25 ^{0}C are shown in Figure 8 and the polarization parameters such as E_{corr}, $i_{corr;}$ anodic and cathodic Tafel slopes (β_a,β_c)are summarized in Table 5. Both anodic and cathodic Tafel slopes were slightly changed on increasing the expired Cidamex concentration. This means that there is no change of the mechanism of the inhibition in presence and absence of expired Cidamex drug and this drug affects both cathodic and anodic reactions, i.e. it is mixed-type inhibitor [44]. The E_{corr} values show that the expired Cidamex behaves as mixed type, with small variations in the E_{corr} values of the blank specimens. The degree of surface coverage (θ) and inhibition efficiency (% IE) were calculated using equation (8):

$$\% IE = \theta \times 100 = [1-(i/ i^{\circ})] \times 100 \quad (8)$$

where i° and i are the current densities in the absence and presence of the extract, respectively.

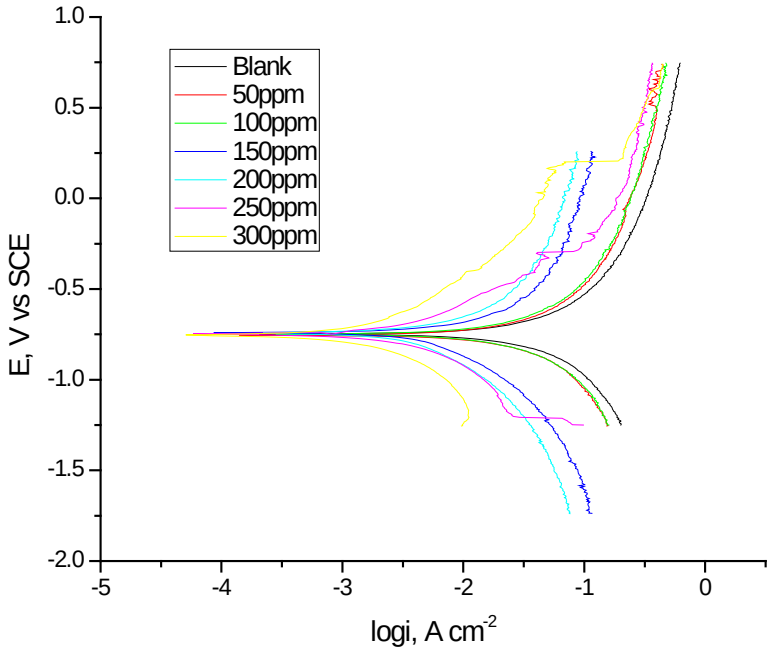


Figure 8 : Anodic and cathodic Tafel polarization curves for aluminum in the absence and presence of various concentrations of Cidamex

Table 5 : Electrochemical kinetic parameters obtained by Tafel polarization technique for aluminum in absence and presence of various concentrations of expired Cidamex

Conc., ppm	$-E_{corr}$, mV vs SCE	i_{corr}, mA cm^{-2}	β_a, V dec^{-1}	β_c, V dec^{-1}	θ	% IE
Blank	749	1590	11.6	32.4		
50	754	970	7.2	43.8	0.390	39.0
100	749	936	13.5	15.7	0.411	41.1
150	742	60.9	2.2	3.2	0.965	96.5
200	745	48.7	0.7	3.6	0.969	96.9
250	749	27.2	1.0	9.2	0.983	98.3
300	755	6.97	1.3	1.2	0.996	99.6

f) Electrochemical impedance spectroscopy (EIS) measurements

EIS was employed to investigate the effect of concentration on inhibitive behavior of inhibitor in 1 M HCl at 25^{0}C. The EIS results in form of Nyquist and Bode phase plots have been presented in Figure 9a,b. The impedance spectra consist of a large capacitive loop at high frequency followed by a small inductive one at low frequency. The values of polarization resistance and double layer capacitance were recorded in Table 6. Generally, the small inductive loop at low frequency (LF) observed for aluminum in HCl [45-48]. The diameter of Nyquist plots (R_p) increases on increasing the inhibitor concentration. These results suggest the inhibition behavior of inhibitor. The Nyquist plots analyzed in terms of the equivalent circuit composed with classic parallel capacitor and resistor Figure 2 [49]. The impedance of a CPE is described by the equation 9:

$$Z_{CPE} = Y_0^{-1} (j_\omega)^{-n} \quad (9)$$

where Y_0 is the magnitude of the constant phase element (CPE), j is an imaginary number, ω is the angular frequency at which the imaginary component of the impedance reaches its maximum values and n is the deviation parameter of the CPE: $-1 \leq n \leq 1$.

The values of the interfacial capacitance C_{dl} can be calculated from CPE parameter values Y_0 and n using equation 10 [50]:

$$C_{dl} = (2\pi f_{max} R_{ct})^{-1} \quad (10)$$

Where f_{max} is the frequency value, at which the imaginary component (Z") of impedance is maximum. The degree of surface coverage (θ) and the inhibition efficiency (% IE) were calculated from the charge transfer resistance (R_{ct}) values using the following equation (11):

$$\%IE = [(R_{ct}-R^0_{ct})/R_{ct}] \times 100 \quad (11)$$

Where R^o_{ct} and R_{ct} are the charge transfer resistance in the absence and presence of inhibitor, respectively, the results listed in Table 6.By increasing the inhibitor concentration, the R_{ct} values increase and the calculated C_{dl} values decrease, as it can be seen from Table 6, the C_{dl} values tend to decrease with the increase of the concentration of inhibitor in 1 M HCl. The decrease in the C_{dl}, which can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggests that inhibitor molecules function by adsorption at the metal/solution interface. Deviations from the ideal semi-circle are generally attributed to the frequency dispersion as well as in homogeneities, roughness of metal surface and mass transport process [51-53]. The resistances between the metal and outer Helmholtz plane (OHP) must be equal to the R_{ct}. The adsorption of expired Cidamex molecules on the metal surface decreases its electrical capacity because they displace the water molecules and other ions originally adsorbed on the metal surface. This modification results in an increase of charge-transfer resistance. The R_{ct} values increased with inhibitors concentrations may suggest the formation of a protective layer on the aluminum surface. This layer makes a barrier for mass and charge-transfer. The Bode plot, Figure 9b Shows resistive region at high frequencies and capacitive region at intermediate frequencies but do not show a clear resistive region (horizontal line and a phase angle = 0) at low frequencies. These plots show two overlapped phase maxima at intermediate and low frequencies. According to act circuit theory, an impedance plot obtained for a given electrochemical system can be correlated to one or more equivalent circuits.

