



Optimization of Green Hydrogen Production in Alkaline Water Electrolysis with Bio Synthesized Lye and Caustic Soda Electrolytes using Taguchi OA

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Abstract

Overreliance on fossil fuels has led to increased interest in alternative, environmentally friendly fuels like hydrogen. Green hydrogen production via alkaline water electrolysis has zero carbon emissions, preserving its environmental benefits. To synthesized green hydrogen by alkaline electrolysis, potassium hydroxide (KOH) and sodium hydroxide (NaOH) were used as aqueous electrolytes made from biochar of plantain peels and electrolysis of sodium chloride in sea water respectively. The alkalis buffer the electrolyte to enhance its ionization strength for dissociating water molecules in the solution. The alkaline water electrolysis was influenced by parameters like electrolyte concentrations, residence times, temperatures and input voltages. The experiments were designed with factorial Taguchi OA design in expert version-13. The aforementioned parameters were varied to investigate the volume of hydrogen gas generated and modelled. The statistical metric, signal to noise ratio were investigated to evaluate the optimal conditions of the process parameters. Findings using KOH, for volume of hydrogen generated the optimum conditions were 30 g/L concentration, 15 minutes residence time, 85 °C temperature and 7 volts applied. Conversely, with NaOH, the optimal conditions for generating hydrogen using NaOH were 35 g/L concentration, 15 minutes residence time, 45°C temperature, and 7 volts applied. The coefficient of determination for the models generated were within the range of 0.98-0.99, indicating that the models were a good fit to represent the alkaline water electrolysis using both aqueous lye.

Keywords

Hydrogen gas, Alkaline water electrolysis, Biolye-caustic soda electrolytes, Taguchi design

INTRODUCTION

The pursuit of sustainable sources of energy has become imperative in the face of global challenges posed by climate change and the depletion of fossil fuels (Sharma & Ghoshal, 2015; Khzouz & Gkanas, 2017). As societies worldwide grapple with the consequences of reliance on non-renewable energy, the quest for alternative, environmentally friendly fuels has gained prominence (Enontiemonria et al, 2016). Fossil fuels, once the primary source of energy, contribute significantly to greenhouse gas emissions, exacerbating the climate crisis (Bhan et al, 2019). This escalating

environmental concern underscores the urgent need for cleaner and more sustainable energy solutions. Hydrogen, with its abundance on Earth and remarkable green energy credentials, emerges as a compelling alternative to traditional fossil fuels (Aggarwal & Briones, 2010). Unlike conventional fuels, hydrogen combustion produces water vapour as its only byproduct, eliminating the release of harmful pollutants (Moriarty & Honnery, 2017). This intrinsic green-energy characteristic positions hydrogen as a key player in the transition towards a sustainable energy future. Hydrogen possesses high energy density, making it an ideal candidate for efficient energy storage and transportation (Saha et al, 2023). Moreover, hydrogen's versatility allows it to serve as a fuel for various applications, including electricity generation, transportation, and industrial processes (Guilbert & Vitale, 2021). The exploration of hydrogen as a replacement for fossil fuels aligns with the overarching goal of building a cleaner, more sustainable energy landscape (Farias et al, 2022). Hydrogen is often categorised by colour depending on how it is produced and the primary energy source used. Currently, the majority of hydrogen produced is 'grey', meaning it is obtained from fossil fuels through processes like steam reforming of methane or coal gasification (Sher et al, 2021). During the production of grey hydrogen, greenhouse gases (GHGs) are emitted. However, if carbon capture systems (CCS) are utilised in these processes to diminish GHG emissions, the resulting hydrogen can be termed 'blue'. Another method involves producing hydrogen from water through electrolysis, where an electric current splits water into oxygen and hydrogen. When this electrochemical process is fuelled by electricity from renewable sources, the produced hydrogen falls under the category of 'green hydrogen' (Nicoletti et al, 2015; Ajanovic et al, 2022; Superchi et al, 2023).

Electrolysis of water

The water electrolysis system consists of two electrodes, an anode and a cathode, which are made of metallic materials with high electrical conductivity, connected through an external direct current power supply, and immersed in a solution (Fig. 1). The solution referred to as the electrolyte is usually an acid, neutral, or aqueous alkali. When an electric current is applied to the connection and the circuit is closed, electrons are released and flow from the anode through the power source negative terminal to the positive terminal to the cathode, as described by the half-cell equation (1). That is, the electrical potential difference due to the current introduced enables water molecules in the electrolyte at the anode to dissociate, giving off oxygen, hydrogen ions, and electrons. The electrons go through the connection circuit while the hydrogen ions traverse to the cathode, where they combine to form hydrogen atoms, as shown in equation (2). The system is designed with a proton-permeable diaphragm that is used to separate the anode and cathode compartments. Air-tight collectors are used to gather hydrogen and oxygen gases, received at the cathode and anode, respectively.

