Hydrogen Production via Ultrasound-Aided Alkaline Water Electrolysis

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***Abstract*—Clean energy is a necessity in today's world bearing in mind the undesirable effects of fossil fuel consumption and contributory industries on climate change and human health. The renewable energy resources are introduced as an alternative choice to fossil-based fuels benefiting from abundance, reliability, cleanness in addition to the fact that they are environmentally friendly. Hydrogen as a clean energy carrier is a great candidate for a safer and cleaner future. Alkaline water electrolysis among various hydrogen production methods is a promising technology to produce pure and clean hydrogen. This technology is optimized when coupled with an ultrasonic field resulting in an enhancement in the rate of hydrogen production. The literature studies point out an improvement in mass transfer and 10%-25% energy saving when using the ultrasound. This research work continues the previous study done in this area by investigating the effects of the ultrasound on a conventional water electrolysis cell, furthermore other important parameters were subject of change namely as electrode active surface area and electrolyte solution. The hydrogen generation was measured using a digital hydrogen flowmeter. The average production efficiency of the electrolysis cell was 78%. It was determined that the ultrasound improved the production efficiency by 4.5% and energy efficiency by 1.3%. The act of the ultrasound resulted in bubble removal from the surface of the electrode and the electrolyte solution therefore prepared the electrode surface for the electrochemical reactions thus enhanced the hydrogen generation.**

***Index Terms*—sonoelectrochemistry, electrolysis, hydrogen, ultrasound, renewable energy**

I. INTRODUCTION

The escalating global demands relating to fossil fuels and detrimental aftereffects of their use call for clean and sustainable energy resources through which a more secure future can be envisaged. At the present time the highest energy demand belongs to the oil, natural gas and coal as stated by BP and Shell. The highest annual amount of energy required per decade applies to crude oil, natural gas and coal as can be seen in Fig 1 while the renewable-based energies mount up every year in demand however yet to catch up with the fossil-based resources [1] and [2].

The hydrogen as a fuel is a great source of energy that could be used in different applications including space industry, fuel cells, rockets, pumping and heating and more. Currently the majority of hydrogen comes from steam reforming of natural gas[1] and partial oxidation of hydrocarbons since other techniques are not economically justified. The majority of technologies to produce hydrogen such as coal gasification, biomass processing and etc are not either sustainable or they yield hydrogen in the amount needed [3]. This leads us to any practice with which the electricity is produced from the renewables. Henceforth this electricity could be used to power an electrolyser to generate hydrogen. Solar, hydropower, wind power and ocean power due to their potentialcould be used to supply electricity therefore help provide the future hydrogen generation, sustainable and renewable [4] and [5].

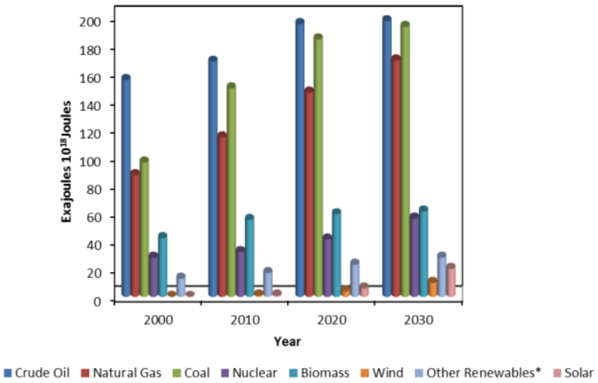

Figure 1. World Primary Energy Demands. * Tidal, geothermal, hydroelectric and waste [1].

The electrolysis of water as a hydrogen production method, accounts for 4%-5% of global production, when combined with the ultrasound, the grouping is said to have increased mass and energy transfer consequently higher hydrogen generation [6]. The electrolysis is a costly method as it requires high energy to advance i.e. 4.5-5 kWh/ m^3, H_2 for most of the industrial electrolysers. The overall efficiency of most electrolysers is normally below 40% since the electricity itself is expensive with average production efficiency of 30%-40%. The solution is to lower the energy requirement for the electrolysis or reducing the electricity costs [7], [8]. It is possible to reduce the energy requirement via reduction of resistances in the process such as electrical and electrochemical, transport-related resistances, development of new electrode materials or refine cell

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Manuscript received May 10, 2013; revised August 1, 2013.

doi: 10.12720/joace.2.1.103-109

geometry. High overpotentials and large ohmic voltage losses are the main reasons to low percentage of global hydrogen production via electrolysis, which brings about the elevated rate of energy consumption and the need for high electrode surface areas. The reason to large ohmic losses is the adsorption of gas bubbles on the surface of electrodes and membrane or distribution within the electrolyte solution [9] and [10].