Table 6 : EIS data of aluminum in 1 M HCl and in the presence of different concentration of expired Cidamex

Conc., ppm	R_{ct}, mΩ cm^{-2}	R_s	$Y^\circ \times 10^6$ $\mu\Omega^{-1}s^n$	n	$C_{dl} \times 10^4$ μF cm^{-2}	θ	% IE
Blank	607.3	2704	195.30	1.059	3.20		
50	853.5	2017	118.40	1.095	2.63	0.148	14.8
100	902.1	3138	210.90	1.085	4.13	0.194	19.4
150	1002.0	4167	240.60	1.010	2.61	0.274	27.4
200	1023.0	2690	67.29	1.123	1.93	0.290	29.0
250	2645.0	2436	85.19	1.061	1.38	0.730	73.0
300	3827.0	2783	108.90	1.014	1.21	0.810	81.0

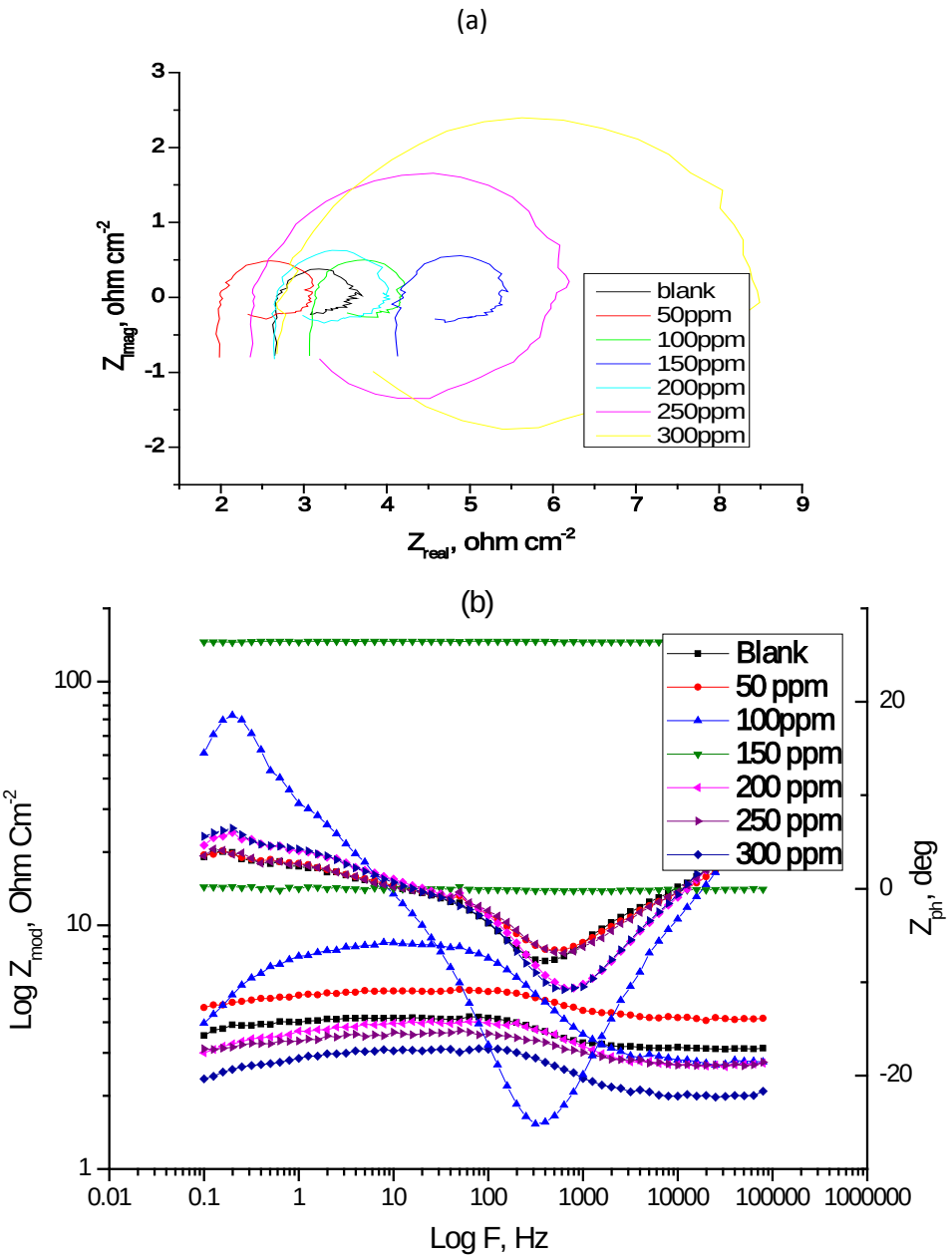


Figure 9 a, b : EIS results for aluminum in the form of Nyquist Bode plots for expired Cidamex at different concentrations and at 25°C

g) Electrochemical frequency modulation (EFM) measurements

The EFM like EIS, it is a small signal ac technique. Unlike EIS, however, two sine waves (at different frequencies) are applied to the cell simultaneously. Because current is a non- linear function of potential excitation. The current response contains not only the input frequencies, but also contains frequency components, which are the sum, difference, and multiples of the two input frequencies. The two frequencies may not choose at random. They must both be small, integer multiples of a base frequency that determines the length of the experiment. The calculated electrochemical parameters at different concentrations of drug at 25°C (i_{corr}, β_a, β_c, CF-2, CF-3 and % IE_{EFM}) are given in Table 7. Figure 10 represents the EFM intermodulation spectra (spectra of current response as a function of frequency) of aluminum in 1 M HCl devoid of and containing 300 ppm of Cidamex. The inhibition efficiency, % IE_{FM} and the degree of surface coverage (θ) of drug was calculated using equation (12):

$$\% IE = \theta \times 100 = [1- (i_{corr}/i^{\circ}_{corr})] \times 100 \qquad (12)$$

Where i°_{corr} and i_{corr} are corrosion current density in the absence and presence of black tea extract. The causality factors calculated from the frequency spectrum of the current response. If the causality factors differ significantly from the theoretical values of 2.0 and

3.0, then it can be deduced that the measurements are influenced by noise. If the causality factors are approximately the predicted values of 2.0 and 3.0, there is a causal relationship between the perturbation signal and the response signal. Then the data are assumed to be reliable [54] From the results of Table 7, it can be seen that by increasing the concentration of extract to the medium the corrosion current density (i_{corr}) decreases, indicating that the extract inhibits the 1 M HCl corrosion of carbon steel through adsorption. The calculated inhibition efficiency % IE_{EFM} enhances with increasing drug concentration.

Table 7 : Electrochemical kinetic parameters obtained by EFM technique for aluminum in 1 M HCl in the absence and presence of different concentrations of expired Cidamex

Conc., ppm	i_{corr} , µA cm^{-2}	β_a, mV dec^{-1}	β_c, mV dec^{-1}	CF-2	CF-3	θ	% IE	$R_{corr.}$ mpy^{-1}
Blank	1329.0	434	445	1.849	1.23	-----	------	792.0
50	331.5	25	79	5.281	1.42	0.750	75.1	197.2
100	215.1	19	25	1.995	2.55	0.838	83.8	128.2
150	213.4	18	26	1.887	2.12	0.839	83.9	127.1
200	211.1	19	24	1.528	2.57	0.841	84.1	125.8
250	210.6	18	25	1.509	2.27	0.842	84.2	125.5
300	208.3	19	23	1.05	3.07	0.843	84.3	124.1