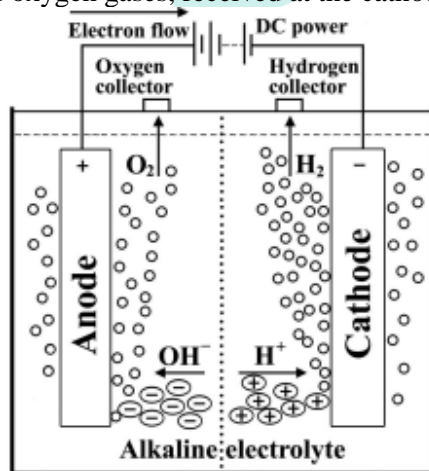
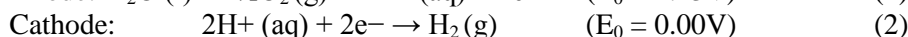
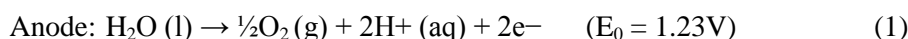


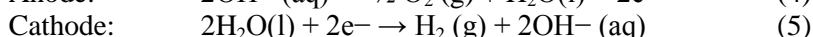
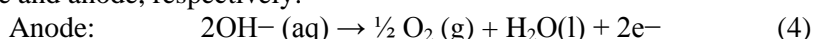
Fig. 1 Fundamental alkaline water electrolysis structure (Santos et al., 2013)



The overall reaction of water electrolysis, given as the difference between the product and reactants, is:



In alkaline water electrolysis, the electrolyte used may consist of high-mobility ions, typically KOH or NaOH in water. KOH is normally chosen over NaOH because of its higher electrochemical conductivity (Gilliam et al, 2007). However, both strong alkalis, usually of group 1 metals, are used as electrolytes to boost conduction and avoid corrosion problems, as is the case with acid electrolytes. When charged with direct electric current, the hydroxide anions are released into the electrolyte solution and transported to the exterior of the anode, where they lose their electrons, which then return through the connecting circuit wires to the positive terminal of the power source. Some common materials for electrodes are iron, tin, nickel, and copper, which have good electrochemical activity and easy availability. The alkaline enhances the ionisation of water molecules in the electrolyte in electrochemical reactions similar to equations (1) and (2), but with the aqueous alkali dissociating into hydroxide ions and allowing the release of hydrogen and oxygen gases in accordance with the reactions at the cathode and anode, respectively.



The overall reactions will generate outcomes given in equation (3), with the same value of thermodynamic cell voltage of -1.23 V (Santos et al, 2013; Brauns & Turek, 2020; Superchi et al, 2023). This paper was aimed at synthesising and utilising sustainable and environmentally friendly alkali KOH and NaOH from waste peels of plantains and seawater, respectively, to synthesise hydrogen gas via alkaline water electrolysis and optimising the output volume of hydrogen gas generated with effects on electrolyte concentration (C_e) loading, reaction residence time (t_r), cell operating temperature (T), and cell input voltage (P).

MATERIALS AND METHODS

The chemical reagents used in this study are distilled water, plantain peels, sea water, and concentrated sulphuric acid. The equipment used is an electronic weigh balance, water bath, oven, 1000°C furnace, sieve mesh, multimeters, copper electrodes, connecting wires, and a variable AC/DC power source.

Preparation of alkalis

Bio-lye preparation: The process of bio-lye preparation involved the use of plantain peels gathered from the vegetable wastes of Afe Babalola University Ado-Ekiti (ABUAD) agro-wastes dumps. Collected plantain peels were chopped into smaller pieces and cleaned with distilled water to get rid of particle dirt, then dried in an oven at 150°C for 4 hours. The dried plantain peels were pulverised and sieved using a mesh size of 200 microns. The powdered peels were then carbonised in an open but controlled environment to produce biochar, which was subsequently dissolved in 250 ml of distilled water to form a solution. The solution was filtered, and the filtrate was allowed to crystallise for a day. The bio-lye was tested for confirmation and molar concentrations following procedures adapted and modified elsewhere (Gilliam et al, 2007; Santos et al, 2013; Enontimonria et al, 2016; Ofori & Awudza, 2017; Brauns & Turek, 2020).

