STUDIES ON THE DIFFERENT TYPE OF CATALYSTS ON AQUEOUS ELECTROLYSIS AS WELL AS MEMBRANE WATER SPLITTING FOR PRODUCTION OF HYDROGEN

Biswajit Mandal[1*], Amalesh Sirkar[2], Parameswar De[3], Avijit Ghosh[4]

[1]Assistant Professor, Department of Chemical Engineering, Haldia Institute of Technology,*
Haldia - 721 657, West Bengal, India.
[2]Professor, of Chemical Engineering, Haldia Institute of Technology, Haldia - 721 657, West Bengal, India
[3]Professor, Department of Chemical Engineering, University of Calcutta, 92 A.P.C. Road, Kolkata - 700 009, India.
[4]Assistant Professor, of Chemical Engineering, Haldia Institute of Technology, Haldia - 721 657, West Bengal, India

Abstract

In search of catalysts for water splitting by electrolysis, the mixture of CuO, ZnO and activated charcoal appears as promising catalyst to enhance hydrogen production rate, and restricted heat generation considerably. The mixture of CuO, ZnO & activated charcoal in the ratio of 2:2:1 practically restrict the heat generation during electrolysis of water for hydrogen generation. The production rate of hydrogen was increased by maximum 15% in presence of said catalysts. Investigation of different catalysts (namely 3%Pt on activated charcoal, NiO, as cathodic and anodic catalysts using cation exchange resin as membrane filler) on membrane water splitting was reported to improve hydrogen production rate manyfold using amide membranes of different composition ranging from 25-75 % acrylamide content in chemically modifying PVOH matrix with poly (acrylic acid –co-acrylamide).

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1. INTRODUCTION

Carbon free hydrogen can be produced by the splitting of water using electrolysis. In order to address the problem associated with low hydrogen production rate by electrolysis using normal alkaline electrolyte, the introduction of suitable catalyst may increase the performance of water splitting. However, till now alkaline water electrolyzer exhibit low energy efficiency due to the high overvoltage of the electrodes. To decrease the activation overvoltage, number of electrocatalysts have been investigated and extensively reviewed. Among these, transition metal oxides and transition metals alloys have been considered most promising for O_2 evolution and H_2 evolution electrode catalysts, respectively [1-3]. Any suspended particle capable of conducting current may act as microelectrode in a close proximity to the macroelectrode. This charge carrying suspended particles assists to decrease the internal resistance in addition with alkali electrolyte. Hence the performance in terms of hydrogen production rate may increase. Further, the suspended particles of semiconductor properties, has the ability to amplify and convert input energy by the addition of impurities or electrical field. The most commonly used semiconductor materials are inorganic crystalline solids. Some oxides also exhibit semiconductor properties that can catalyze the electrolysis. These semiconductor materials are also considered as thermoelectric materials. Thermoelectric materials exhibit thermoelectric effect and thermoelectric effect refer to the phenomena by which either a temperature difference creates an electric field or vice versa.

Some oxides individually or in a form of mixtures of different proportion were reported to be effective for overall water splitting, when suspended in distilled water by magnetic stirring. The effectiveness of mechano-catalytic water splitting was explained by the conversion of mechanical energy supplied by stirring [4].

The mechanism of the effect of electrocatalysts on the reduction of overvoltage was best understood by the chemical as well as physical interaction with the surrounding chemicals and materials. The spin-catalysis concept was introduced in order to describe and classify a wide range of phenomena in which chemical reactions are promoted by substances assisting to overcome spin prohibition. This concept is based on the crucial role of the electron spin in the control of reaction channels in the region of activated complexes. Catalysts can change the rate of reaction by a spin switch with the catalysant in the complex. There are two very effective way to accomplish these changes, firstly by supplying electrons with non-totally quenched orbital angular momentum to the activated complex of the common system (spin – orbit coupling induced spin catalysis) and secondly by supplying nonpaired electrons to the activated complex of the common system (paramagnetic spin catalysis).

When the paramagnetic substance produces a catalytic action upon a diamagnetic substrate reaction, the spin change can easily occur because of the intermolecular exchange interaction. Paramagnetic species accelerate the rate of cis-trans isomerization reaction. Singlet(s) to triplet (T) state mixing in organic molecules induced by exchange interaction with paramagnetic catalysts is responsible for the lowering of the activation energy [5].