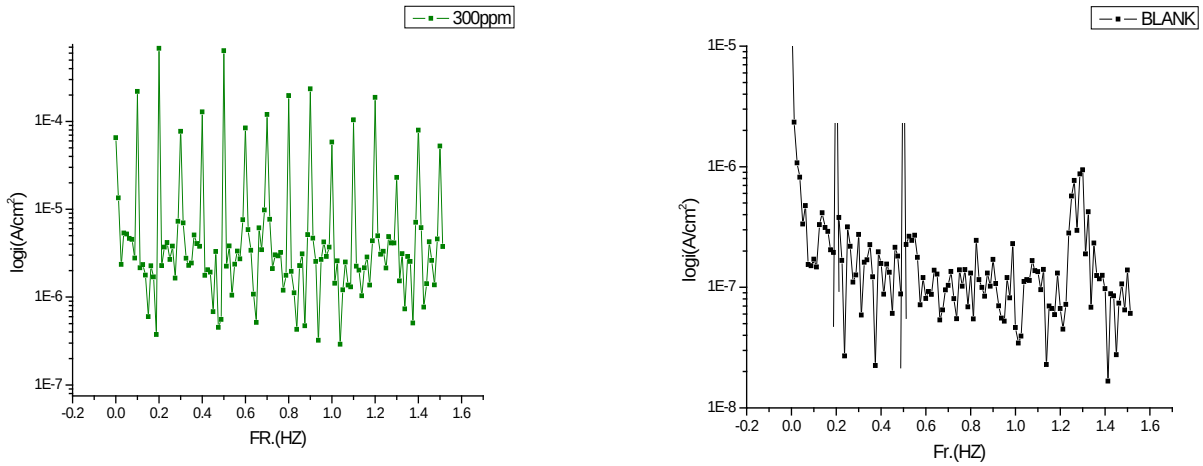


Figure 10 : Intermodulation spectra recorded for carbon steel electrode in 1M HCl solutions in the absence and presence 300 ppm of expired Cidamex

h) Mechanism of inhibition

The above observations and analysis show that the inhibitor is adsorbed on Al. Four mechanisms have been suggested for the adsorption of the inhibitor at the metal-solution interface [55]. These are: i) electrostatic attraction between charged molecules and charged metal ii) interaction of unshared electron pairs in the molecule with the metal iii) interaction of π-electrons with the metal and iv) a combination of all the above. The adsorption of investigated drug compound can be attributed to the presence of polar unit having atoms of nitrogen, sulphur and oxygen and aromatic/heterocyclic rings. Therefore, the possible reaction centers are unshared electron pair of hetero-atoms and π-electrons of aromatic ring [56]. As discussed above the thermodynamic and kinetic parameters, the adsorption is mainly electrostatic. Physical adsorption requires presence of both electrically charged surface of the metal and charged species in the bulk of the solution. In the acid solution, the drug could be protonated due to the interaction between O atom and H^+. The value of pH_{zch}, which is defined as the threshold pH at a point of zero charge, is equal to 9.1 for aluminum [57]. So aluminum surface is positively charge due to accumulation of Al–OH_2^+species in acidic solution [58]. The acid anions (Cl^-) adsorb electrostatically on the positively charged, giving rise in for a net negative charge on the metal surface; and the organic cations are physically attracted to the anions layer which is formed on the metal surface, forming electrostatic protective layer on aluminum.

IV. Conclusions

The results obtained show that expired Cidamex drug is a good corrosion inhibitor for aluminum under acidic conditions. The adsorption of expired Cidamex drug on aluminum surface obeys Langmuir adsorption isotherm. The adsorption process is spontaneous. The adsorption of the expired Cidamex drug onto the aluminum characterized by the decrease in:
(a) Weight loss of aluminum (b) hydrogen evolution (c) the cathodic and anodic current densities observed in the potentiodynamic polarization curves carried out in the presence of drug, (d) the double-layer capacitance computed from electrochemical impedance spectroscopy experiments and (e) the current obtained from electrochemical frequency modulation (EFM). The negative values of ΔG^0_{ads} show the spontaneity of the adsorption. Inhibition efficiency increases with increase in expired Cidamex concentration and decreases with rise in temperature. From this study, it was concluded that these unused drugs can be used as save corrosion inhibitors for water cooling systems, oil pipelines, degreasing solutions, deicing solutions for aircrafts, paints and coatings, inhibitors for concrete fuels and lubricants, metal processing solutions [59]

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Transformations of Vegetables Oils in Supercritical Methyl and Ethyl Acetates in Continuous Flow Reactor

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Keywords : *biofuel, biodiesel, vegetable oil, supercritical methyl and ethyl acetates, tubular reactor, reaction.*

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V.I. Anikeev [α], V.P.Sivcev [σ] & E.Yu. Yakovleva [ρ]

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I. Introduction

The reactions of plant raw materials and food industry wastes in supercritical solvents (lower alcohols, in particular) show some promises for the full-scale production of cheap biodiesel fuel and chemicals [1-14]. First, these reactions proceed in the absence of homogeneous catalysts and therefore allow the use of a lower-grade feedstock. Second, they provide a 90-98% conversion of initial feedstock during short residence times (several tens of minutes) and thus make possible to use the reactors of flow type that considerably enhances the process efficiency. Third, these processes are free of huge amounts of waste water because there is no need to wash up the products from homogeneous alkaline or acid catalysts. Forth, all these factors enable a significant reduction in the cost of biodiesel fuel.

The use of heterogeneous catalysts in the reactions of vegetable oils with alcohols allows [15 - to partially get rid of the homogeneous catalysts drawbacks: elimination the problem corrosion of reactor material, does not require the separation of products and acid, and makes it possible to perform the process in the continuous mode. At the same time, some poisoning of the heterogeneous catalyst is to be expected, along with a reduction in its activity and the emergence of pore diffusion resistance, resulting in a low rate of reaction.

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The surface of the heterogeneous catalyst used in the transesterification of triglycerides must have hydrophobic properties in order to limit glycerol and water adsorption on the active centers of the catalyst, as such adsorption results in a loss of its activity. The properties and nature of heterogeneous catalyst being used determine to a large extent the conditions for conducting the reaction and the method of products separation. Heterogeneous catalysts with acid [19, 20] or basic [21, 22] properties are used in triglyceride transesterification reactions.

The presence of hydroxyl group in alcohol molecule leads to the formation of glycerol at transesterification of triglycerides, which are the main constituents of all vegetable oils. The reactions of fuel synthesis from vegetable oils in methanol, including the supercritical one, yield byproduct glycerol in an equimolar amount to the amount of converted oil. Besides the need to separate glycerol from the reaction products, the problem appears how to utilize it, for example, in the synthesis of useful chemicals.

The use of the acylated alcohol instead of the lower alcohol for transesterification of triglycerides may also lead to the formation of fatty acid esters (biodiesel), but instead of glycerol another product will be formed as a byproduct. For example, the use of methyl- or ethyl acetate may lead to the formation of 1,2,3-triacetoxy-propane known also as triacetin.

Triacetin is itself a valuable compound that can be used in the cosmetic and food industry or as an additive to petrol fuel [18,23]. It is also a good solvent, can be easily mixed with fatty acid esters and used as a fuel additive improving low-temperature stability and viscosity of diesel fuel (triacetin melting point is -78°C).

Methyl acetate (acetic acid methyl ester), being an acyl group donor, has been already used instead of methanol in the enzyme-catalyzed reactions of vegetable oil transesterification (see Scheme 1) [24-26], since methanol inhibits enzyme activity. Although enzyme-catalyzed synthesis of biofuels is proved feasible, it still has serious disadvantages restricting its wide application, such as enzyme susceptibility to the oil type and quality, large residence times to provide sufficient conversion, low process efficiency. Trying to overcome these shortcomings, it was suggested to

perform transesterification of vegetable oils in supercritical methyl and ethyl acetates in a flow reactor.

