

Inhibiting Effects of *Piper guinensis* on Corrosion of Mild Steel in HCl solutions

C. N. Njoku, O. E. Onyelucheya, O. D. Onukwuli, I. K. Madu, C. F. Okey-onyesolu

Abstract — Corrosion inhibiting effects of *Piper guinensis* (PG) on mild steel in HCl solution (0.5M to 2M) was investigated using gravimetric technique at temperatures of 303K to 333K. The results indicate that the extracts inhibited the corrosion process in the acid media and inhibition efficiency improved with inhibitor concentration but decreased with increase in temperature, which suggests a physical adsorption. Analysis of the results showed that the linear and quadratic effects of the process variables were highly significant, as well as, the interaction effect of temperature and time. A mechanism of physical adsorption is proposed for the inhibition behavior. Adsorption of the inhibitors on mild steel surface followed the Langmuir's adsorption isotherm. The values of the free energy of adsorption ΔG_{ads} indicated that all the extracts which mainly contain alkaloid could serve as an excellent green inhibitor for corrosion of mild steel in HCl solution. This study has revealed that *Piper guinensis* extract is a good inhibitor for the corrosion of mild steel in HCl solution.

Index Terms — Corrosion Inhibitors, Gravimetric method, Inhibition efficiency, *Piper guinensis*.

I. INTRODUCTION

The corrosion of metallic materials in acidic solutions causes a considerable increase in costs. In order to reduce the corrosion of metals, several techniques have been applied. The use of inhibitors during acid pickling procedure is one of the most practical methods for protection against corrosion in acidic media. Most of the effective and efficient organic inhibitors are those compounds containing hetero-atoms such as oxygen, nitrogen, sulphur, and phosphorus which allow adsorption on the metal surface [11]. To be effective, an inhibitor must also displace water from the metal surface, interact with anodic or cathodic reaction sites to retard the oxidation and reduction corrosion reactions, and prevent transportation of water and corrosion active species on the surface. Inhibitors, which reduce corrosion on metallic materials, can be divided into three kinds: (i) inorganic inhibitors, (ii) organic inhibitors and (iii) mixed material inhibitors [6]. However, in the application of these inhibitors for corrosion control, factors such as cost, toxicity, availability and environmental friendliness are very important. Thus, recently, researchers are focusing on natural products as corrosion inhibitors.

C. N. Njoku, Department of Chemical Engineering, Federal University of Technology, Owerri, Nigeria. +2348063308673.

O. E. Onyelucheya, Department of Chemical Engineering, Federal University of Technology, Owerri, Nigeria. +2348033691707.

O. D. Onukwuli, Department of Chemical Engineering, Nnamdi Azikiwe University Awka, Nigeria. +2348063635864.

I. K. Madu, Department of Chemical Engineering, Federal University of Technology, Owerri, Nigeria. +2347033846694.

C. F. Okey-onyesolu, Department of Chemical Engineering, Nnamdi Azikiwe University Awka, Nigeria. +2348037856752.

Recent literature has shown that plant materials such as *Azadirachta indica* [14], *Occimum viridis* [16], *Strychnosnux-vomica* [19], *Prosopis cineraria* [22], *Hibiscus sabdariffa* extract [15], olives leaves [10], *Daturastramonium* [20], *Aloe vera* extract [1] as well as *Phyllantusamarus* extract [18] are effective inhibitors for metal in aggressive solutions. The phytochemical analysis and the inhibitive and adsorptive properties of ethanol extract of *Piper guinensis* for the corrosion of mild steel in HCl solution by standard weight loss measurement at different temperatures, concentrations of inhibitors, concentrations of acids, and exposure time were discussed in this study.

II. MATERIALS AND METHODS

A. Corrosion study

The material used for the study was mild steel of composition (wt %) as determined by quantitative method: Mn (0.6), P (0.36), C (0.15) and Si (0.03) and the rest Fe. The mild steel sheets were mechanically pressed, cut into different coupons, with a dimension of 5 x 4 x 0.11 cm for each sheet. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in air before preservation in a desiccator. All reagents used for this study were analytical grade and double distilled water was used for their preparation. Concentrations of HCl used for weight loss studies were 0.5, and 2 M respectively.

B. Phytochemical test

The extracts were subjected to phytochemical screening to detect the presence of secondary metabolites. These includes; alkaloids, flavonoids, tannin and steroids.