Alkaline water electrolysis is a technique in which sodium or potassium hydroxides are dissolved in water to enhance its conductivity hence ion mobility within the solution is increased. Potassium hydroxide is the most widely used material as it can resist enormous corrosion loss, and nickel based electrodes are the preferred ones because of the price factor, availability and high activity [6] and [11].

Alkaline water electrolysis in the presence of the ultrasound is a field in the realm of electrolysis, ultrasound and electrochemistry, this is known as sonoelectrochemisty, which was begun with Morigushi in 1930s who studied the effect of ultrasound on the electrolysis of water [12] and [13]. Sheng-De Li et al [12], researched the effects of the ultrasound on an electrolysis cell where the energy efficiency was considerably enhanced. They have used two practices of Linear Sweep Voltammetry (LSV) and galvanostatic polarization. The gas generation efficiency, cell potential and energy consumption were measured for an alkaline electrolyte solution. The hydrogen production efficiency was improved by a range between 5%-18% at high current density while the efficiency of oxygen gas decreased.

Hydrogen gas in an electrolytic process is produced right at the decomposition potential. The hydrogen production is in the molecular form taking place on the surface of electrodes via electrochemical reaction.Molecular hydrogen gas nucleates at the cavity of electrode surface to hydrogen gas bubbles at the cathode active sites [14]. The hydrogen gas bubbles then start to enlarge at the surface of electrode [15] where the Volmer-Heyrovsky-Tafel mechanism occurs consisting of three parts as follows [16]:

$H_2O+e^- = H_{Adsorbed} + OH^-$ (volmer Reaction)

$H_{Adsorbed} + H_{Adsorbed} = H_2$ (Tafel Reaction)

$H_{Adsorbed}+H_2O+e^- = H_2+OH^-$ (Heyrovsky Reaction)

In this work the sonoelectrochemical production of hydrogen is investigated considering the effect of ultrasound, electrolyte and electrode active surface area.

II. THE EXPERIMENTAL

A. Materials

Sodium hydroxide and potassium hydroxide pellets were used as electrolyte base materials, when dissolved in water; the electrolyte solution with the required molar concentration could be obtained. Nickel-based electrodes were used with the diameters of 0.6 cm.

B. Apparatus and Procedure

The electricity was supplied by a power supply (Thurlby PL320 32V-2A), a digital hydrogen flowmeter (Red-y compact flowmeter (L/min)) was applied in order to measure the rate of hydrogen production at a particular potential and concentration. The electrolyte solution prior to the preparation was stirred using a magnetic stirrer with magnetic beads.

The ultrasound was applied to the solution via an ultrasonic transducer (Sonics Ti Alloy, 47 mm in diameter linked to Vibracell Sonics VCX750 set on 20 kHz frequency). The amplitude of the ultrasonic equipment was set on 30%, the separation gap between the electrodes was 7.5 cm and the temperature control was achieved using a pump and heater (Yellow line ET Basic) through the external jacket of the electrolysis cell.

The experiments were carried out in a custom designed cell with a volume of 1000 cm^3 with the ultrasonic transducer being attached to the bottom of the cell. The research was done for Unsonicated (silent) and Sonicated conditions where the effect of the ultrasound was investigated.

The electrolyte material (NaOH and KOH) and electrode active surface area were other changing parameters during this program. The surface areas were increased by increasing the electrode active length (i.e. area of the electrode in contact with the electrolyte solution).

The electrolyte concentration of 0.1M was applied for KOH and NaOH electrolytes for which 4.0 grams sodium hydroxide and 6.60 grams potassium hydroxide pellets (85%) were separately dissolved in water to achieve a final volume of 1000 cm^3 for each electrolyte.

C. Analysis

The electrolysis cell was filled with the electrolyte solution and sealed off with an air-tight grease to prevent the gas escape. The cell can be seen in Fig. 2. Hydrogen gas was evolved at the cathode and oxygen at the anode.