The use of methyl acetate instead of methanol for supercritical synthesis of glycerol-free biodiesel from vegetable oils is a new process and its study is very limited in the literature. There are several studies in this field, e.g. [27-32]. The authors of [27] tested some oils with different fatty acid composition. The process was also applied to waste oil with higher free fatty acid (FFA) content. The results demonstrate that the oil composition does not significantly influence the biodiesel yield. These authors studied the influence of temperature, pressure and molar ratio of reactants by oils transesterification with supercritical methyl acetate. It has been shown that all oils achieved complete conversion after 50 min at 345^{0}C and 20 MPa with methyl acetate:oil molar ratio equal to 42:1.

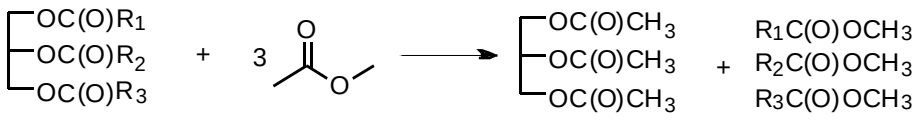
Kinetic of transes terification reactions of four oils with supercritical methyl acetate was studied in [27] in mixed batch reactor. Pseudo-first order equations used for modeling.

Conversion of rapeseed oil and oleic acid with supercritical methyl acetate to free acid methyl esters (FAME) and triacetin (TA) in flow tubular reactor investigated in [28] without catalyst. The results of these studies are shown that the transesterification reaction of triglycerides with methyl acetate can proceed under supercritical conditions, generating FAME and triacetin.

In this work, methyl acetate (T_{cr} = 233.7°C, P_{cr} = 4.63 MPa) and ethyl acetate (T_{cr} = 250.4°C, Pcr = 3.78 MPa) were used in experiments for transformation of vegetables oils in continuous flow reactor.

Fatty acid methyl esters have been successfully produced from noncatalytic transesterification reaction between triglycerides (palm oil) and methyl acetate in batch-type reactor (12ml) [29]. The optimum conditions were found to be 399^{0}C for reaction temperature and residense time of 59 min to achieve 97.6% biodiesel yield.

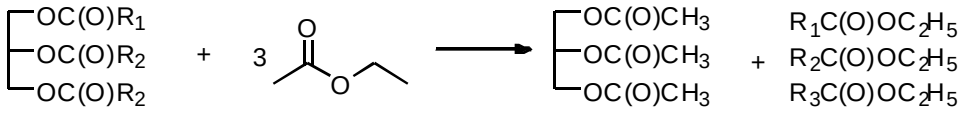
The reactions of triglycerides transesterification by methyl and ethyl acetates are assumed to proceed according to Scheme 1 and Scheme 2.



1 2 3 4

1- triglyceride, 2 – methyl acetate, 3 – triacetin, 4 – fatty acid esters

Scheme 1



1 2 3 4

1- triglyceride, 2 – ethyl acetate, 3 – triacetin, 4 – fatty acid esters

Scheme 2

II. Experimental

Transesterification of vegetable oils with supercritical methyl and ethyl acetates was performed in stainless steel tubular flow reactor of volume ~ 23.5 cm^3, inside diameter of reactor tube ~ 0.3 cm, its length ~ 3.3 m. The experimental setup is described in details in our earlier works [33,34]. The acetate and vegetable oil were fed to a mixer at the reactor inlet as two independent flows. The first flow was pure methyl or ethyl acetate; it was fed to the mixer by a piston pump through a heat exchanger, where it was heated to the reaction temperature. The second flow was sunflower or corn oil; it was fed directly to the mixer by a syringe pump. The "acetate/oil" parameter was calculated as a ratio of volume flow rates (cm^3/min) of acetate and vegetable oil supplied to the reactor.

The cooled product mixture was a homogeneous non-segregating liquid, which was sampled for analysis under fixed stationary experimental conditions.

The present studies were performed using refined edible sunflower and corn oils and an ACROS ORGANICS methyl acetate (>99.8 wt.%), ethyl acetate (>99.5 wt.%).

The residence time of the reaction mixture was calculated as a ratio of the inlet acetate-oil mixture flow rate Q (cm^3/min) to the reactor volume (23.5 cm^3). For example, if Q = 8 cm^3/min, the residence time was ~2.9 min. The reaction was performed in the temperature range of 213-400°C at pressure ~ 200-220 atm. The temperature and pressure providing supercritical state of the reaction mixture were selected on the base of thermodynamic calculations and phase diagram plotting

[35]. Selected reaction conditions provide a single-phase state of the reaction mixture.

Three systems: corn oil/methyl acetate, corn oil/ethyl acetate, sunflower oil/methyl acetate have been chosen for the study at the same parameters.

a) Methods of analysis

Transformations of vegetable oils with acetates are performed usually at low temperatures and in the presence of catalysts, mainly, enzymes. Under these conditions, triacetin and fatty acid methyl or ethyl esters are the main reaction products. Increased temperature and pressure will most likely facilitate the formation of other products. For this reason, in this work we focused special attention on the methods of qualitative and quantitative analysis of the reaction products.

Liquid products were analyzed by a chromato-mass-spectrometer Agilent Technologies 7000 GC/MS Triple Quad, GC System 7890A, using a Zb-Wax column of length 30 m, i.d. 0.25 mm, film thickness 0.25 μm; measurement range m/z: 40 – 500. Heating protocol: 2 min at 50°C, 8°C/min to 260°C, 30 min at 260°C. Carrier gas He, ion source temperature 230°C, flow split ratio 1:20, vaporizer temperature 300°C. The products were identified by comparing the retention times and mass-spectra with the reference libraries NIST and Wiley7. Quantitative analysis of the fatty acids and respective esters was performed using an internal standard (1-hexanol) calibration method.

The content of free fatty acids in the initial sunflower oil was determined by two methods – chromate-mass-spectrometry and titration. According to chromate-mass-spectrometric data, the initial oil contained free fatty acids in the amount of ~2.75 vol.%. Note that the quantitative analysis of fatty acids was complicated by their partial adsorption on the analyzer surfaces. To improve measurement validity, after each analysis a certain amount of pure solvent was injected into the analyzer for washing out the residual fatty acids. Then the amounts of the fatty acid detected in the washout and in the sample were summated that provided correct data on the fatty acid content in the initial oil.

Titration of heated oil-isopropanol mixture with added indicator (phenolphthalein) was performed by a solution of 0.1 g NaOH in 100 g of water (0.1% NaOH aqueous solution) till the mixture turned pale pink. Then the titration was stopped and the consumed amount of the titrant was measured. According to titration analysis, initial oil contained ~2.7% of free fatty acids.

Based on the results of chromate-mass-spectrometric and titration analyses, the total content of fatty acids in the sunflower oil was assumed to be ~ 2.7 vol.%. Among the indentified acids, linoleic acid showed the highest content.

III. Results and Discussions

Before starting experiments on the oils transesterification in supercritical methyl and ethyl acetates, thermal stability of the latter has been studied. To study thermal stability of methyl and ethyl acetates, we used the above setup without oil feeding to the reactor. Thus, methyl acetate showed sufficient thermal stability in the flow reactor at temperatures 200-340°C, pressure 200 atm and residence time ~2.9 min. At temperatures above 350°C, weak gas emission was observed at the reactor outlet, and acetic acid was detected in the liquid-phase products.

