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Fabrication and characterization of printed zinc batteries

M. A. Nazri[1], Anis Nurashikin Nordin[2], L. M. Lim[3], M. Y. Tura Ali[4], I. Mansor[5], M. I. Suhaimi[6], R. Othman[7], S. R. Meskon[8], and Z. Samsudin[9]
[1,2]Department of Electrical and Computer Engineering, Kulliyyah of Engineering, International Islamic University Malaysia (IIUM), Malaysia
[3,4,5,6,9]Manufacturing Technology and Innovation (MTI), Jabil Circuit Sdn. Bhd., Penang, Malaysia
[7,8]Department of Science, Kulliyyah of Engineering, International Islamic University Malaysia (IIUM), Malaysia

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ABSTRACT

Zinc batteries are a more sustainable alternative to lithium-ion batteries due to its components being highly recyclable. With the improvements in the screen printing technology, high quality devices can be printed with at high throughput and precision at a lower cost compared to those manufactured using lithographic techniques. In this paper we describe the fabrication and characterization of printed zinc batteries. Different binder materials such as polyvinyl pyrrolidone (PVP) and polyvinyl butyral (PVB), were used to fabricate the electrodes. The electrodes were first evaluated using three-electrode cyclic voltammetry, x-ray diffraction (XRD), and scanning electron microscopy before being fully assembled and tested using charge-discharge test and two-electrode cyclic voltammetry. The results show that the printed ZnO electrode with PVB as binder performed better than PVP-based ZnO. The XRD data prove that the electro-active materials were successfully transferred to the sample. However, based on the evaluation, the results show that the cathode electrode was dominated by the silver instead of $Ni(OH)_2$, which leads the sample to behave like a silver-zinc battery instead of a nickel-zinc battery. Nevertheless, the printed zinc battery electrodes were successfully evaluated, and more current collector materials for cathode should be explored for printed nickel-zinc batteries.

Corresponding Author:

Anis Nurashikin Nordin
Department of Electrical and Computer Engineering, Kulliyyah of Engineering
International Islamic University Malaysia
Jalan Gombak, 53100, Selangor, Malaysia
Email: anisnn@iium.edu.my

1. INTRODUCTION

In today's world of the internet for everything, personalized wearable devices have a large market share of the overall demand in electronic devices. This market is expected to grow over USD 80 million by the year 2024, driving researchers and industries to pursue the development thin, flexible energy storage devices that can meet the requirements of portable wearable devices [1], [2]. These devices have to be small and portable, while still embedding a multitude of features. Most of the small and portable electronics are still powered by coin cell batteries, or conventional bulky and heavy AAA batteries, which limit the miniaturization of the devices [3]. In addition to this, conventional batteries are not flexible, thick and cannot fit into thin and smaller devices such as smartcards. Currently, the most popular battery is lithium ion [4]-[7], however, this type of battery is not environmentally friendly due to few factors. Firstly, lithium ion batteries are highly flammable and may cause explosions in high pressure environments. Their materials are highly

reactive and toxic [8]. Zinc batteries, on the other hand, are safer, its material is abundant, cheap, and environmentally friendly despite lower their lower energy density, cycle life, and nominal voltage. However, there are still issues such as zinc corrosion, zinc dissolution, and zinc passivation [9]. Recent development shows that zinc battery lifecycles can be prolonged through the introduction of additives such as Bismuth (III) Oxide, Bi_2O_3 to the electrode and electrolyte [10]. This additive basically enhances the electrochemical performance and also improves the electrical conductivity of the zinc electrode.

Printing is an older manufacturing method compared to modern lithographic fabrication, but is advantageous due to its lower fabrication costs, high throughput while maintaining precision and accuracy [11]. Printed battery is a battery with at least one of its components fabricated using printing technologies. There are several examples of printed batteries being developed such as a stretchable silver-zinc battery based on a nano-wire current collector [12] and a stretchable Zn-Ag_2O rechargeable battery printed on a thermoplastic polyurethane (TPU) substrate [13]. Typically, a printed battery consists of 5 major elements namely substrate, current collector, anode, cathode, and electrolyte. Each element plays a significant role in forming a good battery and must be selected carefully. The current collector layer acts as the terminal for the battery since it allows electron flow. It is commonly printed using silver [13] or carbon inks [14]. Silver inks have significantly higher conductivity compared to carbon inks, but carbon inks can provide better electrochemical stability for electrolytes. Another key component of the battery is the substrate which acts as the platform where all the printed battery's elements are placed upon. Selection of the substrate is dependent on the battery's application, whether it should be integrated with other components or should be separated as an independent device. Common substrates used for printed batteries are polyethylene terephthalate (PET) for flexibility [15], [16], thermoplastic polyurethane (TPU) for stretchability and fabrics for clothing. The selected substrate must also be able to withstand high temperatures in most of the cases due to the curing condition of the current collectors and electrodes can reach up to around 135°C.

