

Application of Solar-Irradiated Photocatalyst (Periwinkle Shell Ash) for the Degradation of Ethylbenzene in Aqueous Solution

Felix A. Aisien, Ph.D.¹ and Jude O. Osarumwense, Ph.D.^{2*}

¹Department of Chemical Engineering, Faculty of Engineering, University of Benin, PMB 1154, Benin City, Nigeria.

²Department of Science Laboratory Technology, Faculty of Life Sciences, University of Benin, PMB 1154, Benin City, Nigeria.

E-mail: judeosarumwense@uniben.edu*

ABSTRACT

The degradation of ethylbenzene in aqueous solution using solar-irradiated periwinkle shell ash (PWS) has been investigated. The work was done in a batch system directly under the sunlight and without the sunlight. During the degradation process, the effects of some process variables such as irradiation time, concentration of ethylbenzene, and catalyst dose were investigated. The data obtained were fitted to the pseudo-first and pseudo-second order kinetic equations to determine the rate of reaction. Langmuir-Hinshelwood (L-H) model was also used to determine the rate of adsorption and reaction constants; and the root mean square error (RMSE) was employed to evaluate how well the values obtained from L-H model fit with the experimental values.

The results showed that degradation efficiency of ethylbenzene increased progressive with increase in irradiation time; 92.7% of ethylbenzene was degraded with sunlight and 50.4% without sunlight at optimum conditions of 5g/L catalyst dose for 150 minutes irradiation time. The experimental data followed pseudo-first order equation with maximum regression (R^2) of 0.992. Meanwhile, the L-H kinetics model shows the rate of reaction (K_{LH}) and adsorption constant (k_a) as 1.1962 mg/L.min and 0.015L/mg respectively. The residual error as estimated using RMSE was 0.0884. This is an indication that PWS is a suitable photocatalyst for the degradation of ethylbenzene in aqueous solution.

(Keywords: ethylbenzene, periwinkle shell ash, degradation, photocatalysis, L-H model)

INTRODUCTION

The pollution of groundwater by organic compounds poses a serious risk to potable water supply, and it has caused a major public health interest in many counties (Singh *et al.*, 2016). Despite the contribution of petroleum to the world economy, the environmental pollution due to its refining is of great concern. Wastewater released by petroleum and petrochemical industries are composed of large quantities of hydrocarbon such as BTEX (benzene, toluene, ethylbenzene and xylene), polycyclic aromatic hydrocarbons (PAHs), aliphatic compounds, metal derivatives and other crude oil products (Ray, 1999). Most of the hydrocarbon are hazardous and exist as non-aqueous-phase liquids (NAPLs) in the subsurface environment (Cho *et al.*, 2006). It has been reported that the toxicity of the aromatic compounds is higher than the aliphatic compounds, and more recalcitrant (Mota *et al.*, 2008). The exposure to BTEX compounds for a long period of time can result to skin irritation, central nervous system and respiratory health effects; the major target organs affected by the toxicity of oral and inhaled ethylbenzene are the kidneys and liver (Fayemiwo *et al.*, 2017)

The ineffectiveness and cost implication of purification systems will make wastewaters to become dangerous; this leads to the accumulation of toxic and harmful products in the receiving water bodies with detrimental effects on the ecosystem (Israel *et al.*, 2008). The methods of treatment for groundwater contaminated with hydrocarbons are air stripping, packed tower, adsorption, and biodegradation. The other methods for water treatment include flocculation and filtration, biological treatment, thermal oxidation, and chemical oxidation using hydrogen peroxide, chlorine, ozone, potassium

permanganate and high-energy ultraviolet light (Lin *et al.*, 2005).

The technology of photocatalysis has been studied extensively for its potential as a method for wastewater treatment through its advanced oxidation reaction initiated by solar energy in the presence of a stable metal oxide as catalyst. The involvement of sunlight in the photocatalytic removal of synthetic chemicals from the environment is well documented in many research papers during the last years (Amenghawon *et al.*, 2014; Azuan *et al.*, 2001; Blanco, 2008; Aisien and Osarumwense, 2008). The advantages of photocatalysis over other conventional separation processes such as adsorption, flotation, coagulation and filtration are the destruction of toxic pollutants and consequently inhibiting the generation of toxic residues, while the other separation methods only remove the pollutants and deposit in other places (Mota *et al.*, 2008).