It was found that supercritical ethyl acetate in a flow reactor at temperatures 250-340°C, pressure 200 atm and residence time ~4.7 min remained stable. As the temperatures exceeded 340°C, the release of gaseous products was observed; the liquid phase contained also the acetic acid. The gaseous products included hydrogen, CO, methane, CO_2, ethane and ethylene. With the temperature increase from 360 to 450°C, the outlet concentration of acetic acid increased by more than 6 times. The yield of gaseous products increased similarly. No ethanol traces were found in the liquid phase.

Since the initial reaction mixtures contained no water, and no methanol and ethanol was detected in the reaction products, it seems hardly possible that the acetic acid appeared by the reaction of methyl or ethyl acetate hydrolysis (a reverse reactions to the synthesis of methyl acetate from acetic acid and methanol, and ethyl acetate from acetic acid and ethanol). It should be noted that methanol or ethanol formation cannot be excluded entirely – they may be formed and then rapidly consumed in the reaction of triglycerides transesterification. Thus, transformation of methyl or ethyl acetates under the reaction conditions proceeds by thermal decomposition to produce acetic acid and gaseous products. Since acetic acid appears in the reaction mixture in trace amounts, it catalytic effect on the oil transesterification reaction is insignificant.

a) Sunflower transesterification in supercritical methyl acetate

As shown in our earlier studies of vegetable oil (including sunflower one) transesterification with supercritical methanol [33,34], fatty acid esters and glycerol were the main reaction products.

Table 1 presents the product distribution in the reactions of sunflower transesterification in supercritical methyl acetate at various temperatures. Obviously, the qualitative and quantitative product compositions in this reaction are strongly different from those in the reaction of sunflower oil transesterification in supercritical methanol [33-35] under similar reaction conditions.

As the temperature increased from 380 to 400°C, the yield of glyceryl linolate and glyceryl oleate

increased approximately threefold. Previously [33-35], we found a weak effect of pressure on the of oils transesterification reaction in supercritical alcohols. We explain this effect in the absence of significant changes in the solvent properties under pressure changes. For this reason, the effect of the pressure on the reaction rate of oils with acetates has not been studied.

Table 1: Product distributions in the reaction of sunflower transesterification in supercritical methyl acetate at various temperatures

Sample	0	1	2	3	4	5	6	7
T, °C	27	213	271	301	334	347	380	400
Methyl acetate	0.967	0.924	0.924	0.904	0.838	0.743	0.556	0.426
1-Hexanol	0.017	0.022	0.018	0.016	0.018	0.020	0.011	0.010
Acetic acid			0.0008	0.0014	0.0006	0.0007	0.002	0.003
Methyl palmitate					0.0003	0.0005	0.003	0.005
M_w=318					0.0002	0.0004	0.004	0.007
Methyl stearate					0.0002	0.0005	0.002	0.005
Methyl oleate					0.0004	0.0010	0.006	0.011
Methyl linoleate			0.001	0.003	0.023	0.042	0.172	0.262
Sum of esters			0.001	0.003	0.0241	0.0444	0.186	0.290
Triacetin					*Traces*	*Higher than 4*	*Higher than 5*	*Higher than 6*
Palmitic acid	0.005	0.008	0.009	0.015	0.021	0.036	0.042	0.047
Stearic acid			0.004	0.003	0.007	0.015	0.015	0.018
Oleic acid	0.003	0.011	0.008	0.013	0.019	0.030	0.048	0.049
Linoleic acid	0.007	0.036	0.035	0.044	0.073	0.112	0.140	0.157
Sum of acids	0.015	0.054	0.056	0.075	0.119	0.192	0.245	0.271
Triglycerides	0.361	0.360	0.365	0.359	0.308	0.297	0.178	-0.048**
Conversion, %	1.89	2.11	0.68	2.30	16.13	19.16	51.61	~100.0

***Includes experimental error*

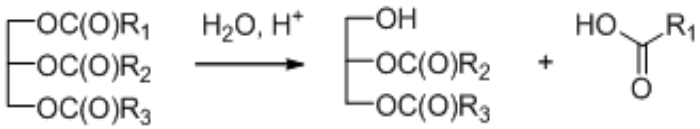
In Table 1, the product content (besides triglycerides) is presented as a volume fraction of the product with regard to total volume of the analyzed compounds. The content of non-converted triglycerides is given as a volume fraction with respect to the fixed volume (V = 0.5 μm) of the sample injected into the chromato-mass-spectrometer. The volume of non-converted triglycerides $V_{prod.\ trigl}$ was calculated on the base of total volume balance of the reaction products:

$$V_{prod.\ trigl.} = V - V_{MA} - V_{hex.} - V_{FAME} - V_{FFA} - V_{acet.acid}, \quad (1)$$

where V_{MA} – volume of methyl acetate, V_{hex} - volume of 1-hexanol, $V_{acet.acid}$ – volume of acetic acid, V_{FAME} - volume of fatty acid methyl esters, V_{FFA} - volume of free fatty acids

All components of equation (1) were calculated on the base of chromato-mass-spectrometric data. The results obtained showed that the content of fatty acids and respective esters in the reaction products increased with increasing reaction temperature. The most significant increase was observed for oleic and linoleic acids, and respective esters.

Formation of fatty acids and their increasing content in the reaction products with the increasing temperature would be explained by hydrolysis of triglycerides according to Scheme 3, if the reaction mixture contained large amounts of incompletely transesterificated triglycerides and a source of protons.



Scheme 3

However, in our experiments incompletely transesterificated triglycerides, such as glyceryl linolate and glyceryl oleate, appeared in small and almost equal amounts only at high temperatures. Moreover, although the content of all fatty acid esters significantly increased with increasing temperature, small amount of triacetin was detected only at 347°C; at higher temperatures it increased almost twice.

The fractions of methyl oleate and methyl linolate increased most strongly with increasing temperature (Table 1).

The oil conversion was calculated according to equation:

$$\text{Conversion} = (1 - \alpha_{prod\ trigl}) \cdot 100\%,$$

where $\alpha_{prod\ trigl}$ – volume fraction of triglycerides in the reaction products, calculated by eq. (2):

$$\alpha_{prodtrigl} = V_{prod\ trigl} / V_{0\ trigl} \quad (2)$$

where $V_{0\ trigl}$ – volume of triglycerides in the initial oil; $V_{prod\ trigl}$ – volume of triglycerides in the reaction products, calculated by eq. (1).

Precise determination of the free fatty acid content in the initial oil makes possible to calculate the content of triglycerides ($V_{0\ trigl}$).

As the residence time was increased from 2.9 to 5.9 min, the oil conversion and the yield of the target products (methyl esters of fatty acids) increased considerably (Table 2). It is clearly seen that the temperature increase of 20°C at this residence time caused a three-fold increase in the esters yield. This result seems very important, because slight elongation of the tubular reactor makes feasible to reach complete oil conversion at a lower reaction temperature.