Caustic soda preparation: NaOH was prepared from seawater via electrolysis. It involved the use of two containers, one filled with 100 ml of distilled water and the other with 100 ml of sea water. The containers are connected using a salt bridge, and copper electrodes are placed in both, and these electrodes are connected to a DC source of 12 volts. The voltage causes the sodium chloride present in sea water to break down into its elemental and ionic constituents. The sodium ions are readily combined with water molecules to form a solution of NaOH. The caustic soda was tested for confirmation and molar concentrations following procedures adapted and modified elsewhere (Hou *et al*, 2018).

Electrolytic cell setup configuration

The study was carried out with a single-cell electrolytic setup (Fig. 2). The electrolytic cell was constructed using a plastic container where the electrolyte solution was placed, two 100-mL measuring cylinders held firmly by burette stands with clamps, which collect the various gases, and two copper electrodes of 5 mm in diameter and 130 mm in length. A variable AC/DC power source was connected to both ends of the electrodes to measure the voltage going into the system, with two multimeters connected in series to the electrolytic cell to measure the current passing through the system. Both electrodes were kept on opposite sides of the plastic container, and a fixed distance between the electrodes was maintained. Thereafter, solutions of 25, 30, and 35 g/L concentrations of the synthesised KOH and NaOH were introduced as electrolytes for different runs (Farias et al, 2022). The gases produced at the anode and cathode were measured separately from the two measuring cylinders based on differences between the initial and final displaced electrolyte fluids above the electrodes. The hydrogen gas from the cathode was withdrawn and verified by the flame pop sound test (Yuvaraj & Santhanaraj, 2013; Superchi *et al*, 2023).

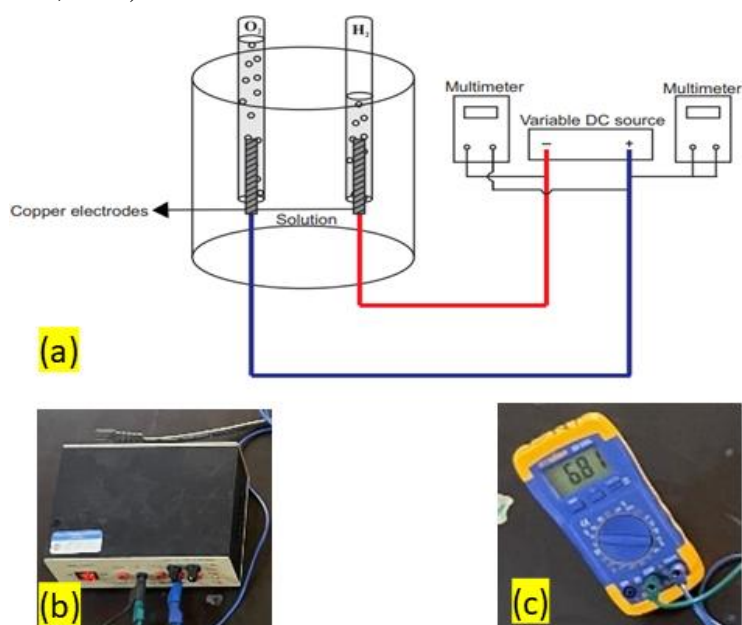


Fig. 2 (a) Schematic of an experimental electrolytic cell setup, (b) Variable DC power source, (c): Multimeter

Design of Experiments

In order to determine the optimum volume of hydrogen gas generated, the Taguchi OA (orthogonal array) design embedded in the design expert version 13 was adopted. The Taguchi designs use factorial analysis to predict the responses of varied process parameters and operating conditions for the alkaline electrolytic process across several runs. Nine runs of experiments (L9) were carried out, which varied four process parameters for each run using a 3-level-4-factor array. Table 1 shows the input alkaline water electrolysis process parameters of electrolyte concentration (Ce), reaction residence time (tr), cell operating temperature (T), cell input voltage (P), and their levels considered in the study.

Table 1 Electrolysis parameters and their levels

Parameters	Symbol	Level 1	Level 2	Level 3
Electrolyte concentration (Ce), (g/L)	A	25	30	35
Reaction residence time (tr), (minute)	B	1	8	15
Cell operating temperature (T), (°C)	C	45	65	85
Cell input voltage (P), (volt)	D	5	7	9

The electrolyte concentration, reaction residence time, cell operating temperature, and cell input voltage are the independent process parameters that vary to yield varying responses or dependent variables of the volume of hydrogen gas produced. The full factorial experimental design of the Taguchi orthogonal array used is shown in Table 2.