Ion exchange membranes are used in electrochemical separation process as well in other applications such as fuel cells. Ion exchange membranes are generally categorized as cation and anion exchange membrane. Proton exchange membranes of different categories are potential to use in electrochemical cell as well as for water splitting. There are three ion exchange membrane were used to make clearer the role of water splitting in development of electro convection. The contribution of the gravitational connection in the ion transfer is negligible. The performance of membrane water splitting depends on the mechanism of mass transfer near membranes/solution interface and on psycho-chemical surface properties. Electro convection occurs due to the action of imposed DC electric field on the electric field produced by the same field [6]. It was also reported that the influence of direct current improved the water splitting efficiency 10^6 to 10^7 times faster than free solution using bipolar ion exchange membrane [7]. Hence the present work was performed using CuO, ZnO and activated charcoal as the potential catalyst in alkaline electrolyzer. In addition ion exchange membrane water splitting was carried out using NiO, 3% Pt on activated charcoal as electrode catalyst and cation exchange resin as modified PVOH matrix with poly (acrylic acid –co-acrylamide) membrane filler.

2. EXPERIMENTAL PROCEDURE

The experiments were performed in an electrolyzer shown in Fig. 1 to study the effect of catalysts on hydrogen production from water. An electrolyzer of capacity of 3 lit., having a double compartment was used. In order to study the effect of catalyst on the rise of temperature during electrolysis, it was recommended to minimize the heat loss from the surface of electrolyzer and was performed by insulating the electrolyzer with asbestos wire. Hydrogen gas was collected by downward displacement of saline water to measure rate of production. Flat type electrodes (each of length: 20.5 cm and surface area: 230.5 cm^2) made of graphite were used. Direct current was imposed to the electrlyzer using a bridge rectifier with varying voltage of 0 – 120 V. Measurements of current and voltage used in the operation were made by an ammeter (0–20 A) and a voltmeter (0–120 V) respectively connected in series and parallel to the electrolyzer.

Fig-1: Experimental set-up for alkaline electrolyte water splitting

The membrane water splitting was performed using an alternative Polymer Electrolyte Membrane for electrolyzer. In this, an attempt was made to synthesize amide membrane. The objective of chemically modifying the PVOH matrix with poly (acrylic acid –co-acrylamide) was twofold. By incorporation of poly (acrylic acid –co-acrylamide) in PVOH matrix, the crystallinity of PVOH would be reduced to give enhanced water permeability [8].

Fig-2: Proton Exchange Membrane Electrolyzer

Further, the introduction of cation exchange resin as membrane filler would give added hydrophilicity to the modified PVOH matrix with poly (acrylic acid –co-acrylamide). After copolymerization of acrylic acid and acrylamide within the matrix of PVOH, the latter is crosslinked with glutaraldehyde to produce a semi interpenetrating network polymer (SIPN), and tested its applicability in water splitting process [9].

Fig-3: Experimental set-up for polymer electrolyte membrane water splitting

To study the effect of catalyst on the membrane, it was loaded by cation exchange resin and tested the effect of electrode catalysts, experiments were carried out with NiO, 3%Pt on activated charcoal individually and in a combined form. Fig. 2 & 3 shows the fuel cell type electrolyzer containing cation exchange resin loaded polymer electrolyte membrane for water splitting.

3. RESULTS AND DISCUSSION

The experimental results showed in Table 1 increase in hydrogen production rate by 24.86% using 2gm/lit of CuO as an additive in 1 (N) KOH solution in comparison with only KOH solution of same concentration. It was also noticed in the present experiments that the rate of hydrogen production was always higher irrespective of voltages and amount of CuO additives than without this. Moreover, the effect of said oxide in electrolyte played an important role to control the rise in temperature during electrolysis due to overpotential loss. Best result with respect to control of temperature rise was attributed with 2gm/lit CuO in 1(N) KOH solution. However, the enhancement of hydrogen production rate might be due to the formation of a thin catalyst layer which subsequently reduced the overpotential loss. It was also logically justified by the fact of mitigation of temperature rise of electrolyzer during its operation. On the contrary, it was also noticed that the excess amount of oxide might not be beneficial towards the better performance of electrolysis because of irregular interaction between ions within electrodes.

To understand the effect of various metal oxides on electrolysis, it was further studied using ZnO of same proportion and working condition. A perusal of Table 2 indicated the same trend of performance but at lower magnitude. However, presence of ZnO in electrolyte was comparatively less effective in terms of hydrogen production rate as well as controlling rise of temperature, but its response was not at all negligible. Unlike the considerable effect of CuO on electrolysis, ZnO exhibited limited effect possibly because of its comparatively low electrical conductivity.