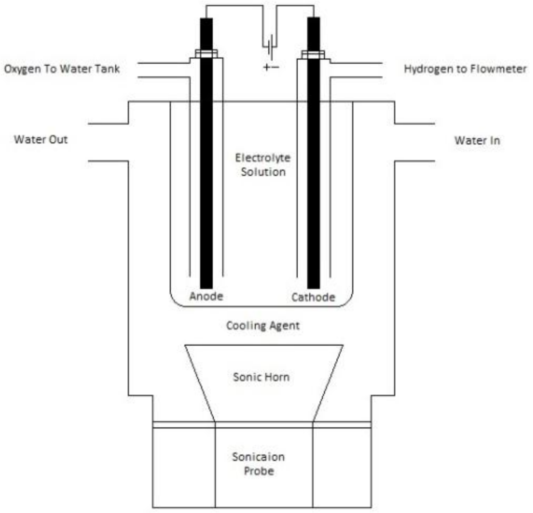

Figure 2. The electrolysis cell with a total volume of 1000 cm^3

The water was used to keep the temperature at the required degree using the cooling jacket shown in Fig 2. The oxygen gas was directed to the water tank and the generated hydrogen gas was measured via a digital hydrogen flowmeter. The water tank is used to balance out the pressure differences inside the gas capturing tubes. The efficiency of the generated hydrogen gas can be calculated using Equation 1:

$$\text{Efficiency}(\%) = \frac{V_{\text{Real}}}{V_{\text{Ideal}}} \times 100\% \qquad (1)$$

Equation 1 is the comparison of the actual hydrogen gas produced against the ideal value. $V_{\text{Real}}(\text{cm}^3)$ is the hydrogen gas produced per unit of time read by the flowmeter and $V_{\text{Ideal}}(\text{cm}^3)$ is the ideal volume of hydrogen gas generated, which can be calculated using the ideal gas law equation at the given conditions. The ideal volume may be calculated using the relation below [12] and [17] :

$$V_{\text{Ideal}}(cm^3) = \frac{\text{SIt}}{\text{nF}} \times \frac{\text{RT}}{\text{P}} \qquad (2)$$

where S is the stoichiometric coefficient, I is applied current, t is the time of operation, n is number of electrons transferred, F is Faradic constant (96484 C.mol^{-1}), T is the operating temperature in Kelvin, R is ideal gas constant (8.314 J.K^{-1}.mol^{-1}) and P is the pressure (atmospheric in Pascal).

The generation rates of hydrogen and oxygen gases may also be found on a molar basis from the equations below in respective order [17] and [18]:

$$N_{H_2} = \frac{JA}{2F} \qquad (3)$$

$$N_{O_2} = \frac{JA}{4F} \qquad (4)$$

where N_{H_2} and N_{O_2} are the hydrogen and oxygen producing rates (mol.s^{-1}), J is the current density (A cm^{-2}), A is surface area (cm^2) and F is Faraday's constant (96485 C mol^{-1})

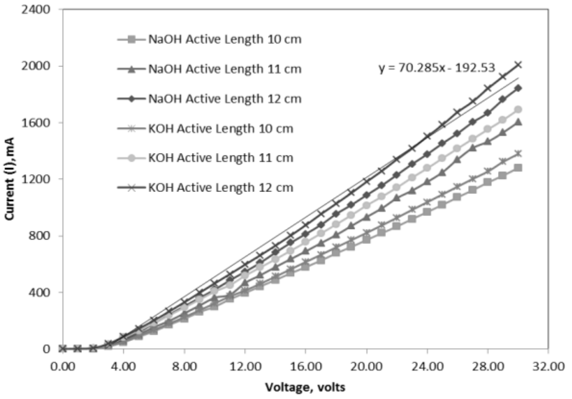

Figure 3. Current-voltage graph for 0.1M experiments at 25 ℃

In Fig. 3 the current-voltage graph is shown where an increase in potential clearly results in an enhancement in the current generation. Potassium hydroxide is a stronger electrolyte than sodium hydroxide hence it is more electrically conductive. This could be observed on the graph in Fig 3. The reason to the better conductivity of potassium hydroxide is that its solution has smaller ions in comparison to the sodium hydroxide, since a solution with smaller ions has less viscous drag in them. The conductivity is inversely proportional to the radius of hydrated ion (i.e. $\lambda \frac{1}{\text{Radius}}$) [19]. The solvation/hydration shells in sodium ions are larger than potassium ions and also sodium ions attract more water to become hydrated as they have greater charge density, which in turn causes larger radius [20] and [21].