Table 2 Full factorial Taguchi L₉ (3⁴) design for the alkaline water electrolysis

Experiment no.	Factor A	Factor B	Factor C	Factor D
1	25	1	45	5
2	25	15	85	9
3	30	1	65	9
4	30	8	85	5
5	35	8	45	9
6	35	15	65	5
7	30	15	45	7
8	35	1	85	7
9	25	8	65	7

RESULTS AND DISCUSSION

Analysis and evaluation of Taguchi OA experimental design results

The responses and analyses of the orthogonal array design outputs using the two alkalis KOH and NaOH electrolytes, which are the volumes of hydrogen gas generated and experiential models of process variables, are described in the following sections.

Analysis of the signal-to-noise (S/N) ratio

Volumes of hydrogen gas generated in AWE with KOH and NaOH electrolytes were measured via the Taguchi OA experimental design for each combination of the control factors: electrolyte concentration, Ce, reaction residence time, tr, cell operating temperature, T, and cell input voltage, P. The optimisation of the control factors was provided by the signal-to-noise ratio. The maximum feasible volume of gas produced is desired for both electrolyte types. Hence, the “larger the better” equation was used for the calculation of the S/N ratio in the design. Table 3 show the design expert-generated S/N ratios for each volume of produced hydrogen gas using KOH and NaOH electrolytes. At the end of the experiments, the average volumes of hydrogen gas produced were 11.4 mL with KOH and 9.1 mL with NaOH electrolytes, respectively. Similarly, average S/N ratio values for the hydrogen gas produced were 17.058 dB and 14.999 dB with KOH and NaOH electrolytes, respectively (Kivak, 2014).

Experimental outcomes with signal-to-noise ratios

Table 3 present the nine Taguchi output responses and signal-to-noise ratios for KOH and NaOH for improved hydroxide ionisation in the AWE system of this study. The response is the volume of hydrogen gas generated as a function of the input variables being electrolyte concentration, Ce, reaction residence time, tr, cell operating temperature, T, and cell input voltage, P.

Table 4 presents the response levels of the S/N ratio of each control factor for hydrogen gas production using the KOH and NaOH electrolytes. It identifies the optimal levels of each control factor for the optimum volume of hydrogen gas production in KOH and NaOH electrolytes. The optimal values of the control factors Ce, tr, T, and P for hydrogen gas production in KOH and NaOH electrolytes are clearly displayed in Fig. 3. These were the maximum or peak S/N responses of the various control factors.

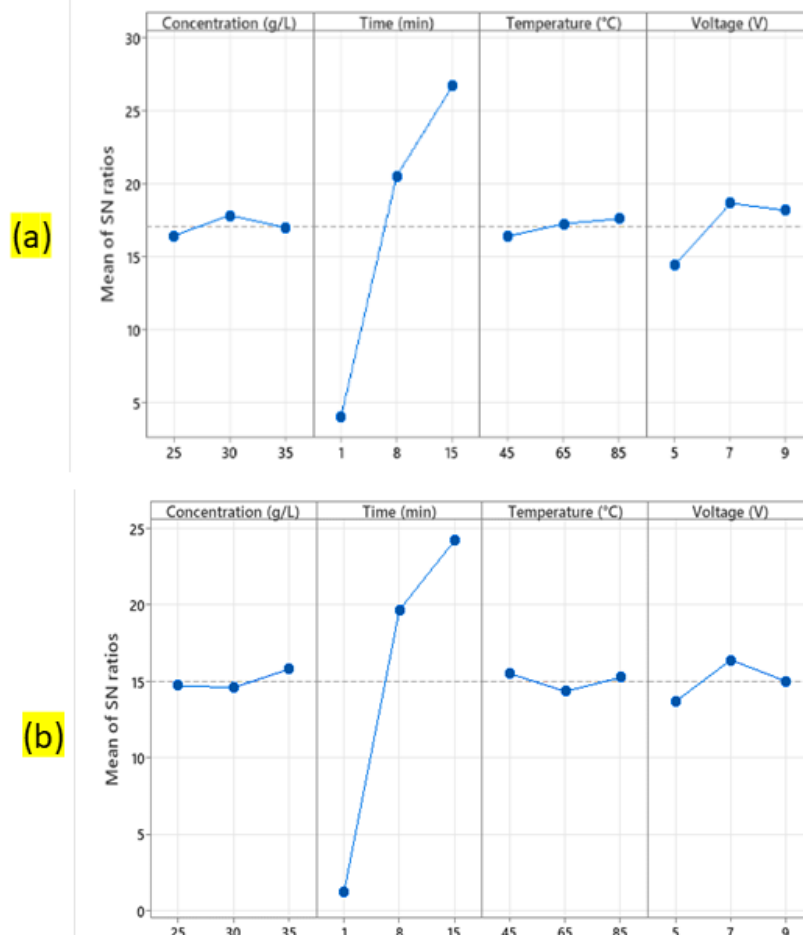
Table 3 Responses of experimental runs and S/N ratio values with KOH and NaOH electrolyte