Activated carbon assisted electrolysis was also investigated in search of suitable catalysts mixture in electrolyte for hydrogen production. It was clearly indicated that the activated charcoal in suspended condition in electrolyzer improved hydrogen production rate as well as, and was able to control the rise of temperature and represented by Table 3. The improved hydrogen production might be due to the increase in ion mobility assisted by activated charcoal particles. Again one of the plausible reasons for this enhancement is the adherence of charcoal particle on the graphite electrodes that may increase the effective surface area and leads to decrease in internal resistance between electrodes. A suspended activated carbon particle in electrolyte solution under potential difference could behave like microelectrode when it was positioned near to the graphite electrode (consider as macroelectrode). Electrolysis phenomenon might be favorable due to smaller gradients in the particles themselves compared to those of the macroscopic graphite electrodes [10].

Again the effect of such metal oxides and activated charcoal were also studied using different electrolyte such as 1(N) NaOH. Surprisingly, all experiment came up with better hydrogen production rate and better control over temperature rise than the experiment in absence of such additives. The individual performance of metal oxides and activated charcoal were encouraged to observe their combined effect on water electrolysis. In this regard, electrolysis was carried out with the mixture (50:50) of CuO and ZnO in 1(N) KOH solution. It was interesting to notice that the combined effect was better than individuals, as shown in Table 4. Though the reasoning behind the activity of the mixture of oxides on electrolysis was not clear, still it might be explained by the sensitivity of the oxide mixtures as semiconductor materials and the increased sensitivity to both the Faradaic and non-Faradaic currents generated within the pore of the suspended microparticles. The enhancement of overall performance could be due to the sensitivity of microparticles towards potential difference or simply due to circumstantial variations in microparticle chemistry [11].

The combined effect of oxides on water splitting was almost same but with different magnitude for other electrolytic solution such as 1(N) NaOH. The results are presented in Table 5. Based on this above mentioned performance, it was logical to perform the experiment with the mixture of CuO, ZnO and activated charcoal (2:2:1) in 1(N) KOH electrolytic solution for hydrogen production. Eventually, the experimental results shown in Table 6 reveal both the enhancement of hydrogen production rate and control over rise in temperature. The control over the rise in temperature was achieved almost in every case and best result was noticed using mixture of oxides and activated charcoal. One of the logical explanations might be due to the thermoelectric properties of CuO and ZnO, and in addition this mixture reduces the overpotential loss. The introduction of activated charcoal into the mixture of oxides as additives for electrolyte further improves the results because of the additional microelectrodic effect as explained earlier and this mixtures in the proportion of 2:2:1 (CuO, ZnO and Activated charcoal) appeared as the best catalyst mixture within the present limit of experiments.

In the case of different types of amide (% composition) membranes the results were found to be best for the 75% membrane. One of the prime areas of concern in the case of membranes is the stability factor. The membrane usually got swelled after a few hours of run. Theoretically 90% membrane should also give good results but due to casting problem or composition problem the experiments did not give the desired results. Average hydrogen production rate using membrane electrolyzer was maximum 5.75cc/min using cation exchange resin loaded membrane with 3% Pt on activated charcoal and NiO as electrodes catalysts.Moreover, Table 8 indicates that the rate of hydrogen production was minimum (1.11cc/min) using cation exchange resin loaded 75% amide membrane in absence of catalyst. This clearly indicated that the performance of membrane in water splitting not only depends on the conducting as well as proton exchange properties of membrane rather on the electrode catalyst compositions. This phenomenon could be further justified by comparing the hydrogen production rate using different catalyst compositions.

There was an irregular flow of hydrogen from the PEM electrolyzer due to inside gas hold up that led to intermittent flow of Hydrogen. To minimize this problem, grooving had to be done properly and average hydrogen flow rate was recorded. The irregular gas output was not only because of channeling inside the graphite plate of PEM electrolyzer but due to its chemical interaction with catalysts and this might be best understood by voltage vs time plot which can be termed as cyclic voltammograms. Although the shape of cyclic voltammograms depends on parameters like purity, turn-round and sweep rate, they are highly reproducible. In this voltammograms, a periodic peaks was observed corresponding to an adsorption and desorption of oxygen ions and protons, leading to an oxidation or a reduction of the metal cations on the membrane-electrode surface. The peak current attained a maximum when no gas bubbles were evolved. This might be due to formation of chemisorbed hydride and oxide layers on the electrode surface [12].

Fig. 4 reveals the potential time behavior on the working electrode using 75% amide membrane with 3% Pt on activated charcoal and NiO as electrode catalysts. It was observed that the cell voltage fluctuated with time. Again this may have happened due to the smooth flow of hydrogen from PEM electrolyzer to gas reservoir, and voltage was decreasing due to consumption of power by electrolyzer for hydrogen production. Latter on the voltage was found to increase because of hydrogen gas hold up which subsequently reduced the contact between aqueous solution and catalyst loaded membrane.