The decomposition potential gives an idea of how much initial energy in the form of electricity is required to decompose the electrolyte solution. Decomposition potential can be determined using the current-voltage graph. The equation of the line can be constructed as shown in Fig. 3, when y=0 then x gives the value of decomposition potential. The overpotential can be found using the Equations 5 and 6 however Equations 7 and 8 may be used to determine the overpotential separately for anode and cathode. For the separate calculation a three-electrode system (voltammetry) is required where anode and cathode half-cell reactions can be studied regardless of one another [22] and [23]. Overpotential [η (V)] is referred to as the divergence of electrode cell potential from its value at equilibrium, which can be negative or positive [24]. The electrochemical reactions in a system can proceed when various types of overpotentials are overcome.

$$\eta = E_D - E_{\text{Rev}}^{\text{Cell}} \qquad (5)$$

$$E_{\text{Rev}}^{\text{Cell}} = |\, E_{\text{Rev,C}} - E_{\text{Rev,a}} \,| \qquad (6)$$

where η is the overpotential in volts, E_D is the decomposition potential and $E_{\text{Rev}}^{\text{Cell}}$ is the reversible potential with the value equals to 1.23 volts for pure water and can be found using Nernst equation for different concentrations [22].

$E_{\text{Rev,C}}$ and $E_{\text{Rev,a}}$ in Equation 6 are the reversible potential of cathode and anode in respective order.

Hydrogen and oxygen overpotentials are given in Equations 7 and 8 respectively:

$$\eta_{\text{Cathode}} = 2.3\frac{\text{RT}}{\alpha\text{F}}\log\frac{i}{i_0} \qquad (7)$$

$$\eta_{\text{Anode}} = 2.3\frac{\text{RT}}{(1-\alpha)\text{F}}\log\frac{i}{i_0} \qquad (8)$$

where i_0 is the exchange current density of reaction, R is the universal gas constant (8314 $\text{JK}^{-1}\text{mol}^{-1}$), T is temperature in kelvin, α is transit (transfer) coefficient (dimensionless), F (96,485 C/mole) faraday constant and i is the current (amperes).

The standard potential (E) is calculated using Nernst Equation where a simple redox reaction is considered [25] and [26].

$$\text{O} + n\text{e}^- \rightleftharpoons \text{R}$$

The Nernst equations are:

$$E = E^{o\prime} + \frac{RT}{nF}\ln\frac{C*_O}{C*_R} \qquad (9)$$

$$E = E^{o} + \frac{RT}{nF}\ln\frac{a_O}{a_R} \qquad (10)$$

where E^o(V) is the standard potential, $E^{o'}$ is the formal potential, C*(mol L^{-1}) is the bulk concentration for the specified element, subscripts O and R refer to oxidised and reduced species, a (mol L^{-1}) is the activity, R is the universal gas constant ($JK^{-1}mol^{-1}$) and F is Faraday's constant (C/mol). The value of formal potential depends on the properties of supporting electrolyte and solvent [17] and [25].

$E_{Rev,C}$ and $E_{Rev,a}$ can be found employing Equations 9 and 10 for half-cell reactions at the anode and cathode. Hydrogen half-cell reaction at the cathode [23]:

$$4H_2O\ (l) + 4\ e^- \rightarrow 2H_2\ (g) + 4OH^-(aq)$$

where number of electrons n=4, F= 96485 c/mol, T=298.15K, R= 8.314J.K^{-1}.mol^{-1} and Standard Electrode Potential (SEP) E^0=0 for hydrogen.

Oxygen- water half-cell reaction at the anode is given by:

$$2H_2O\ (l) + 4e^- + O_2\ (g) \rightarrow 4OH^-\ (aq)$$

where number of electrons n=4, Standard Electrode Potential (SEP) E^0=-1.23V for Oxygen and the rest is the same as above.

The decomposition potential and overpotential for the experiment can be found in Table I.