Heterogeneous photocatalytic oxidation process is a novel technology based on the irradiation of a catalyst (usually a semiconductor), which may be photo-excited to form reducing sites (electron-donor sites) and oxidizing sites (electron-acceptor sites), providing great scope of reduction-oxidation (redox) reactions (Liu *et al.*, 2014).

A heterogeneous photocatalytic oxidation adheres to the five discrete steps: the transfer of liquid or gaseous phase reactants to the catalytic surface, adsorption of reactant on catalytic surface, reaction in the adsorbed phase, desorption of product/products and lastly, removal of products from the interface region (Ozogu *et al.*, 2016). The catalytic reaction occurs in the adsorbed phase.

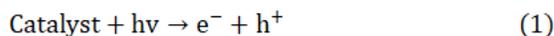
The molecular orbital of semiconductor (catalyst) has a band structure. The bands of interest are the occupied valence band (V_B) and the unoccupied conductance band (C_B), separated by an energy band gap (E_{bg}) as shown in Figure 1. When the catalyst is illuminated with photon ($h\nu$) of greater energy than that of the band gap, an electron (e^-) is promoted from the valence to the conduction band leaving a positive hole (h^+) in the valence band (Pirkanniemi and Sillanpaa, 2002).

At the catalyst surface, the excited e^- and h^+ take part in redox reactions with adsorbed species such as water (H_2O), hydroxide ion (OH^-), organic compounds, or oxygen (O_2). The charges can also react directly with adsorbed pollutants, but reactions with water are more likely because the

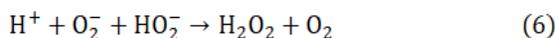
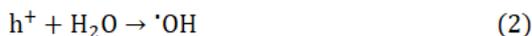
water molecules are far more populous than contaminant molecules (Poulios *et al.*, 1999). Oxidation of H_2O or OH^- by the h^+ produces a powerful and indiscriminate oxidant known as the hydroxyl radical ($\cdot OH$).

For oxidation of pollutants to occur, the oxidation potential of V_B must be higher than the material under consideration (Parra, 2001). The $\cdot OH$ is able to react with organic compounds because of its high oxidation potential, which results to mineralization of the organic compounds by the formation of water and carbon dioxide, or the conversion of the pollutant into less toxic products (Gaya and Abdullah, 2008). The simple mechanism for the oxidation of organic pollutant is expressed with the following equations:

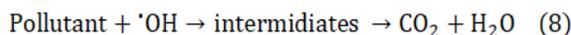
A photo excited catalyst generates e^- and h^+



The e^- and h^+ take part in redox reactions with adsorbed species to generate $\cdot OH$ and super oxide ion (O_2^-)



The photon converts hydrogen peroxide H_2O_2 to more of the hydroxyl radicals:



Finally, the $\cdot OH$ oxidizes the organic pollutant molecules adsorbed on the catalyst surface through intermediates to water (H_2O) and carbon dioxide (CO_2) (Ozogu *et al.*, 2016).

MATERIALS

The Periwinkle shells ash (PWS) used in this work was collected from Chemical Engineering Laboratory, University of Benin, Benin City. Wastewater was simulated by preparing various concentration of ethylbenzene. Flat surface orbital shaker (Model OS-752, Optima, Japan) was used for all experiments. UV/VIS spectrophotometer (Model: T70, PG Instrument) was made use of during the experiment to determine the absorbance of ethylbenzene at wavelength of 220nm. The residual concentrations of ethylbenzene were extrapolated from the standard curve of absorbance versus concentration. All experiments carried out under the sunlight were performed between the hours of 12 noon and 3 pm at a temperature of $32 \pm 2^\circ\text{C}$.

METHODS

Effect of Sunlight and Irradiation Time on the Degradation of Ethylbenzene

One hundred milliliters of 200mg/L ethylbenzene solution was mixed with 5g/L of PWS in a 500 mL Erlenmeyer flask. The flask was placed on a flat orbit shaker and shaken at a speed of 1600 rpm for 3 hours under the sunlight. The effect of irradiation time was studied by withdrawing samples at every 20 minutes and filtered prior to UV/VIS spectrophotometric analysis. Similarly, the experiment was repeated inside the laboratory (without sunlight). The Erlenmeyer flask was wrapped with foil paper to prevent UV-ray from sunlight from penetrating into the solution. A third flask was set up without PWS but placed under the sunlight (Crittenden *et al.*, 1996; Osarumwense and Aisien, 2012).