C. Plant extract

Weighed amounts of the dried and ground plant materials were refluxed at 30°C for 3hrs in ethanol. The mixture was cooled to room temperature and filtered to get stock solutions of the extracts. The residue was dried and weighed to determine the amount of extracted material and hence the concentration of the stock solutions.

D. Gravimetric method

In the gravimetric experiment, a previously weighed metal (mild steel) coupon was completely immersed in 200 ml of the test solution in an open beaker. The beaker was covered with aluminum foil and inserted into a water bath maintained at 303K. After every 3 h, the corrosion product was removed by washing each coupon (withdrawn from the test solution) in a solution containing 50% NaOH and 100 g l⁻¹ of zinc dust. The washed coupon was rinsed in acetone and dried in the air before reweighing. The experiment was repeated at 333 K. In

each case, the difference in weight for a period of 12 h was taken as the total weight loss. From the average weight loss (mean of three replicate analysis) results, the inhibition efficiency (%I) of the inhibitor, the degree of surface coverage (θ) and the corrosion rate of mild steel(CR) were calculated using equations 1, 2 and 3 respectively [7]:

$$\%I = \left(1 - \frac{W_1}{W_2}\right) \times 100 \dots\dots\dots (1)$$

$$\theta = \left(1 - \frac{W_1}{W_2}\right) \dots\dots\dots (2)$$

$$CR = \frac{\Delta W}{A_t} \dots\dots\dots (3)$$

Where,
 W_1 & W_2 - weight losses (g) for mild steel in the presence and absence of the inhibitor,
 θ - degree of surface coverage of the inhibitor,
 A - area of the mild steel coupon (in cm^2),
 t - period of immersion (in hours) and
 ΔW ($W_2 - W_1$) - weight loss of mild steel after time, t .

III. RESULTS AND DISCUSSIONS

A. Phytochemical Test

The objective of this analysis is to determine the content of phytochemicals present in the extract. The qualitative analysis tests were carried out to ascertain the presence of the different phytochemicals in the plant before conducting the quantitative analysis. The phytochemical composition of the ethanol extract of PG, shown in table I contains tannins, and saponins, alkaloids. All these constituents combined to form the inhibitory properties of the samples [23]. The results indicated that ethanol extract contains more of saponnins, tannins, flavonoids, and alkaloids [24]. This indicates that the inhibition efficiency of the extract is due to the presence of some or all of the above listed phytochemical constituents [8]. It also stated that saponins, tannins and alkaloids are active constituents of most green inhibitors.

Table I: Qualitative and Quantitative Determination of Phytochemicals presents in the ethanol Extract of PG in gram percent.

Phytochemical	Qualitative	Quantitative (% composition)
Alkaloid	+++	0.76
Flavanoid	+	0.30
Saponin	++	0.55
Tannin	++	0.54

Key: +++ = Present in high quantity.
 ++ = Present in appreciable quantity.
 + = Traceable.

B. Effects of inhibitor concentration on inhibition efficiency

Inhibition efficiency generally increased with increasing extract concentration suggesting that the inhibiting action is

concentration dependent. Also corrosion rates in the presence of the extract reduced remarkably indicating a corrosion inhibiting effect. Fig. 1 shows the variation of inhibition efficiency versus the different concentration of the plant extract on mild steel in 0.5M HCl at 303K after 3hours. The increase in inhibition efficiency as concentration of extracts increases may be due to the presence of complex chemical composition of extracts. It means those inhibitor molecules were adsorbed at mild steel sample interface, where the adsorbed species mechanically screen the coated part of the mild steel surface from action of corrosive medium. The mechanism of adsorption of the inhibitor on the mild steel surface suggests a physical adsorption [15].

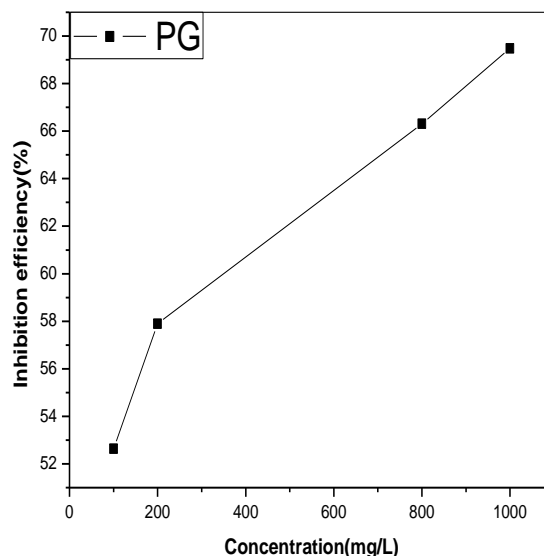


Figure 1. Variation of Inhibition efficiency with concentration of PG on mild steel in 0.5M HCl at 303K at 3hours.