Table 2 : The yield of fatty acid methyl esters at sunflower oil transesterification in supercritical methyl acetate at residence time 5.9 min and various temperatures

Sample	1	2
T, °C	330	350
	Volume fraction*)	
Palmitic acid methyl ester	0.08	0.18
Stearic acid methyl ester	0.03	0.08
Oleic acid methyl ester	0.07	0.17
Linoleic acid methyl ester	6.96	20.25

*) Volume fraction was calculated as a ratio of the ester volume to the fixed volume (5 μm) of the sample injected to the analyzer

b) Transesterification of sunflower and corn oils by ethyl acetate

i. *Reaction of sunflower oil with ethyl acetate*

Table 3 presents the product distribution (after deduction of acetic acid and ethyl acetate) for the reaction of sunflower oil transesterification by supercritical ethyl acetate at various temperatures. Note, the qualitative and quantitative product compositions in this reaction vary strongly from those in the reaction with supercritical methanol [33,34], other conditions being the same. The main differences are low content of fatty acid esters, high content of free fatty acids and incompletely substituted products.

Table 3 : Product distribution in the reaction of sunflower oil transesterification with ethyl acetate

Temperature , °C / Product ,vol. %	260	300	340	360	400	425
glyceryl 1,2-diacetate				0.61	1.03	5.02
palmitic acid ethyl ester				1.48	3.88	6.35
stearic acid ethyl ester				0.95	2.64	3.04
oleic acid ethyl ester			0.89	2.8	11.14	12.22
linoleic acid ethyl ester			1.96	4.2	13.83	11.18
Sum of esters			2.85	9.43	31.49	32.79
palmitic acid	2.7	2.6	2.64	2.77	3.85	3.3
stearic acid	1.1	1.1	1.08	1.57	2.22	2.3
oleic acid	6.0	6.6	6.85	7.83	10.86	9.71
linoleic acid	7.8	13.1	15.06	13.63	12.75	11.55
Sum of acids	17.6	23.4	25.63	25.8	29.68	26.86
glyceryl palmitate, 2,3-diacetate				2.14	0.98	4.3
glyceryl oleate, 2,3-diacetate				1.59	3.45	2.92
glyceryl linoleate				0.67	8.66	4.39

It is seen that even at low temperatures the reaction products contain fatty acids that proves the presence of initial oil. Analysis of initial oil supports this suggestion. However, the content of fatty acids increases slightly with increasing temperature, i.e. they form during the reaction. It is reasonable to suggest that the free fatty acids are formed during acid-catalyzed hydrolysis of triglycerides.

ii. *Reaction of corn oil transesterification with ethyl acetate*

The studies showed no significant differences in the reactions of sunflower and corn oils transesterification in *sc* ethyl acetate (Table 4). In both cases, formation of free oleic and linoleic acids was observed with increasing temperature; no traces of triacetin–the product of complete grycerin transesterification – were detected.

Table 4 : Product distribution in the reaction of corn oil transesterification with ethyl acetate

Temperature, °C / *Product ,vol. %*	360	400	425
glyceryl 1,2-diacetate	1.5	1.3	4.3
palmitic acid ethyl ester	6.0	6.7	7.7
oleic acid ethyl ester	11.6	12.6	19.2
linoleic acid ethyl ester	12.2	14.0	15.0
Sum of esters	29.7	33.33	41.84
palmitic acid	8.7	8.6	6.2
stearic acid	1.8	1.9	1.4
oleic acid	5.6	12.1	10.7
linoleic acid	10.9	11.9	14.0
Sum of acids	26.9	34.58	32.39
glyceryl palmitate, 2,3-diacetate	1.2	5.0	4.1
glyceryl oleate, 2,3-diacetate	2.0	1.4	1.4
glyceryl linoleate	5.5	1.9	3.4

IV. Conclusions

Although transesterification of vegetable oils with supercritical methyl and ethyl acetates has some advantages over this process with lower alcohols (methanol, ethanol), the obtained products are still below the biofuel quality standards at the selected parameters of reaction. The reaction of oils with supercritical methyl acetate yields fatty acid esters and free fatty acids. The fraction of the latter in the reaction products attains up to 50% at high temperatures.

Product distribution at transesterification of sunflower and corn oils in supercritical ethyl acetate was quite different – it showed small content of fatty acid esters, and high content of free fatty acids and partially (incompletely) substituted triglycerides.

Nevertheless, the obtained data on vegetable oils conversion in supercritical methyl and ethyl acetates at the same fixed parameters in a flow reactor at short residence times are the starting point for the optimization of the transformation conditions providing the desired product composition.

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Combustion Calorimetry and Thermodynamic Functions of Cyanocobalamin

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Abstract- In a calorimeter with a static bomb and an isothermal shield, the energy of combustion of the cyanocobalamin has been measured at 298.15 K. Physico-chemical methods established the products of combustion of cyanocobalamin in the conditions of calorimetric experiment. The enthalpy of combustion $\Delta_c H°$ and the thermodynamic parameters $\Delta_f H°$, $\Delta_f G°$ of the cyanocobalamin at T = 298.15 K and p = 0.1 MPa have been calculated. Thermodynamic parameters $\Delta_f H°$, $\Delta_f S°$ were determined and used to calculate the enthalpy of formation of cyanocobalamin.

Keywords: vitamin B_{12}; cyanocobalamin; combustion calorimetry; thermodynamic functions.

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I. Introduction

Cyanocobalamin, also called vitamin B_{12} (PubChem CID: 5479203), is a water-soluble vitamin with a key role in the normal functioning of the brain and nervous system, and for the formation of blood. Vitamin B_{12} is a cobalt-containing compound synthesized by bacteria and an essential nutrient in mammals, which take it up from diet [1]. The significance of vitamin B_{12} adequate nutritional status throughout life span is established and the adverse effects of vitamin B_{12} deficiency in human health are currently recognized [2-4]. In addition to the well-described reversible hematological and often irreversible neurological changes of severe vitamin B_{12} deficiency, epidemiological studies revealed a more common condition, the low vitamin B_{12} status particularly in elder and pregnant women [5-6]. Because vitamin B_{12} is essential for DNA synthesis and cellular energy production, a low vitamin B_{12} status may be a risk factor for altered cellular metabolism and age-related diseases including cognitive decline and cardio-vascular disease [7].

This work is a continuation of systematic studies of vitamins B. Earlier in the articles [8-10], we have investigated the thermodynamic properties of vitamins B_2, B_3 and the temperature dependence of the heat capacity of cyanocobalamin. The goals of this work include calorimetric determination of the standard thermodynamic functions of the cyanocobalamin.

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II. Experimental

i. *Sample*

Cyanocobalamin was purchased from Fluka. For phase identification, an X-ray diffraction pattern of the vitamin B_{12} sample was recorded on a Shimadzu X-ray diffractometer XRD-6000 (CuK_α radiation, geometry θ-2θ) in the 2θ range from 5° to 60° with scan increment of 0.02°. The X-ray data and estimated impurity content (0.1 wt %) in the substance led us to conclude that the cyanocobalamin sample studied was an individual crystalline compound. Cyanocobalamin can crystallize in three modifications [4]: 1) "as-purchased"; 2). "wet"; 3) "dry". According to X-ray diffraction and solid-state NMR spectroscopy, we investigated the "as-purchased" sample cyanocobalamin. This sample is usually obtained by rapid crystallization from water at 343 K.

ii. *Apparatus and measurement procedure*

The energy of combustion, $\Delta_c U$, of cyanocobalamin was measured in a calorimeter (V-08) with a static bomb and an isothermal shield. The calorimeter design, the procedure of measuring the energies of combustion and the results of calibration and testing are given elsewhere [11]. It should be noted that while checking the calorimeter by burning succinic acid, prepared at D.I. Mendeleev Research Institute of Metrology (the value of the standard enthalpy of combustion of the acid coincided with the certificate value within ±0.017%). For complete combustion of cyanocobalamin we used paraffin as an auxiliary substance.