Effect of Catalyst Dose

The optimum catalyst dose to ensure an efficient degradation of ethylbenzene was determined by mixing 100mL sample of 200mg/L ethylbenzene with varying amount of PWS (1 to 10g/L). The mixture was stirred continuously using the orbit shaker at a speed of 1600rpm for 3 hours under the sunlight with constant aeration. After the 3 hours of reaction, the samples were centrifuged, filtered and analyzed.

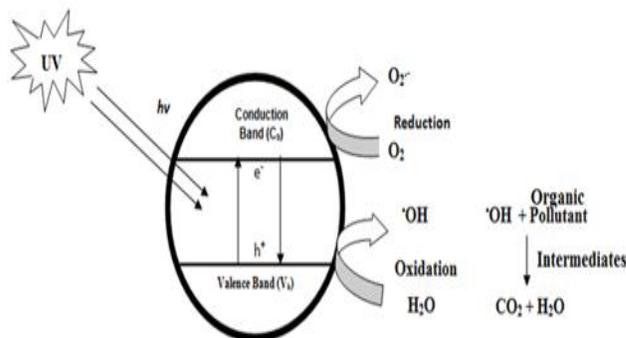


Figure 1: A simplified diagram of photocatalytic degradation of organic pollutant

Effect of Initial Concentration of Ethylbenzene

Initial concentration of a pollutant in wastewater is one of the most important parameters, which has to be analyzed as it may affect the efficiency of the treatment process. Aliquot of 50, 100, 200, 300 and 400 mg/L ethylbenzene solution (100mL) were placed separately in five Erlenmeyer flasks; 5g/L PWS was added to each flask and shaken for 3 hours. After the pre-defined time of 20, 40, 60, 90, 120, 150 and 180 minutes, the samples were withdrawn and analyzed.

Analysis of Experimental Data

The percentage of ethylbenzene degraded was calculated using Equation (9) given as:

$$\% \text{ Removal of ethylbenzene} = \frac{C_0 - C_e}{C_0} \times 100 \quad (9)$$

C_0 = initial concentration of pollutant in sample (mg/L)

C_e = equilibrium concentration of pollutant in sample (mg/L)

The ethylbenzene photodegraded at irradiation time t , and at equilibrium by the periwinkle shell ash were calculated according to Equations (10) and (11).

$$q_t = \left(\frac{C_0 - C_t}{m} \right) V \quad (10)$$

$$q_e = \left(\frac{C_0 - C_e}{m} \right) V \quad (11)$$

where, q_t and q_e are the quantities of ethylbenzene (mg/g) degraded at time t and equilibrium respectively, C_o, C_t and C_e are the initial, instantaneous and equilibrium concentration of ethylbenzene, respectively, in solution, m is mass of the catalyst used and V is the volume of the solution.

RESULTS AND DISCUSSION

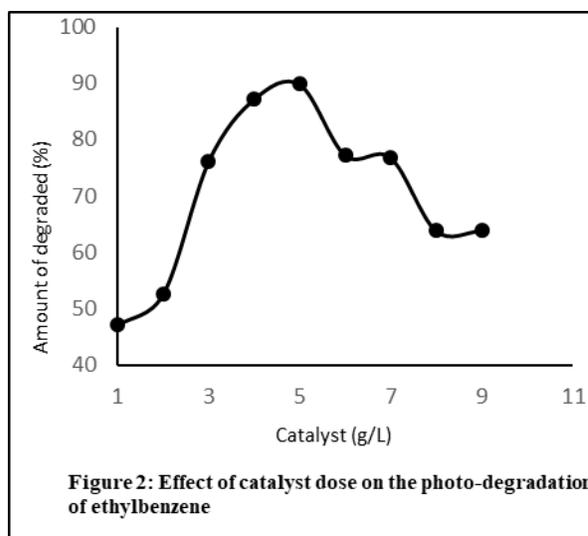
Characterization of Periwinkle Shell Ash (PWS)