Experiment no.	Control factors				Vol. of hydrogen, V(H ₂ _KOH) (mL)	S/N ratio for V(H ₂ _KOH) (dB)	Vol. of hydrogen, V(H ₂ _NaOH) (mL)	S/N ratio for V(H ₂ _NaOH) (dB)
	A: Ce (g/L)	B: tr (min)	C: T (°C)	D: P (volt)				
1	25	1	45	5	1	0.000	1	0.000
2	25	15	85	9	24	27.604	16	24.082
3	30	1	65	9	2	6.021	1	0.000
4	30	8	85	5	9	19.085	8	18.062
5	35	8	45	9	11	20.828	11	20.828
6	35	15	65	5	16	24.082	14	22.923
7	30	15	45	7	26	28.300	19	25.575
8	35	1	85	7	2	6.021	1.5	3.522
9	25	8	65	7	12	21.584	10	20.000

Table 4 S/N responses to the volume of hydrogen gas produced

Levels	Factors							
	V _{H₂-KOH}				V _{H₂-NaOH}			
	A	B	C	D	A	B	C	D
1	16.396	4.014	16.376	14.389	14.694	1.174	15.468	13.661
2	17.802	20.499	17.229	18.635	14.546	19.630	14.308	16.366
3	16.977	26.662	17.570	18.151	15.757	24.193	15.222	14.970
Delta	1.406	22.648	1.194	4.245	1.212	23.019	1.160	2.704
Rank	3	1	4	2	3	1	4	2

The best level for each input factor was obtained as the highest S/N ratio of that factor within the three levels defined in the factorial design. Thus, the levels and S/N ratios giving the maximum volume of hydrogen generated using KOH electrolyte solution are Ce of level 2 with a S/N of 17.802, tr of level 3 with a S/N of 26.662, T of level 3 with a S/N of 17.57, and P of level 2 with a S/N of 18.635. That is, in comparison with Table 1 values, to obtain optimum volume production of hydrogen gas, KOH electrolyte concentration Ce of 30 g/L at a reaction residence time of 15 minutes with a cell operating temperature of 85 °C and a DC power voltage of 7 volts. Similarly, using NaOH electrolytes, the control factors conditions of levels and S/N ratios that gave the best volume of hydrogen gas generated are Ce level 3 with S/N of 15.757, tr level 3 with S/N of 24.193, T level 1 with S/N of 15.468 and P level 2 with S/N of 16.366. This means an optimum volume of hydrogen generated was obtained with a Ce of 35 g/L at a tr of 15 minutes, a T of 45 °C, and a P of 7 volts (Kivak, 2014; Chakik *et al*, 2017; Brauns *et al*, 2020).

**Fig. 3** Effect of control factors on the average S/N ratio for hydrogen gas volume (a) using KOH, (b) using NaOH

Evaluation of Experimental Results

The different response volumes of hydrogen gas produced using the KOH and NaOH electrolytes obtained from the experimental study in relation to the controlled process factors are shown in Fig. 4. An increase in reaction residence time had a significant and increasing effect on the volume of hydrogen generated for both KOH and NaOH. Hence, it is an important factor in predicting the volume responses of produced hydrogen gas.