Table II: Inhibition efficiencies ($\eta\%$) of ethanol extract of *Piper guinensis* on mild steel in HCl solutions at 303K and 3hrs.

Conc. (mg/l)	$\eta\%$ for			
	0.5M HCl	1.0M HCl	1.5M HCl	2.0M HCl
100	52.64	50.14	46.52	40.17
200	57.89	53.91	48.68	42.01
800	66.31	57.51	51.39	45.09
1000	69.48	60.12	55.73	51.87

C. Effects of immersion time on inhibition efficiency

Table III shows the variation of inhibition efficiency with time for the corrosion of mild steel in 0.5M, and 2M concentration of HCl containing various concentrations of ethanol extract of the plants at 303K. From the table, it can be seen that inhibition efficiency decreases with an increase in exposure time. The longer the metal remains in the corrodent, the higher the corrosion rate. Reference [9] noted that the phytochemical constituents of plant extract, such as tannin, saponin, alkaloids, and steroids are the major factors that determine the inhibition efficiency of the plant extract.

Table III: Variation of Inhibition efficiency with time for the corrosion of mild steel in acid concentration containing different concentrations of *Piper guinensis* at 303K.

Time (hrs)	100mg/l 0.5M HCl	1000mg/l 0.5M HCl	100mg/l 2.0M HCl	1000mg/l 2.0M HCl
3	52.92	69.19	48.16	52.85
6	51.32	63.72	47.23	50.89
9	50.21	59.45	45.93	48.30
12	49.92	56.25	43.87	47.34

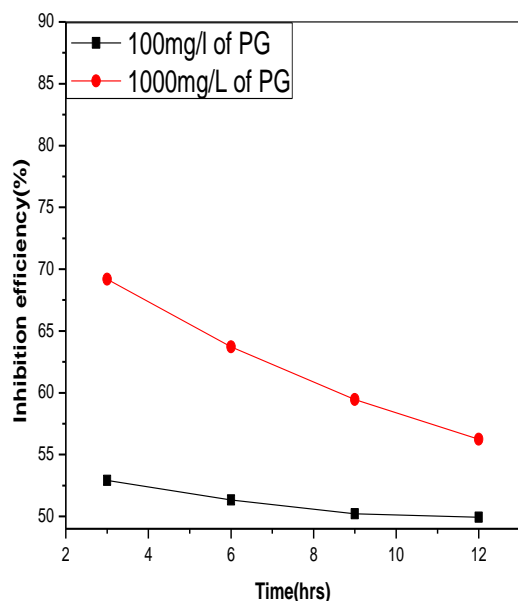


Figure 2: Variation of inhibition efficiency with time for the corrosion of mild steel in 0.5M acid concentration containing various concentrations of the plant extracts at 303K.

D. Effects of acid concentration on inhibition efficiency

Fig. 3 shows the variation of inhibition efficiency with acid concentration for the inhibitor on mild steel at 303K. It was observed that inhibition efficiency decreased with increase in acid concentration. It clearly reveals a corrosion-inhibiting effect of the plant extract on all the corrodents, which becomes more pronounced with increasing extract concentration, implying a dependence of the inhibition process on the amount of the inhibiting species present in the system; the higher the acid concentration, the higher the corrosion rate. The corrosion – inhibiting effect of PG can be attributed to higher quantities of phytochemical constituents including carboxylic acids, and ascorbic acid. The different constituents may react with freshly generated Fe^{2+} ions on a corroding metal surface forming organo-metallic complexes. The inhibiting effect of such complexes then depends on their stability and solubility in the aqueous corrodent, which from our results as a function of the extract concentration and nature of the corrodent.

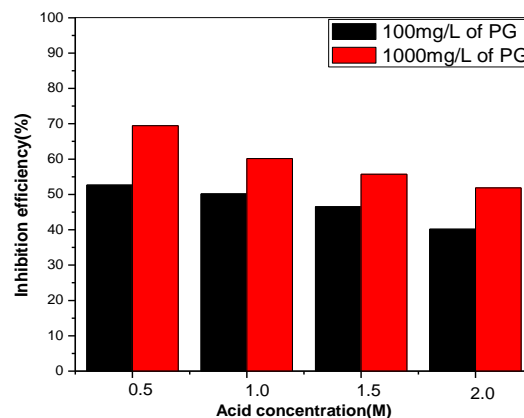


Figure 3: Variation of Inhibition efficiency with acid concentration for *Piper guinensis* on Mild steel at 303K.