The characterization analysis of PWS have been reported in Amenaghawon *et al*, (2014) and Aisien *et al*, (2013). According to them, the FTIR analysis revealed several peaks showing various functional groups. The peaks at 1374 cm^{-1} , 1797.58 cm^{-1} , 1561 cm^{-1} and 3626.59 cm^{-1} in the infrared spectrum correspond to C-H, C=O, C=C and O-H bonds respectively. The peaks in the range of $2700\text{-}1430\text{ cm}^{-1}$ and $900\text{ to }1380\text{ cm}^{-1}$ indicate the presence of phenyl groups and carboxylic groups in periwinkle shell ash.

In a slightly acidic medium, the strong bond of the various functional groups are protonated to be strong adsorption sites for organic molecules. It was ascertained from their studies, as shown in the XRD analysis that periwinkle shell ash is composed of calcium oxide (41.3%), silica (33.2%) aluminum oxide (9.2%), iron (19.2%) and Zinc (16.5%). It was reported in Amenaghawon *et al*, (2014) that oxides of metals exhibit catalytic properties.

Effect of Catalyst Dose

Figure 2 presents the effect of PWS dose on the photo-degradation of ethylbenzene. As the catalyst dose increases from 1g/L to 5g/L, the degradation percentage increases from 47 to 90%. Further increment beyond 5g/L did not show significant increase in the degradation efficiency. Researchers have reported that the active sites available on the catalyst were responsible for the degradation of the organic molecules because the active sites increase with increasing catalyst dose. But further increase in catalyst dose, the excess catalyst particle may have blocked the sunlight from penetrating the solution, thus preventing the formation of OH, hence decrease in degradation of ethylbenzene (Azuan *et al*, 2001; Amenaghawon *et al*, 2014).



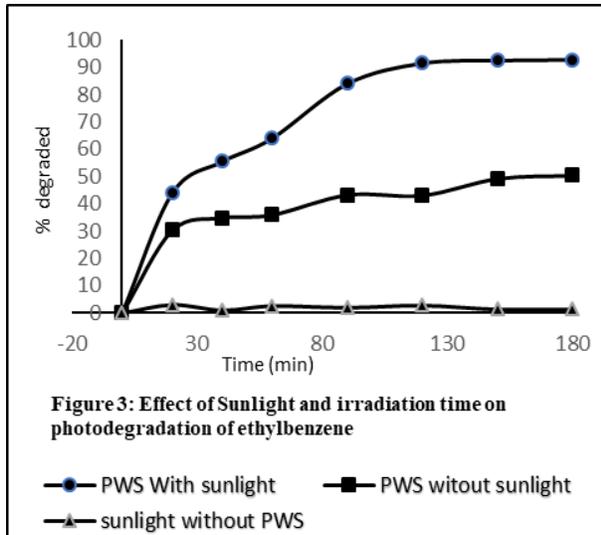
Effect of Sunlight and Irradiation Time on the Degradation of Ethylbenzene

The effect of naturally sourced UV-rays from sunlight on the degradation was studied by exposing the mixture of PWS and ethylbenzene solution to sunlight in the first case; secondly, the mixture of ethylbenzene and PWS without sunlight, and thirdly, the ethylbenzene solution without PWS but exposed to the sunlight. The results show that 92.7% degradation of ethylbenzene was achieved with sunlight, 50.4% without sunlight and 6.9% with sunlight but no catalyst as presented in Figure 3.

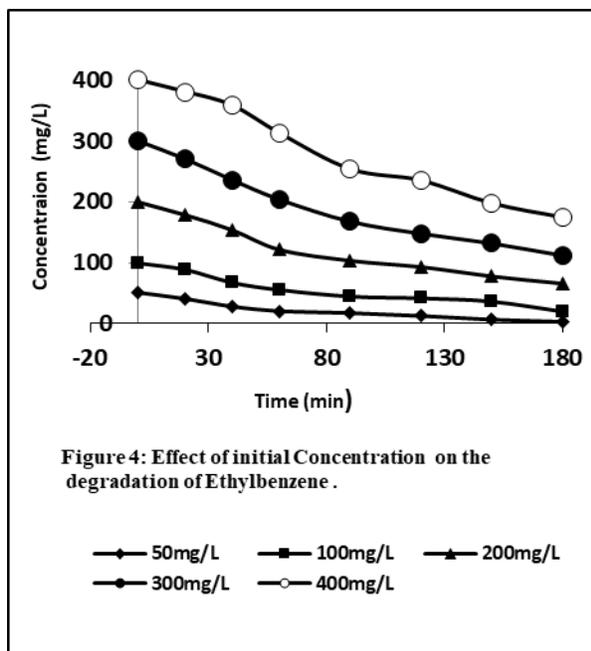
The efficiency of degradation of ethylbenzene (with or without sunlight) shows a progressive and steady increase with increase in irradiation time until about 150 minutes when equilibrium was attained. This is in conformity with previous works (Osarumwense *et al.*, 2015).