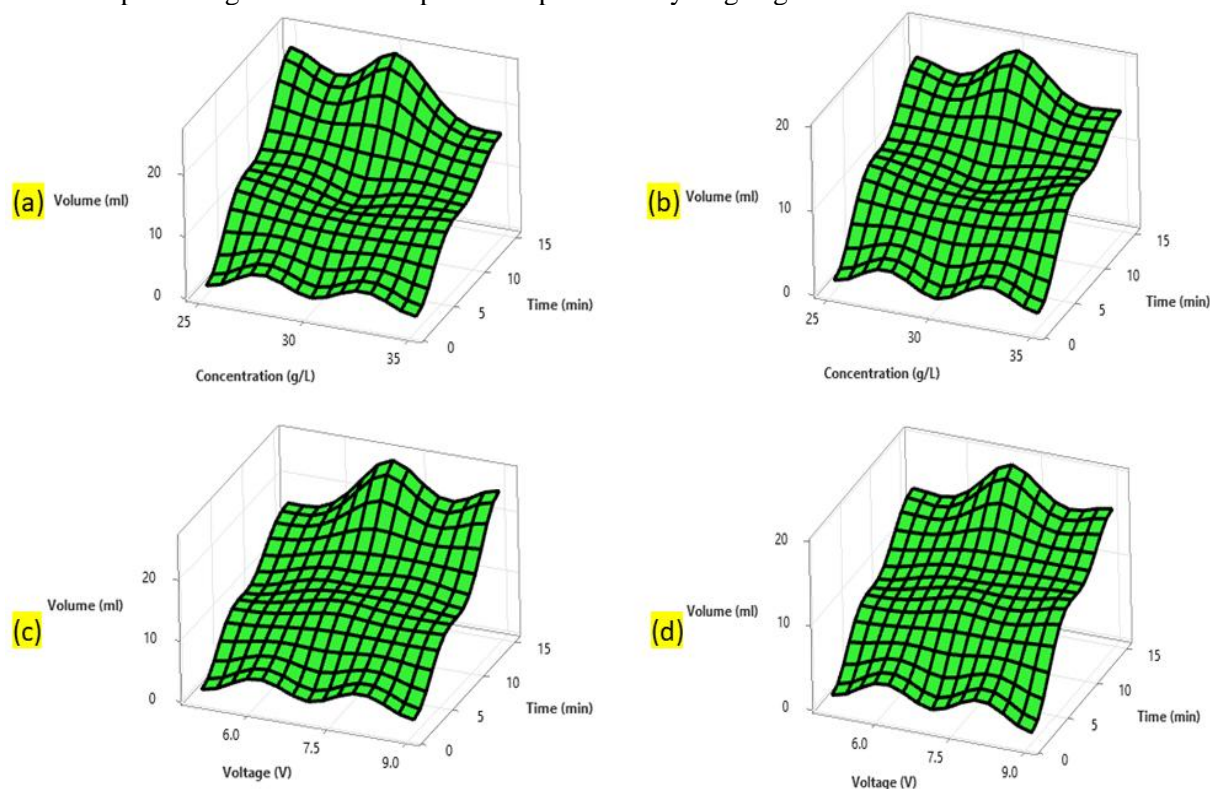


Fig. 4 Volume of produced hydrogen gas versus (a) concentration/reaction time using KOH electrolyte, (b) concentration/reaction time using NaOH electrolyte, (c) input dc power/reaction time using KOH electrolyte, (d) input dc power/reaction time using NaOH electrolyte

Also, an increase in electrolyte concentration and cell input voltage increases the volume of hydrogen gas produced. However, the continuous increase of both factors decreases the volume of hydrogen gas produced. It was also observed that the KOH electrolytes performed better than the NaOH electrolytes based on the observed volumes of hydrogen gas recorded. In addition, showing the effects of each control factor obtained with the design analysis in Fig. 4 verified the findings from the experimental studies on the optimal volume of hydrogen gas produced via KOH and NaOH electrolytes (Chakik *et al*, 2017).

ANOVA for the volume of hydrogen gas produced

The analysis of variance (ANOVA), a statistical tool, was adopted for determining the interactions of the various input factors and their responses. That is, the significance of factors Ce, tr, T, and P represented as A, B, C, and D on the volume yield of hydrogen gas (V_{H_2}) produced using KOH and NaOH electrolytes in the AWE process is presented in Table 5.