Table 1. Effect of CuO on hydrogen production rate and rise in temperature while 1(N) KOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of CuO in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of CuO in electrolyte solution (gm/lit)				
	1	2	3	4	5	1	2	3	4	5
5	4.56	4.57	4.55	4.37	4.29	1	1	1	1	1
10	6.83	6.86	6.85	6.74	6.66	1	1	1	2	2
12	9.57	9.61	9.62	9.53	9.45	1	1	2	3	3
20	11.22	11.32	11.34	11.31	11.28	2	1	2	3	4
24	13.15	13.25	13.35	13.22	13.12	2	2	2	4	5

Table 2. Effect of ZnO on hydrogen production rate and rise in temperature while 1(N) KOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of ZnO in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of ZnO in electrolyte solution (gm/lit)				
	1	2	3	4	5	1	2	3	4	5
5	3.86	3.90	3.91	3.90	3.78	2	2	2	2	2
10	5.33	5.42	5.45	5.41	5.38	2	2	2	3	3
12	7.11	7.31	7.33	7.29	7.22	2	2	3	4	4
20	9.75	9.79	9.81	9.78	9.72	3	3	4	4	5
24	11.63	11.77	11.79	11.69	11.51	4	3	4	5	6

Table 3. Effect of Activated charcoal on hydrogen production rate and rise in temperature while 1(N) KOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of Activated charcoal in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of Activated charcoal in electrolyte solution (gm/lit)				
	1	2	3	4	5	1	2	3	4	5
5	4.87	4.98	5.02	4.88	4.93	1	1	1	1	1
10	7.12	7.35	7.33	7.11	7.11	1	1	1	1	2
12	9.96	10.03	10.10	9.97	10.00	2	1	1	1	2
20	11.77	11.87	11.91	11.85	11.88	2	2	2	2	3
24	13.78	14.11	14.12	14.10	13.98	3	3	2	3	4

Table 4. Effect of (50:50) mixture of CuO & ZnO on hydrogen production rate and rise in temperature while 1(N) KOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of (50:50) mixture of CuO & ZnO in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of (50:50) mixture of CuO & ZnO in electrolyte solution (gm/lit)				
	2	4	6	8	10	2	4	6	8	10
5	5.34	5.36	5.35	5.33	5.32	1	1	1	1	1
10	7.63	7.66	7.67	7.61	7.58	1	1	1	2	2
12	10.45	10.49	10.48	10.43	10.37	1	1	2	2	3
20	12.32	12.38	12.36	12.33	12.30	1	1	2	3	4
24	14.11	14.20	14.18	14.14	14.12	2	2	2	3	4

Table 5. Effect of (50:50) mixture of CuO & ZnO on hydrogen production rate and rise in temperature while 1(N) NaOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of (50:50) mixture of CuO & ZnO in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of (50:50) mixture of CuO & ZnO in electrolyte solution (gm/lit)				
	2	4	6	8	10	2	4	6	8	10
5	4.25	4.34	4.32	4.28	4.23	2	2	3	3	3
10	6.37	6.41	6.37	6.33	6.23	2	2	3	3	3
12	8.22	8.32	8.26	8.21	8.14	3	2	3	4	4
20	10.54	10.53	10.51	10.46	10.41	3	2	4	5	5
24	12.64	12.69	12.62	12.55	12.51	4	3	4	5	6

Table 6. Effect of (2:2:1) mixture of CuO , ZnO and activated charcoal on hydrogen production rate and rise in temperature while 1(N) KOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount of 2:2:1) mixture of CuO , ZnO and activated charcoal in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^{o}C) at different amount of 2:2:1) mixture of CuO , ZnO and activated charcoal in electrolyte solution (gm/lit)				
	2	4	6	8	10	2	4	6	8	10
5	5.45	5.46	5.45	5.47	5.48	1	1	1	1	1
10	7.74	7.76	7.75	7.81	7.82	1	1	1	1	1
12	10.63	10.62	10.63	10.75	10.70	1	1	1	1	1
20	12.57	12.61	12.66	12.70	12.69	2	2	2	1	1
24	14.33	14.34	14.38	14.41	14.42	2	2	2	1	2

Table 7. Effect of (2:2:1) mixture of CuO , ZnO and activated charcoal on hydrogen production rate and rise in temperature while 1(N) NaOH was used.