Effect of Initial Concentration of Ethylbenzene

It is well known that the initial concentration of reactants plays an important role on the photodegradation of organic compounds as reported in previous study (Hong *et al*, 2001). It is an important parameter, which has to be analyzed as it may affect the efficiency of the treatment process. Therefore, the study was carried out by varying the initial concentration of ethylbenzene from 50mg/L to 400 mg/L keeping the amount of PWS constant at 5g/L.



From the result, it was shown that the degradation follows a steady decay curve from initial concentration of 50mg/L to 3.1mg/L at 3 hours. The same pattern was observed with the other concentrations. Figure 4 shows that the lower concentration degraded more rapidly than the higher concentrations. This was further revealed when the data were subjected to kinetics models.



Kinetics Modeling of Photodegradation of Ethylbenzene

It is important, for the purpose of engineering to determine the rate of reaction that fit the kinetics data of ethylbenzene degradation. The kinetics studies were investigated using pseudo-first order, pseudo-second order, and Langmuir-Hinshelwood models. The pseudo-first order equation is generally expressed as:

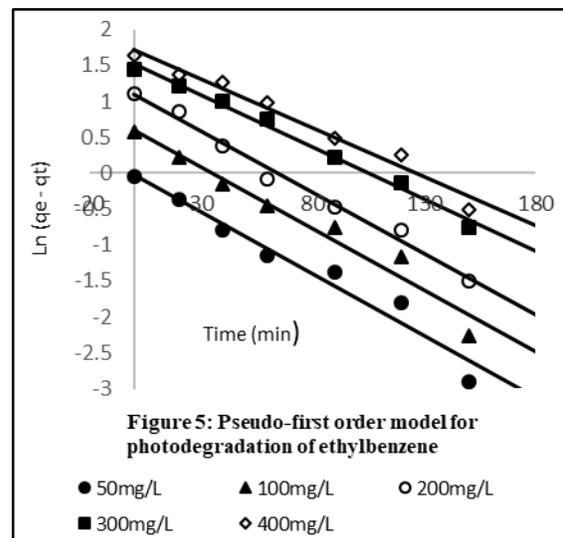
$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (12)$$

The plot of $\ln(q_e - q_t)$ versus t as expressed in Figure 5, gives a linear relationship from which the pseudo-first order rate constants, k_1 (min^{-1}) were determined. The equation of pseudo-second order and the linearized form are given in Equation (13) and (14), respectively.

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (13)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (14)$$

k_2 ($\text{gmg}^{-1}\text{min}^{-1}$) is the rate constant for pseudo-second order equation. The linear plot of Equation (14) as shown in Figure 6 was used to determine the kinetic parameters of the model. The kinetics constants are presented in Table 1.



The photodegradation of ethylbenzene data fitted more to the pseudo-first order than the second order kinetic equation as the reaction rate constants k_1 and the linear regression R_1^2 for pseudo-first order were significantly higher than the values of k_2 and R_2^2 for pseudo-second order. However, both kinetics constants decrease as initial concentration increases. The rate constant decreased from 0.017 to 0.005 min^{-1} for pseudo-first order and 0.009 to 0.001 $\text{gm}^{-1}\text{min}^{-1}$ for pseudo-second order as the initial concentrations increase from 50mg/L to 400 mg/L.

According to Daneshvar *et al.* (2007) and Byrappa *et al.* (2006), decrease in rate constant may be due to the reduction in the number of active sites on the surface of the catalyst because as the concentration increases, more of the active sites are used up and thereby reaction rate decreases.