Table 5 ANOVA for volume of hydrogen gas produced

Source	Degree of freedom (DF)	Sum of squares (SS)	Mean square (MS)	F-value	p-value	Contribution rate (%)
$V_{(H_2_KOH)}$	6			20.61	0.047	
A	2	14.22	7.11	1.31	0.4336	2.11
B	2	622.89	311.44	57.20	0.0172	92.51
D	2	36.22	18.11	3.33	0.2311	5.38
Error = C	2	10.89	5.44	-	-	-
Total	8	684.22	-	-	-	100.0
$V_{(H_2_NaOH)}$	6			311.86	0.0032	
B	2	346.72	173.36	891.57	0.0011	95.20
C	2	7.39	3.69	19.00	0.0500	2.03
D	2	9.72	4.86	25.00	0.0385	2.67
Error = A	2	0.3889	0.1944	-	-	0.10
Total	8	364.22	-	-	-	100

The model $V_{(H_2_KOH)}$ is statistically significant with F- and p-values of 20.61 and 0.0470, respectively. It implies that there is a good relationship between the volume of hydrogen gas produced and input factors. However, only three of the independent variables, Ce, tr, and P, contributed to the model significance, while the variable T, designated as factor C, impaired it and was placed under the residual error row. In addition, the most significant independent variable with a 92.51 % contribution rate was the reaction residence time factor, with F- and p-values of 57.20 and 0.0172, respectively. On the other hand, model $V_{(H_2_NaOH)}$, F- and p-values indicated a highly significant relationship between the input factors and the response variable. Despite the level of significance obtained, the change in V_{H_2} was only influenced by tr, T, and P factors, while the variable Ce, designated as factor A, showed a negative effect on the model and was placed under the residual error row. The most contributing predictor variable at 95.2 % was the reaction residence time factor, with F- and p-values of 891.57 and 0.0011, respectively (Kivak, 2014).

Regression analyses of the volume of hydrogen gas produced

A predictive model equation for the volume of hydrogen gas ($V_{H_2_KOH}$) produced using KOH electrolyte was developed using regression analyses. The technique analyses the relationships between the independent variables and the dependent variable. This model demonstrates a precise linear relationship between electrolyte concentration (Ce), reaction residence time (tr), and cell input voltage (P) against V_{KOH} without the cell temperature (T) factor, whose presence significantly impairs the model. The linear regression model is given in equation (6) below

$$V_{H_2_KOH} = 11.44 + 0.89Ce - 9.78tr - 2.78P \quad (6)$$

[R^2 : 98.41 %; R^2 (*adj.*): 93.63 %; SD: 2.33; precision adequacy: 12.2]

With an R^2 of 98.41 %, the volume of hydrogen gas produced can be explained by the model with the independent variables. Hence, it indicates a strong correlation and a well-fitting model. The R^2 is supported by a very good R^2 (*adj.*) of 93.63 %, which accounts for the number of independent variables for a conservative estimate of model fit. The low standard deviation of 2.33 was an indication of a low variability in the measured volume of produced hydrogen gas observed around the mean value, which implies that the model is accurate and dependable. The adequacy of precision values measures the signal-to-noise ratio. With a ratio of 12.2 greater than 4.0 (Design Expert-13), the signal-to-noise ratio is highly adequate and a better-quality model with high prediction precision. In a similar way to KOH electrolyte, a predictive model equation for the volume of hydrogen gas ($V_{H_2_NaOH}$) produced using NaOH electrolyte was developed using regression analyses. The model shows a linear relationship between the defined independent and dependent variables. However, only three of the four independent variables participated, namely, tr, T, and P, against $V_{(H_2_NaOH)}$ leaving out the Ce factor, which had no significant effect on the model. The linear regression model is given in equation (7) below.

$$V_{H_2_NaOH} = 9.06 - 7.89tr + 1.28T - 1.39P \quad (7)$$

[R^2 : 99.89 %; R^2 (*adj.*): 99.57 %; SD: 0.44; precision adequacy: 46.5]

The model's R^2 of 99.89 % implies that the volume of hydrogen gas produced can be accurately explained with the independent variables in the equation. It was confirmed by 99.57 % R^2 (*adj.*) of the number of independent variables for a very good model fit. The very low standard deviation of 0.44 was an indication of the insignificant errors in the measured volume of produced hydrogen gas observed in the mean value and that the model is accurate and reliable. The adequacy of precision values of 46.5 shows negligible impact of noise on the data signal and hence high model prediction accuracy (Kivak, 2014).