Physico-chemical methods established the products of combustion of cyanocobalamin in the conditions of calorimetric experiment. Firstly, the solid products of combustion were identified by X-ray diffraction (Shimadzu X-ray diffractometer XRD-6000). Secondly, the formed liquid droplets were analyzed for phosphorus content using atomic absorption spectrophotometry (Shimadzu atomic absorption spectrophotometer AA-6300). Thirdly, the liquid droplets were titrated for total inorganic acids (Mettler Toledo pH meter Five Easy FE-20). Fourthly, the analysis of the gas phase was carried out by gas chromatography (Shimadzu GC 2010 Plus).

III. Results and Discussion

The experimental data on burning of cyanocobalamin are presented in Table 1. As a result, the energies and enthalpies of combustion of riboflavin at T = 298.15K and standard pressure were determined. The values are for the reaction:

$$C_{63}H_{88}CoN_{14}O_{14}P(cr)+79.75\cdot O_2(g)\rightarrow 63\cdot CO_2(g)+42.875\cdot H_2O(l)+0.75\cdot CoO(cr)+$$
$$+0.125\cdot Co_2P_2O_7(cr)+0.75\cdot H_3PO_4(sl\text{-}n,700H_2O)+7\cdot N_2(g)$$

In brackets are given the physical states of reagents: (cr), crystalline; (g), gaseous; (l), liquid; (sl-n), solution. It should be noted that we have used a significant amount of physico-chemical methods (see section 2.2) in the study of combustion products of cyanocobalamin which is an organometallic compound. The data on the enthalpy of combustion of the crystalline cyanocobalamin was used to estimate enthalpy of combustion and formation at T = 298.15K and p = 0.1MPa (Table 2). Due to the fact that the standard enthalpy of formation of dicobalt diphosphate absent in the literature, we calculated the standard enthalpy and entropy of formation of $Co_2P_2O_7$ at 298.15 K (Table 2). In works [12, 13], the absolute entropy and the standard Gibbs function of formation of dicobalt diphosphate were determined.

The Gibbs function of formation $\Delta_fG°$ of the cyanocobalamin was evaluated from the $\Delta_fH°$ and $\Delta_fS°$ [10] values (Table 2). The values conform to the following process:

$$63\cdot C(gr)+44\cdot H_2(g)+7\cdot N_2(g)+7\cdot O_2(g)+Co(cr)+P(cr)\rightarrow C_{63}H_{88}CoN_{14}O_{14}P(cr)$$

where in the brackets are indicated the physical states of reagents: (gr), graphite; (g), gaseous; (cr), crystalline.

IV. Conclusions

The general aim of these investigations was to report the results of the thermodynamic study of the cyanocobalamin. The standard enthalpy of formation is determined by using combustion calorimetry. Much of the work is devoted to the study of the mechanism of combustion of cyanocobalamin and determination of thermodynamic functions of the combustion products.

V. Acknowledgements

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Table 1 : Experimental data on combustion energy for cyanocobalamin at $T = 298.15$K

Value	Experiment					
	1	2	3	4	5	6
m_{sam} (g) [a]	0.15345	0.1600	0.1466	0.1580	0.1595	0.1607
m_{par} (g) [a]	0.6920	0.6989	0.7024	0.7021	0.7050	0.7059
m_{thread} (g) [a]	0.0025	0.0022	0.00235	0.00215	0.0021	0.0019
W ($J \cdot K^{-1}$) [b]	14805	14805	14805	14805	14805	14805
Δt (K) [c]	2.443105	2.475455	2.463935	2.483210	2.493200	2.497915
$-\Delta_c U_{\Sigma}$ (J) [d]	36170.4	36649.1	36478.6	36763.9	36911.8	36981.6
$-\Delta_c U_{par}$ (J) [e]	32347.1	32668.2	32831.8	32818.7	32954.3	32997.3
$-\Delta_c U_{thread}$ (J) [e]	42.0	36.5	39.3	36.0	35.6	32.1
$-\Delta_c U_{HNO3}$ (J) [f]	5.9	10.5	8.2	8.8	9.4	11.7
$-\Delta_c U_C$ (J) [g]	9.8	16.4	16.4	-	26.2	26.2
$-\Delta_c U$ ($J \cdot g^{-1}$) [h]	24666.0	24689.4	24663.7	24686.1	24694.0	24683.9

$-\Delta_c \overline{U} = 24681 \pm 10$ $J \cdot g^{-1} = 33452 \pm 14$ $kJ \cdot mol^{-1}$, the mean energy of combustion of cyanocobalamin, $-\Delta_c U^\circ = 33435 \pm 14$ $kJ \cdot mol^{-1}$, the energy of combustion of cyanocobalamin at standard pressure.

[a] m_{sam}, m_{par}, m_{thread}, masses of the tested sample, paraffin and a cotton thread, respectively.

[b] W, the energy equivalent of the calorimeter.

[c] Δt, the temperature increase in the experience, adjusted for heat transfer.

[d] $\Delta_c U_{\Sigma}$, the total energy released during the experiment.

[e] $\Delta_c U_{par}$, $\Delta_c U_{thread}$, amounts of energy released on burning paraffin and cotton thread, respectively.

[f] $\Delta_c U_{HNO3}$, the energy of formation of nitric acid.

[g] $\Delta_c U_C$, the energy incomplete combustion of carbon.

[h] $\Delta_c U$, the energy released on burning of cyanocobalamin.

Table 2 : Enthalpy of combustion and thermodynamic characteristics of formation of cyanocobalamin and dicobalt diphosphate ($T = 298.15$ K. $p = 0.1$MPa)

Compound	$-\Delta_c H^\circ$ ($kJ \cdot mol^{-1}$)	$-\Delta_f H^\circ$ ($kJ \cdot mol^{-1}$)	$-\Delta_f S^\circ$ ($J \cdot K^{-1} \cdot mol^{-1}$)	$-\Delta_f G^\circ$ ($kJ \cdot mol^{-1}$)
$C_{63}H_{88}CoN_{14}O_{14}P$	33459 ± 14	5017 ± 15	7281 ± 5 [10]	2846 ± 15
$Co_2P_2O_7$	–	2273 ± 7	654.3 ± 1.2	2078 ± 7 [13]

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The Board can also play vital role by exploring and giving valuable suggestions regarding the Standards of "Open Association of Research Society, U.S.A (OARS)" so that proper amendment can take place for the benefit of entire research community. We shall provide details of particular standard only on receipt of request from the Board.

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Note :

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- In future, if the board feels the necessity to change any board member, the same can be done with the consent of the chairperson along with anyone board member without our approval.
- In case, the chairperson needs to be replaced then consent of 2/3rd board members are required and they are also required to jointly pass the resolution copy of which should be sent to us. In such case, it will be compulsory to obtain our approval before replacement.
- In case of "Difference of Opinion [if any]" among the Board members, our decision will be final and binding to everyone.