E. Effects of temperature on inhibition efficiency

It was found that the rate of corrosion of mild steel is affected by concentration of the plant extracts and temperature. Analysis of the temperature dependence of inhibition efficiency in absence and presence of the different inhibitors gives an insight into the possible mechanism of inhibitor adsorption. Also, Fig. 4 shows a decrease in inhibition efficiency with rise in temperature. These results suggest physical adsorption of the organic matter on mild steel surface in the acidic environment [2]. It is believed that the increase in temperature resulted in inhibitor desorption from the metal surface, exposing a greater area of the metal surface to the corrodent. It has been suggested by [15] that physical adsorbed molecules are attached to the metal at local cathodes and essentially retard metal dissolution by stifling the cathodic reaction, whereas, chemical adsorbed molecules protect the anodic areas and reduce the inherent reactivity of the metal at the sites where they are attached. Values of inhibition efficiency (%IE) obtained at 333K were lower than the values obtained at 303K indicating that at higher temperatures, the inhibitors are gradually desorbed from the surface mild steel.

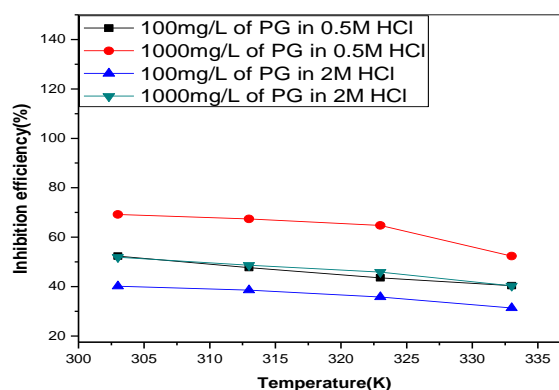


Figure 4: Variation of inhibition efficiency with temperature for the corrosion of mild steel in 0.5M and 2M acid concentration with extract concentrations of *Piper guinensis*.

Table IV: Inhibition efficiency ($\eta\%$) of PG on mild steel corrosion in 0.5M HCl and 2M HCl at different temperatures.

Temp. (K)	HCl conc. (M)	$\eta\%$	
		100mg/l extract	1000mg/l extract
303	0.5	52.32	69.19
	2.0	40.17	51.85
313	0.5	47.65	67.35
	2.0	38.56	48.64
323	0.5	43.51	64.73
	2.0	35.82	44.88
333	0.5	40.36	52.35
	2.0	31.33	40.34

F. Thermodynamics and Adsorption Considerations

In order to calculate the apparent activation energy, E_a for corrosion reaction of mild steel in absence and presence of various concentrations of inhibitor, the Arrhenius equation was used:

$$\log\left(\frac{\ell_1}{\ell_2}\right) = E_a/2.303R[(1/T_1) - (1/T_2)] \dots\dots\dots (4)$$

Where,

ℓ_1 & ℓ_2 - corrosion rates at temperatures T_1 and T_2 respectively,
 R - gas constant.

The heat of adsorption (Q_{ads}) of inhibitor on surface of mild steel was evaluated using equation (5) [3].

$$Q_{ads} = 2.303R \left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right) \right] \times \left[\frac{T_1 T_2}{T_2 - T_1} \right] \dots\dots\dots (5)$$

Where,

θ_1 & θ_2 - degree of surface coverage at temperatures T_1 and T_2 respectively,

The calculated values of apparent activation energy E_a , and heat of adsorption, Q_{ads} of different inhibitor extracts are shown in Table V.

The values of free energy of adsorption, ΔG_{ads} of inhibitor on mild steel surface were calculated using equation (6) [12]:

$$\Delta G_{ads} = -2.303RT \log(55.5K) \dots\dots\dots (6)$$

Where,

R - gas constant,
 T - temperature,
 K - equilibrium constant of adsorption, given as $K = (\theta/1-\theta)$, and

55.5 - concentration of water in solution.