TABLE I. DECOMPOSITION POTENTIAL AND OVERPOTETNIAL

		0.1M NaOH	0.1M KOH
Decomposition Potential (volts)	Silent	2.57	2.52
	Sonicated	2.31	2.14
Overpotential (volts)	Silent	1.34	1.30
	Sonicated	1.09	0.92

III. RESULT AND DISCUSSION

A. Hydrogen Generation

TABLE II. IMPROVEMENT IN HYDROGEN PRODUCTION. * ELECTRODE SURFACE AREA

Experiments at 25 ℃	Improvement in hydrogen Production (%)
0.1M NaOH Various *ESA	13.82
0.1M KOH Various ESA	25

The higher the cell voltage is the greater is the rate of hydrogen generation, the application of the ultrasound [27] and [28] also increased the production of hydrogen. The hydrogen production for sodium hydroxide and potassium hydroxide solutions was improved by about 14% and 25% in respective order when the ultrasonic field was on. This can be seen on Table II, and Fig. 4 also demonstrates an enhanced generation per unit time compared to that of silent condition. The sound waves with the applied low frequency (20 kHz) diffuse more energy compared to high frequency, which causes the formation of cavitation bubbles. This is accountable for cleaning action of the ultrasound [29], which scrubs the surface of the electrode and makes it ready for more electrochemical reactions hence better hydrogen productivity [30]. The ultrasound can also overcome the resistance to heat and mass transfer at the fluid-fluid and fluid-solid boundary layer if its energy is concentrated in the region [28] and [31].

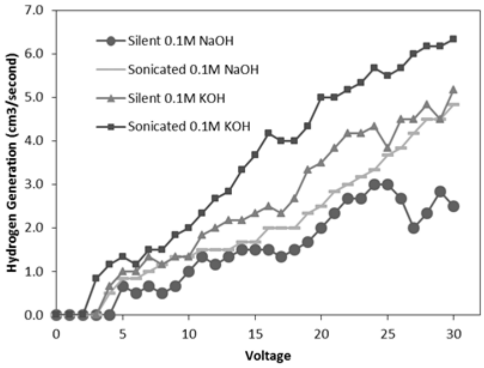

Figure 4. Hydrogen generation for experiments done at 25°C, 10 cm active length (surface area: 32 cm^2)

Hydrogen production efficiency can be found using Equations 1 and 2. The maximum efficiencies of 96% have been mentioned in the literature using electrodes such as low carbon steel [7] and 10 vol% 1-butyl-3-methyl-imidazolium-tetrafluoroborate (MBI.MF4) in water as electrolyte under current density of 44 mA/cm^2 [32] although commercial and industrial electrolysers are usually less than 73% efficient[33].

The production efficiency of 78% was calculated for our work, which is above the industrial electrolysers however considerably less than the maximum possible efficiencies mentioned in the literature [7]. The determined efficiency for hydrogen production can be seen on table 3. The improvement in efficiency was also calculated when the ultrasound was used, which was 5.31% and 3.26% for sodium hydroxide and potassium hydroxide electrolytes respectively. The aforementioned results regarding to the production efficiency and improvement in efficiency were expected to be opposite since KOH is more conductive [20] and [21] and results in more production of hydrogen when compared to NaOH.

The cell design may be the main reason to this as not all gases were allowed into the capturing tube due to its diameter. It was also observed that more gas bubbles were produced for KOH electrolyte but many of which drifted away from the tube and dispersed through the medium.

TABLE III. PRODUCTION EFFICIENCY FOR SILENT EXPERIMENTS

Experiment	**Production Efficiency (%)**
0.1M NaOH at 25 ℃	78.44
0.1M KOH at 25 ℃	77.1

B. Electrode Surface Area

The true definition of electrode real surface area depends on numerous properties and more experimental techniques may be needed to determine the real surface area. The following factors are critical in determination of real surface area including electrode surface heterogeneity or homogeneity, surface micro/macro roughness and dispersion of active materials within the medium solution. The electrode active surface area however is the area in contact with the electrolyte where the electrochemical reaction takes place [17] and [34].

In-situ and ex-situ techniques can be applied to find the real surface area. The in-situ refers to methods like voltammetry, hydrogen and oxygen adsorption from the solution, ion exchange capacity, drop weight, capacitance ratio, Parsons-Zobel plots and negative adsorption.

The ex-situ includes X-ray diffraction, Porosimetry, microscopy, adsorption of probe molecules from the gas phase and etc. [34]. The usual approaches are adsorption processes and capacitance ratio used for solid electrodes, which are based on diffusion-controlled mass transfer when the current is equally distributed. In the 2nd method the differential capacitance is considered in the electrical double layer area using Gouy-Chapman theory [35] and [36].

The active electrode surface area for this research work is assumed equal to the geometrical electrode surface area in contact with the electrolyte, the fluctuations in the level of the electrolyte solution inside the capturing tube is ignored.