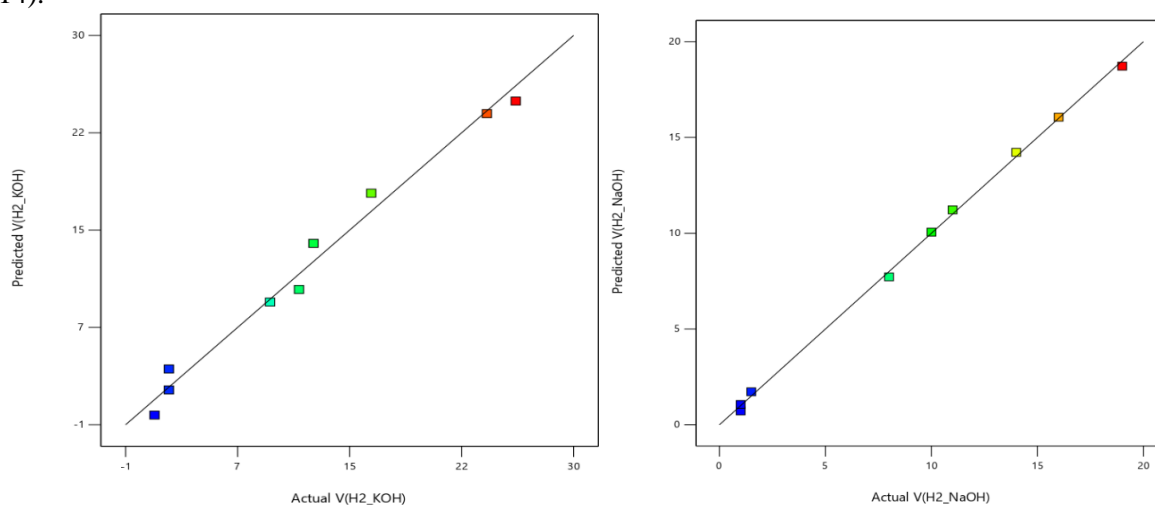


Fig. 5 Correlations of the regression model with experimental results for $V_{(H_2_KOH)}$ and $V_{(H_2_NaOH)}$

Fig. 5 shows the comparison of actual experimental results and predicted values determined by the linear regression model (Kivak, 2014). Although a more thorough prediction could be noticed with the NaOH regression model compared to that of KOH, since both models possess R^2 values of 98.41 % and 99.89 %, this implies a very good relationship between predicted values and observed results. The relation between the experimental outcomes and predictions of the volume of hydrogen gas produced suggested that the developed model adequately forecasts the outputs with the observed process input parameters over the specified range.

CONCLUSION

In this study, green hydrogen gas was produced in alkaline water electrolysis using two varieties of locally synthesised alkali KOH and NaOH, which was carried out and optimised by the Taguchi OA design technique to determine the optimum. The experimental results were analysed and evaluated using ANOVA. The following conclusions may be established:

1. KOH and NaOH aqueous electrolytes were developed from the biochar of plantain peel and the electrolysis of NaCl in sea water as natural, sustainable, renewable, and environmentally friendly sources.
2. Using the factorial design method of Taguchi OA, the factors electrolyte concentration, reaction residence time, cell operating temperature, and DC input voltage considered at three different levels are appropriate factors that affect the AWE process to obtain the volume of hydrogen gas produced as responses.
3. The experimental study generated regression models whose R^2 values were within the range of 0.98-0.99, which indicates that the models were a good fit to represent the alkaline water electrolysis for each response using KOH and NaOH electrolytes and also identified the optimum conditions for the volume of hydrogen gas produced.
4. Using KOH electrolytes, a maximum volume of hydrogen was produced at an optimum condition of 30 g/L electrolyte concentration, 15 minutes reaction residence time, 85 °C cell operating temperature, and 7 volts DC input power. In like manner, using NaOH, the optimum volume of hydrogen generated occurred at a 35 g/L KOH electrolyte concentration, 15 minutes reaction residence time, 45 °C cell operating temperature, and 7 volts DC input power.

ABBREVIATIONS

AWE	Alkaline water electrolysis
Taguchi OA	Taguchi orthogonal array
GHGs	Greenhouse gases
CCS	Carbon capture system
DC	Direct current
ANOVA	Analysis of variance
CV	Coefficient of variation
S/N	Signal to noise

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AUTHORS' CONTRIBUTION

Anthony Owoicho Ijiga:	Writing and preparation of the original draft, set-up fabrication, and Experimental study.
Sylvia Igbafe:	Regression analysis, modelling, and review of grammar.
Akeem Aderibigbe Adebomehin:	Design expert software simulation and optimisation.
Anselm Iuebego Igbafe:	Conceptualisation, methodology, supervision, overall project administration.

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DATA AVAILABILITY

The article contains the data and materials used to support the findings of this study.

DECLARATION OF CONFLICT

The authors declare that they have no competing interests

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