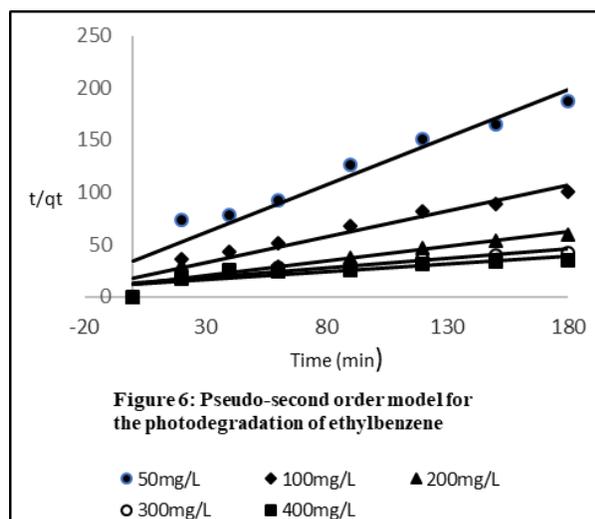


Table 1: Pseudo-first and second order rate constants for ethylbenzene using PWS

Conc (mg/L)	Pseudo-first order		Pseudo-second order	
	K_1 (min^{-1})	R^2	K_2 ($\text{gm}^{-1}\text{min}^{-1}$)	R^2
50	0.017	0.963	0.009	0.796
100	0.012	0.989	0.008	0.929
200	0.007	0.956	0.005	0.902
300	0.006	0.989	0.002	0.412
400	0.005	0.992	0.001	0.401

Langmuir-Hinshelwood (L-H) Kinetic Model

Langmuir-Hinshelwood (L-H) equation is a kinetic model used for photocatalysis. The attack or reaction of the target specie takes place on the surface of catalyst. The simple form of L-H equation is expressed in the following equation:

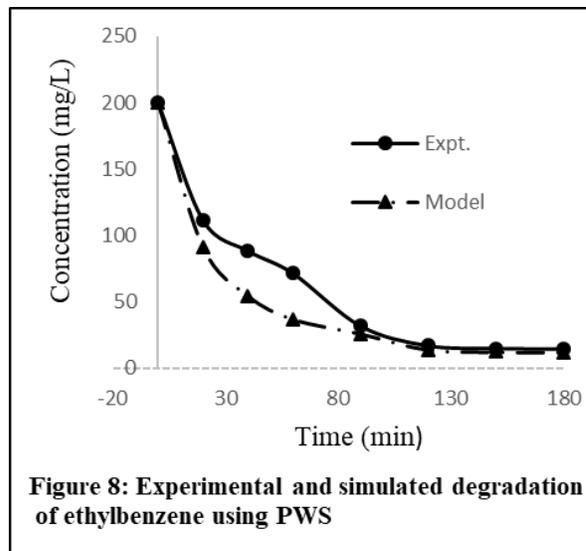
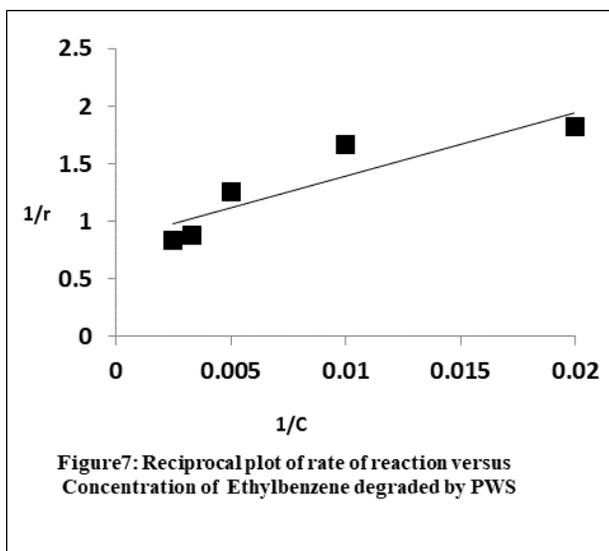
$$r = \frac{K_{LH}k_a C}{1 + k_a C} \quad (15)$$