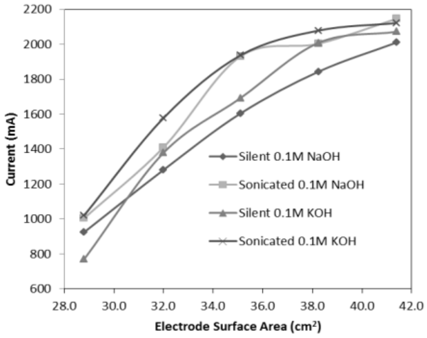

Figure 5. Current vs. surface area for different experiments at 25 ℃ and 22V.

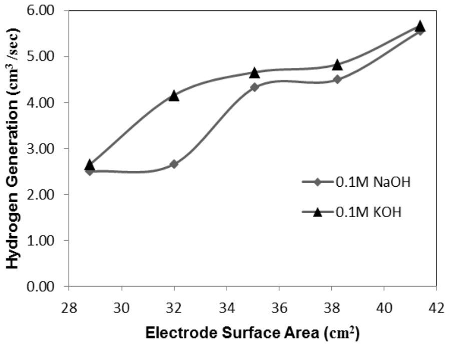

Figure 6. Hydrogen production versus electrode surface area silent experiment at 25 ℃ and 22V.

The hydrogen production at lower potentials between 2V-5V was enhanced by roughly 50% when the electrode surface area was increased by 43.75%.This was determined finding the increase in surface area and consequent enhancement in the hydrogen production resulted from that. Fig. 5 and Fig. 6 illustrate the effect of electrode surface area on the current generation and hydrogen production. The reason to this enhancement is the availability of more active areas for the electrochemical reactions.

The trend shows the current and hydrogen generation are both increased by increasing electrode surface area. In Fig. 5 increasing the surface area by 12.6 cm^2 from 28.8 cm^2 to 41.4 cm^2 increases the current generation by:

-1087 mA for silent 0.1M NaOH

-1143 mA for sonicated 0.1M NaOH

-1303 mA for silent 0.1M KOH

-1102 mA for sonicated 0.1M KOH

It was calculated that increasing the electrode surface area by 43.75% (from 28 cm^2 to 42 cm^2) increased the current generation in a range between 70%-150% for different applied potentials. It was found that 70% was attributed to potentials between 2-5 volts and above 70% belonged to higher potential until 150%, which related to 29V and 30V potentials. The hydrogen generation was also enhanced by improving the surface area and the range was between 50%-130%.

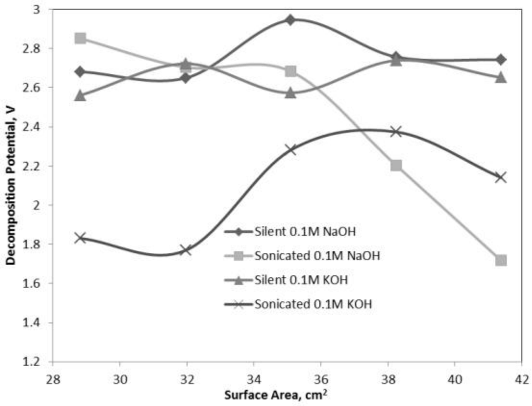

Figure 7. Decomposition potential vs. electrode active area

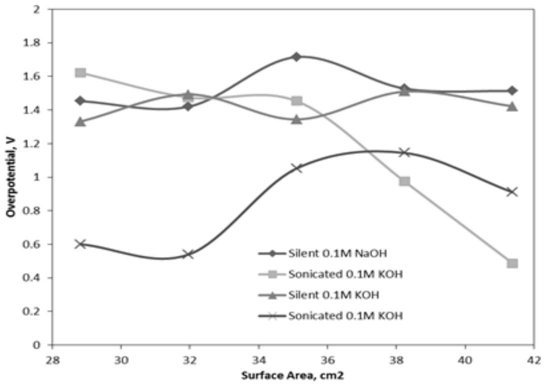

Figure 8. Overpotential vs. electrode surface area

In Fig. 7 the effect of ultrasound and surface area on the decomposition potential is graphically demonstrated where an almost clear observation can only be seen for a reduction in the decomposition potential when the ultrasound is applied. The effect of increased electrode surface area on the decomposition potential does not follow a regular pattern. Fig. 8 specifies the changes in overpotential with an increase in the electrode surface area for sonicated and silent conditions. The pattern is similar to the graph in Fig. 7 since the overpotetnial is the difference between the reversible potential of a cell and decomposition potential as demonstrated in equation 5.Therefore a reduction in the overpotential is considered when the ultrasonication is utilised however the increase in the electrode surface area in Fig. 8 does not lead to a regular alteration in the value of overpotential, this is resulted from the fluctuations in the level of current and hydrogen generation rooted from the pressure differences. The pressure difference is caused by:

- The height and angle of gas conveying tube (from the cell to the gas flowmeter and water tank) as opposed to the level of the electrolyte inside the cell (see Fig. 2)
- The difference in the level of fluid inside the gas capturing tubes where anode and cathode are located [8] and [37].