Applied DC voltage (V)	Hydrogen production rate (cc/min) using different amount (2:2:1) mixture of CuO , ZnO and activated charcoal in electrolyte solution (gm/lit)					Rise in electrolyte temperature (^oC) at different amount (2:2:1) mixture of CuO , ZnO and activated charcoal in electrolyte solution (gm/lit)				
	2	4	6	8	10	2	4	6	8	10
5	4.31	4.36	4.35	4.38	4.37	1	1	2	2	2
10	6.40	6.41	6.39	6. 42	6.43	1	1	2	2	2
12	8.32	8.37	8.36	8.41	8.40	2	2	2	2	2
20	10.60	10.64	10.61	10.65	10.64	2	2	3	2	2
24	12.71	12.72	12.69	12.73	12.71	3	2	3	3	3

Table 8. Effect of different catalysts on membrane water splitting at 30^oC temperature using 12 V DC, 2 amp supply.

Polymer electrolyte membrane (% of Acrylamide)	Avg. hydrogen production rate (cc/min) using NiO as cathodic and anodic catalysts	Avg. hydrogen production rate (cc/min) using 3% Pt in activated carbon as cathodic and anodic catalysts	Avg. hydrogen production rate (cc/min) using NiO and 3% Pt in activated charcoal as anodic and cathodic catalysts	Avg. hydrogen production rate (cc/min) using cation exchange resin as membrane filler	Avg. hydrogen production rate (cc/min) using resin loaded membrane and 3%Pt as cathodic, NiO as anodic catalysts
25	1.01	1.33	1.43	0.85	1.71
50	1.27	1.87	2.22	0.98	2.87
75	2.91	5.69	5.70	1.11	5.75

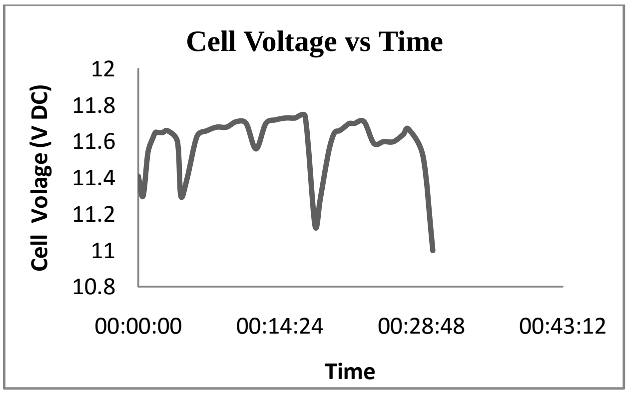

Fig-4: Potential-time behavior at working electrode during cyclic volumetry (75% amide membrane with 3% Pt on activated charcoal and NiO as electrode catalysts).

4. CONCLUSION

It was found that under certain operating parameters, the mixture of oxides (CuO and ZnO) contributed to increased hydrogen production rate as well as control of the rise in temperature of elelctrolyte (KOH) for water electrolysis to generate hydrogen. Further, introduction of activated charcoal reported improved role of oxides mixture on electrolysis in terms of hydrogen production rate and overpotential loss. In this paper, based on detailed performance of ion exchange membrane water splitting using catalytic amide membrane of three compositions. 3% Pt on activated charcoal as cathodic and NiO as anodic catalysts using cation exchange resin loaded, 75% amide membrane composed of PVOH matrix with poly (acrylic acid –co-acrylamide) appeared as the best one within the present limit of investigation.

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BIOGRAPHIES

Biswajit Mandal is working as assistant professor in Chemical Engg. Department at Haldia Institute of Technology for seven years. Email Id: **bmandal_1977@rediffmail.com**

Professor Amalesh Sirkar is one of the renowned researcher on coal liquefaction process is working at HIT for 13 years. He owned seven patents on different fields including coal liquefaction process. Email Id: **sirkar_a@hotmail.com**

Parameswar De is Professor in the Department of Chemical Engineering at the University of Calcutta. He has engaged himself in teaching and research for over 30 years. His research areas include mass transfer & hydrodynamics in multiphase flow, biochemical & electrochemical engineering and reaction engineering. He has published papers in national and international journals. Email Id: **parameswar_de@rediffmail.com**

Dr. Avijit Ghosh, received his PhD degree from IIT Guwahati and M.Tech from Calcutta University. His research interest in the energy conversion device, graphene synthesis and its application, fuel cell. Presently, he is an assistant professor in Haldia Institute of Technology, Kolkata. His research paper is published in high impact factor peer review journals including fuel cells, international journal of hydrogen energy.