where r is the rate of degradation of substrate, k_a is adsorption constant of the substrate on adsorbent, C_o is the initial concentration of target specie and K_{LH} is the rate of reaction. The linear form of Equation (15) is given as:

$$\frac{1}{r} = \frac{1}{k_a K_{LH} C} + \frac{1}{K_{LH}} \quad (16)$$

The reciprocal plot of initial reaction ($1/r$) versus initial concentration ($1/C$) gives a linear regression (see Figure 7) where the values of the kinetic constants such as the equilibrium constant for adsorption of substrate (k_a) and reaction rate constant (K_{LH}) were determined. The L-H kinetics model has been used for photocatalysis to show the relationship between the rate of degradation and the initial concentration (Zahraa *et al.*, 2006; Byrappa *et al.*, 2006). The molecule adsorbed on the surface of the catalyst reacts with either the photo-activated catalyst itself (acting as electron donor or acceptor) or the hydroxyl radical ($\cdot\text{OH}$) formed during the oxidation of adsorbed water or hydroxyl ion on the catalyst surface.

The reaction rate and adsorption constants as obtained from Figure 7 are 1.1962 $\text{mg/L}\cdot\text{min}$ and 0.015L/mg respectively. The value of the adsorption constant (k_a) indicate the affinities between the substrate and the catalyst surface, while the Langmuir-Hinshelwood (K_{LH}) rate constant expresses the relationship between the chemical reaction rate and the concentrations of the reacting substances.



To show the performance and validity of Langmuir-Hinshelwood equation, the model was subjected to a standard statistical metrics.

The root mean square error (RMSE) was employed to show how well the values obtained from Langmuir-Hinshelwood model equation deviate from the experimental values. The RMSE is a useful statistical metric widely used to determine model performance in research studies. It actually measures the differences between predicted and observed values. RMSE is a non-negative value, and the value of zero indicates a perfect fit to the data; generally, a lower RMSE value is better than a higher one (Chai *et al.*, 2013). The following formula was used to calculate the RMSE:

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (q_{cal} - q_{exp})^2} \quad (17)$$

where q_{exp} and q_{cal} = the experimental and calculated amount of ethylbenzene degraded.

The result obtained from Equation (17) shows that the prediction error was 0.0884. This is an indication that the estimated degradation of ethylbenzene is slightly deviated from the experimental values because as the RMSE tends towards zero, the better the model. This slight deviation of the experimental values from the calculated values is demonstrated in Figure 8.

CONCLUSION

The photocatalytic degradation of ethylbenzene in aqueous mixture using solar-irradiated PWS was carried out in a batch slurry system directly under the sunlight and in the laboratory. The degradation efficiency of ethylbenzene showed a progressive and steady increase with increase in irradiation time.

About 92% of ethylbenzene was degraded with sunlight and 50.4% without sunlight at optimum conditions of 5g/L catalyst dose for 150 minutes irradiation time. The process fitted more to pseudo-first order than pseudo-second order equation.

Meanwhile, the Langmuir-Hinshelwood kinetics model shows the rate of reaction (K_{LH}) and adsorption constant (k_a) as 1.1962 mg/L.min and 0.015L/mg respectively. Root mean square error was employed to show how well the values obtained from Langmuir-Hinshelwood model fitted with the experimental values.

The estimated RMSE was 0.08841. In conclusion, PWS has been shown as a suitable photocatalyst for the degradation of organic compounds such as ethylbenzene in aqueous solution

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ABOUT THE AUTHORS

Dr. Felix A. Aisien, is a Professor of Chemical Engineering in the Department of Chemical Engineering, Faculty of Engineering, University of Benin, Benin City, Nigeria. He holds a Ph.D. degree in Chemical Engineering from the University of Benin. His research interests are in environmental engineering and pollution control. He is a Registered Engineer and a member of the Nigerian Society of Chemical Engineers.

Dr. Jude O. Osarumwense, is a Lecturer in the Department of Science Laboratory Technology, Faculty of Life Sciences, University of Benin, Benin City, Nigeria. He holds a Ph.D. degree in Chemical Engineering from the University of Benin. His research interests are in renewable energy utilization and environmental engineering. He is a Registered Engineer, a member of the Nigerian Society of Chemical Engineers, and an Associate Member of the Nigerian Institute of Science Laboratory Technology (ANISLT).