According to Vladimir M. Nikolic et al a reduction in cell potential is obtained either by changing the catalytic nature of the overpotetnial for Hydrogen Evolution Reaction (HER) or by increasing true surface area of an electrode [7]. The lowest overpotential was determined for potassium hydroxide with the presence of the ultrasound. This again confirms the privilege of KOH over NaOH where less energy is needed for the breakdown of electrolyte components.

A photograph of the experimental setup can be found in Fig. 9 showing the glass cell, yellow pump and heater and power supply.

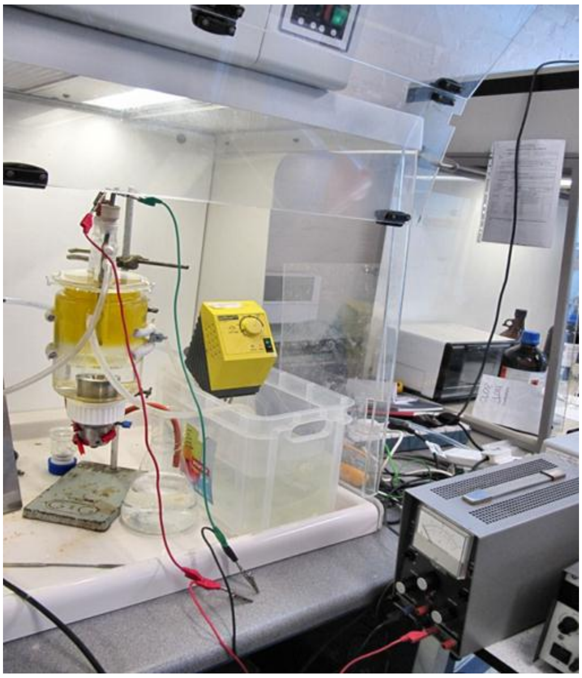

Figure 9. The custom glassware during electrolysis of KOH

IV. CONCLUSION

The electrolysis cell had an average production efficiency of about 80%. The production efficiency is similar to the values mentioned in the literature in a range of 70-96% [7], [38] and [39]. The ultrasound increased the production efficiency by about 4.5%, which is near the 5%-18% range mentioned in the literature [12].

A 43.75% increase in the electrode active surface area improved the current generation by about 70% for potentials between 2 to 5 volts whereas the hydrogen generation was enhanced by about 50% for this potential range.

The decomposition potential and over potetnial were both smaller for potassium hydroxide than sodium hydroxide. Furthermore the ultrasound reduced the values, which helped the electrolysis consume less energy compared to the silent case. The energy saving achieved was 1.3% when the ultrasonic filed was turned on, which is considerably lower than the literature range of 10%-25% [12].

V. FUTURE WORK

The cell geometry could be refined by increasing the diameters of capturing tubes and the application of pressure regulator on the cell to reduce the fluctuations in the electrolyte level within the tube. The ultrasonic transducer could be replaced with the ultrasonic tips, which could be used as electrodes as well hence the rate of electrode cleaning, current and hydrogen generations could be improved significantly since the sound wave distribution would be more localised.

The electrode surface may be modified using slits, cuts and perforations in order to increase the contact with the electrolyte solution. The electrolyte solution could be purged with argon or nitrogen gas for a specified period of time throughout the experiments to remove the air from the cell. The resistance to mass transfer may be reduced using this technique [17].

The use of magnetic field in the electrolysis could be studied applying ferromagnetic electrode materials and potassium hydroxide as electrolyte solution. This could be coupled with an ultrasonic field in order to enhance the production of hydrogen. The direction of magnetism will determine the direction of gas bubble motion and the convection of electrolytic solution as reported by Ming-Yuan et al in their work. They displayed the direction of the electrolyte flow field under the magnetic force effect. Therefore the direction of the ultrasonic irradiation should be set in the direction of magnetism for maximum impact [17] and [40].

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