"

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1. General,

2. Ethical Guidelines,

3. Submission of Manuscripts,

4. Manuscript's Category,

5. Structure and Format of Manuscript,

6. After Acceptance.

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The recommended size of original research paper is less than seven thousand words, review papers fewer than seven thousands words also.Preparation of research paper or how to write research paper, are major hurdle, while writing manuscript. The research articles and research letters should be fewer than three thousand words, the structure original research paper; sometime review paper should be as follows:

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- One should avoid outdated words.

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Numerical Methods: Numerical methods used should be clear and, where appropriate, supported by references.

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References

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25. Take proper rest and food: No matter how many hours you spend for your research activity, if you are not taking care of your health then all your efforts will be in vain. For a quality research, study is must, and this can be done by taking proper rest and food.

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27. Refresh your mind after intervals: Try to give rest to your mind by listening to soft music or by sleeping in intervals. This will also improve your memory.

28. Make colleagues: Always try to make colleagues. No matter how sharper or intelligent you are, if you make colleagues you can have several ideas, which will be helpful for your research.

29. Think technically: Always think technically. If anything happens, then search its reasons, its benefits, and demerits.

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33. Report concluded results: Use concluded results. From raw data, filter the results and then conclude your studies based on measurements and observations taken. Significant figures and appropriate number of decimal places should be used. Parenthetical remarks are prohibitive. Proofread carefully at final stage. In the end give outline to your arguments. Spot out perspectives of further study of this subject. Justify your conclusion by at the bottom of them with sufficient justifications and examples.

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Informal Guidelines of Research Paper Writing

Key points to remember:

- Submit all work in its final form.
- Write your paper in the form, which is presented in the guidelines using the template.
- Please note the criterion for grading the final paper by peer-reviewers.

Final Points:

A purpose of organizing a research paper is to let people to interpret your effort selectively. The journal requires the following sections, submitted in the order listed, each section to start on a new page.

The introduction will be compiled from reference matter and will reflect the design processes or outline of basis that direct you to make study. As you will carry out the process of study, the method and process section will be constructed as like that. The result segment will show related statistics in nearly sequential order and will direct the reviewers next to the similar intellectual paths throughout the data that you took to carry out your study. The discussion section will provide understanding of the data and projections as to the implication of the results. The use of good quality references all through the paper will give the effort trustworthiness by representing an alertness of prior workings.

Writing a research paper is not an easy job no matter how trouble-free the actual research or concept. Practice, excellent preparation, and controlled record keeping are the only means to make straightforward the progression.

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To make a paper clear

· Adhere to recommended page limits

Mistakes to evade

- Insertion a title at the foot of a page with the subsequent text on the next page
- Separating a table/chart or figure - impound each figure/table to a single page
- Submitting a manuscript with pages out of sequence

In every sections of your document

· Use standard writing style including articles ("a", "the," etc.)

· Keep on paying attention on the research topic of the paper

· Use paragraphs to split each significant point (excluding for the abstract)

· Align the primary line of each section

· Present your points in sound order

· Use present tense to report well accepted

· Use past tense to describe specific results

· Shun familiar wording, don't address the reviewer directly, and don't use slang, slang language, or superlatives

· Shun use of extra pictures - include only those figures essential to presenting results

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Choose a revealing title. It should be short. It should not have non-standard acronyms or abbreviations. It should not exceed two printed lines. It should include the name(s) and address (es) of all authors.

Abstract:

The summary should be two hundred words or less. It should briefly and clearly explain the key findings reported in the manuscript--must have precise statistics. It should not have abnormal acronyms or abbreviations. It should be logical in itself. Shun citing references at this point.

An abstract is a brief distinct paragraph summary of finished work or work in development. In a minute or less a reviewer can be taught the foundation behind the study, common approach to the problem, relevant results, and significant conclusions or new questions.

Write your summary when your paper is completed because how can you write the summary of anything which is not yet written? Wealth of terminology is very essential in abstract. Yet, use comprehensive sentences and do not let go readability for briefness. You can maintain it succinct by phrasing sentences so that they provide more than lone rationale. The author can at this moment go straight to shortening the outcome. Sum up the study, with the subsequent elements in any summary. Try to maintain the initial two items to no more than one ruling each.

- Reason of the study - theory, overall issue, purpose
- Fundamental goal
- To the point depiction of the research
- Consequences, including definite statistics - if the consequences are quantitative in nature, account quantitative data; results of any numerical analysis should be reported
- Significant conclusions or questions that track from the research(es)

Approach:

- Single section, and succinct
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- A conceptual should situate on its own, and not submit to any other part of the paper such as a form or table
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- Exact spelling, clearness of sentences and phrases, and appropriate reporting of quantities (proper units, important statistics) are just as significant in an abstract as they are anywhere else

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The **Introduction** should "introduce" the manuscript. The reviewer should be presented with sufficient background information to be capable to comprehend and calculate the purpose of your study without having to submit to other works. The basis for the study should be offered. Give most important references but shun difficult to make a comprehensive appraisal of the topic. In the introduction, describe the problem visibly. If the problem is not acknowledged in a logical, reasonable way, the reviewer will have no attention in your result. Speak in common terms about techniques used to explain the problem, if needed, but do not present any particulars about the protocols here. Following approach can create a valuable beginning:

- Explain the value (significance) of the study
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- Present a justification. Status your particular theory (es) or aim(s), and describe the logic that led you to choose them.
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Approach:

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Materials:

- Explain materials individually only if the study is so complex that it saves liberty this way.
- Embrace particular materials, and any tools or provisions that are not frequently found in laboratories.
- Do not take in frequently found.
- If use of a definite type of tools.
- Materials may be reported in a part section or else they may be recognized along with your measures.

Methods:

- Report the method (not particulars of each process that engaged the same methodology)
- Describe the method entirely
- To be succinct, present methods under headings dedicated to specific dealings or groups of measures
- Simplify - details how procedures were completed not how they were exclusively performed on a particular day.
- If well known procedures were used, account the procedure by name, possibly with reference, and that's all.

Approach:

- It is embarrassed or not possible to use vigorous voice when documenting methods with no using first person, which would focus the reviewer's interest on the researcher rather than the job. As a result when script up the methods most authors use third person passive voice.
- Use standard style in this and in every other part of the paper - avoid familiar lists, and use full sentences.

What to keep away from

- Resources and methods are not a set of information.
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- Leave out information that is immaterial to a third party.

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The principle of a results segment is to present and demonstrate your conclusion. Create this part a entirely objective details of the outcome, and save all understanding for the discussion.

The page length of this segment is set by the sum and types of data to be reported. Carry on to be to the point, by means of statistics and tables, if suitable, to present consequences most efficiently.You must obviously differentiate material that would usually be incorporated in a study editorial from any unprocessed data or additional appendix matter that would not be available. In fact, such matter should not be submitted at all except requested by the instructor.

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- Sum up your conclusion in text and demonstrate them, if suitable, with figures and tables.
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- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
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- Do not present the similar data more than once.
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- Never confuse figures with tables - there is a difference.

Approach

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- Put figures and tables, appropriately numbered, in order at the end of the report
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- Give details all of your remarks as much as possible, focus on mechanisms.
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- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

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Methods and Procedures	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
Result	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
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References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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