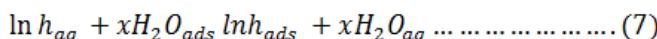
Table V: Activation Energy, Heat of adsorption and free energy of adsorption for Ethanol Piper guinensis Extract in 0.5M HCl of mild steel

	100mg/l	200mg/l	800mg/l	1000mg/l
Activation energy, E_a (KJ/mol)	66.937	67.333	62.474	61.761
Heat of adsorption, Q_{ads} (KJ/mol)	-27.427	-24.298	-10.591	-8.853
Free energy of adsorption, ΔG_{ads} at 303K (KJ/mol)	-10.385	-10.921	-11.825	-12.192
Free energy of adsorption, ΔG_{ads} at 333K (KJ/mol)	-8.698	-9.596	-11.948	-12.523

* E_a of Blank is 55.977KJ/mol

These values are larger than the values for the blank in each concentration, confirming that the extract retard the corrosion of mild steel in the various acid concentrations [10]. The result shows that activation energy increased in the presence of the different extracts in the corrodent. This suggests that the inhibitor is capable of inhibiting corrosion at room temperature but its efficiency become considerably low at elevated temperature, as a result of physical adsorption [14]. The values of heat of adsorption (Q_{ads}) were negative indicating that the adsorption of the extract is exothermic. This trend of heat of adsorption is consistent with the proposed inhibitor adsorption characteristics where negative values indicate physical adsorption [10]. The values of ΔG_{ads} were negative in 303K and 333K indicating spontaneous adsorption of inhibitor on surface of mild steel, and the proposed mechanism is physical adsorption ($\Delta G_{ads} < 40\text{kJ/mol}$) [4].

The adsorption of inhibitor on surface of corroding mild steel sample may be regarded as a substitution process between inhibitor compound in aqueous phase and water molecules adsorbed on mild steel surface:



Where, x is size ratio, the number of water molecules displaced by one molecule of organic inhibitor.

When the equilibrium as described in equation (7) is reached, it is possible to obtain different forms of adsorption isotherm. Langmuir and Florry-Huggins adsorption isotherms were tested for their fit to the experimental data in 0.5M HCl at 303K for the inhibitor as shown from Figures 5 and 6 respectively.

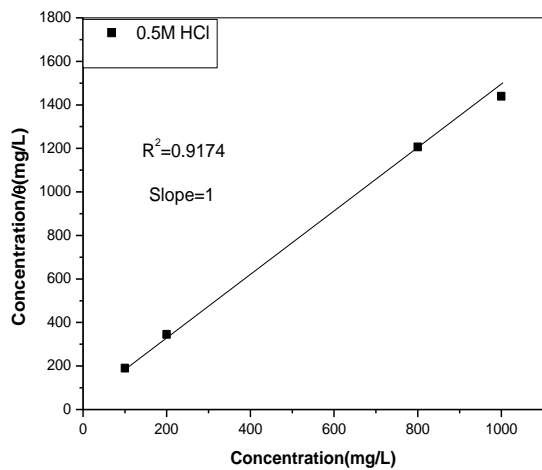


Figure 5: Langmuir adsorption isotherm for ethanol extract of *Piper guinensis* on mild steel in 0.5M HCl at 303K.

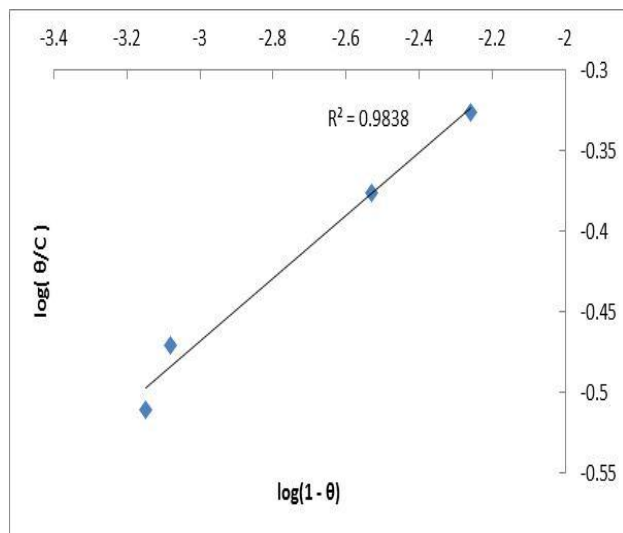


Figure 6: Flory-Huggins adsorption isotherm for ethanol extract of *Piper guinensis* on mild steel in 0.5M HCl at 303K.

In the present study, Langmuir and Flory – Huggins adsorption isotherm agree with a degree of linearity as measured by values of the correlation coefficients (R^2) of 0.9174 and 0.9838 respectively. From the results, Langmuir isotherm was found to be suitable for the experimental findings and had been used to describe the adsorption characteristic of the inhibitor.

Langmuir adsorption isotherm is expressed in equation (8) [21].

$$\frac{C_{inh}}{\theta} = \frac{1}{K} + C_{inh} \dots \dots \dots (8)$$

Where,
 C_{inh} - inhibitor concentration,
 K - equilibrium constant of adsorption,
 θ - degree of surface coverage.

Flory – Huggins isotherm is expressed in equation (9) [17]:

$$\log\left(\frac{\theta}{C_{inh}}\right) = \log K_c + x \log(1 - \theta) \dots \dots \dots (9)$$

Where,
 θ - degree of surface coverage,
 C_{inh} - inhibitor concentration,
 x - number of water molecules replaced by one inhibitor molecule, and
 K_c - is equilibrium constant for adsorption process.

Due to adsorption, inhibitor molecules block the reaction sites and reduce the rate of corrosion. The inhibitor molecules inhibit the corrosion of mild steel by adsorption on the mild steel surface; the adsorption provides the information about the interaction around the adsorbed molecules themselves as well as their interaction with the mild steel surface. Linearity accounts for physical adsorption [15].

IV. CONCLUSIONS

This study indicates that extracts of *Piper guinensis* inhibits corrosion on mild steel surface in HCl solutions. Inhibition efficiency increased with an increase in inhibitor concentration, but decreased with rise in temperature suggesting physical adsorption, and was found to obey the Langmuir isotherm. Also inhibition efficiency decreased with increase in time and acid concentration, respectively. The activation energy of different extracts increases as concentration of inhibitor increases. The negative values of ΔG_{ads} shows that adsorption of alkaloid molecules present in the extract was a spontaneous process, exothermic and was typical of physical adsorption.

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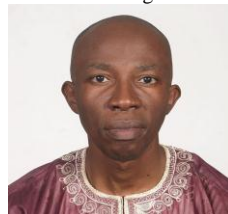
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Chigoziri Nnaemeka Njoku received his B.Eng and M.Eng degrees in chemical engineering at the Nnamdi Azikiwe University, Awka. He is currently an assistant lecturer and Staff Adviser for Students at the Chemical Engineering Department, Federal University of Technology, Owerri, Nigeria and a member of the Nigerian Society of Chemical Engineers. Research interests are on Corrosion, optimization and environmental engineering. He has publications in international journals.



Okechukwu Elechi Onyelucheya has a PhD degree in Chemical Engineering and has been involved in Chemical Engineering teaching and research for 15 Years. He is also a Senior Lecturer with the Department of Chemical Engineering, Federal University of Technology Owerri Nigeria. He has publications in various local and international journals and his current research interests are in Biofuels and Green corrosion inhibition. He is a member of several professional bodies including the American Institute of Chemical Engineers.



Okechukwu Dominic Onukwuli received his B.Eng and M.Eng degrees in chemical engineering at the University of Veszprem, Hungary and the Ph.D degree at the University of Lagos, Nigeria. He has taught for over twenty five years at University of Benin (as a youth corper), at Anambra State University of Technology, Enugu state university of science and technology and at Nnamdi Azikiwe University, Awka. He is a professor of chemical engineering at Nnamdi Azikiwe University, Awka and a Fellow of the Nigerian Society of Chemical Engineers and Nigerian Society of Engineers. His research interests include Chemical kinetics, heterogeneous catalysis and raw material sourcing. He has published in journals both local and international.



Ifeanyi Kizito Madu has a First class degree in Chemical Engineering and is currently a Graduate assistant with the Department of Chemical Engineering, Federal University of Technology Owerri, Nigeria. He is a member of the Nigerian Society of Chemical Engineers.



Chinenye Faith Okey-onyesolu received her B.Sc and M.Sc degrees in chemical engineering at the Nnamdi Azikiwe University, Awka and presently doing her Ph.D in the same institution. She is presently a lecturer II at the chemical engineering department, Nnamdi Azikiwe University, Awka, Nigeria and a member of the Nigerian Society of Chemical Engineers and a member of Nigerian Society of Engineers, and also Staff Adviser for NSCHE Students, Dept. of Chemical Engineering, Committee member, research development and Innovation, Faculty of Engineering UNIZIK. Her research interests include Chemical kinetics, adsorption and modeling.