Electrodes are the core elements of the battery that determine the capacity of the battery, operating voltage, recharge-ability, life cycle, etc. There are two electrodes: anode and cathode, which act as negative and positive terminal, respectively. Depending on which electrochemical system chosen for the battery, the selection of the electrodes will be done accordingly. For example, Ni-Zn battery uses nickel hydroxide as the cathode, while zinc oxide as the anode [17]. Meanwhile, for silver-Zn battery, the anode is ZnO, whereas the cathode is silver [12]. The current collector allows the electron to flow across load or power source, the electrolyte, on the other hand, allows ionic flow between anode and cathode to complete the electrochemical system. The electrolyte used for Ni-Zn and other zinc battery systems such as the Ag-Zn system is potassium hydroxide (KOH) [18], with molarities between 3M to 8M [17]. The electrolyte may be applied to the battery in many forms, such as aqueous [19], gel electrolyte [20], solid-state [21], etc.

For this work, we describe the fabrication and characterization of a nickel-zinc battery with nickel hydroxide and zinc oxide electrodes. Nickel-zinc battery has potential to be developed as a printed battery, due to its higher nominal voltage of 1.7 V compared to 1.2 V for NiMH. It is also suitable for high current devices due to its fast recharge capability and good specific energy [22]. This paper will be study the effect of using two different binders, which are polyvinyl butyral (PVB) and polyvinyl pyrrolidone (PVP), on the performance of printed nickel-zinc battery via cyclic voltammetry and charge-discharge analysis. Other characterization methods such as SEM and XRD were also done in order to evaluate the surface morphology and to validate the presence of the active material within the printed electrode. Section 2 of this paper will explain the materials selection and the experimental setup for this research. Section 3 displays all the results based on the experiment done. Then, the result will be thoroughly discussed in section 4. Finally, the insight for further development of printed Ni-Zn battery is suggested in section 5.

2. RESEARCH METHOD

The three dimensional view of the battery is shown in Figure 1(a). The lateral design was chosen implementation of this battery as it has less risk of the short circuit and it relatively easier to be assembled as compared to the sandwich approach [14]. The electrode's top view and dimensions are shown in Figure 1(b). The cross-section of the device is shown in Figure 1(c) where PET substrate, silver current collector, zinc oxide anode, nickel hydroxide cathode and potassium hydroxide (KOH) electrolyte. The snapshot of the electrodes and current collector printed on the PET substrate is shown in Figure 1(d).

The raw materials for the printed battery electrodes were mixed and printed on printed silver current collector using screen printing technique [23]. A few sets of samples were fabricated and dried accordingly to be used for both half-cell and full-cell electrode evaluation. 3-electrode-cyclic voltammetry was used to study each of both half-cell electrodes electrochemical performance using graphite as the counter electrode, Hg/HgO (1M NaOH) as the reference electrode, whereas the working electrode was changed depends on which electrode to be studied. In this project, two different printed ZnO anode were studied by using

different types of polymeric binder, which are PVB and PVP. Both printed ZnO anode were printed on printed silver current collector (CI-1036) to be evaluated and compared to select the best out of the two electrodes. Meanwhile at cathode, the PVB-based printed $Ni(OH)_2$ electrode was printed on printed silver current collector (CI-1036) and the CV data was evaluated and recorded. The finalized version of the printed battery electrodes was then sent for SEM and XRD to observe the morphology of the electrodes and to validate the presence of the active materials on the printed electrode. Once the electrochemical performance of each electrode were studied, a new fresh printed battery sample was assembled and sealed to evaluate its actual performance using charge-discharge analysis and the redox peak of the complete printed battery sample was measured using the two-electrode cyclic voltammetry. All the data collected was analysed and discussed in the next section.

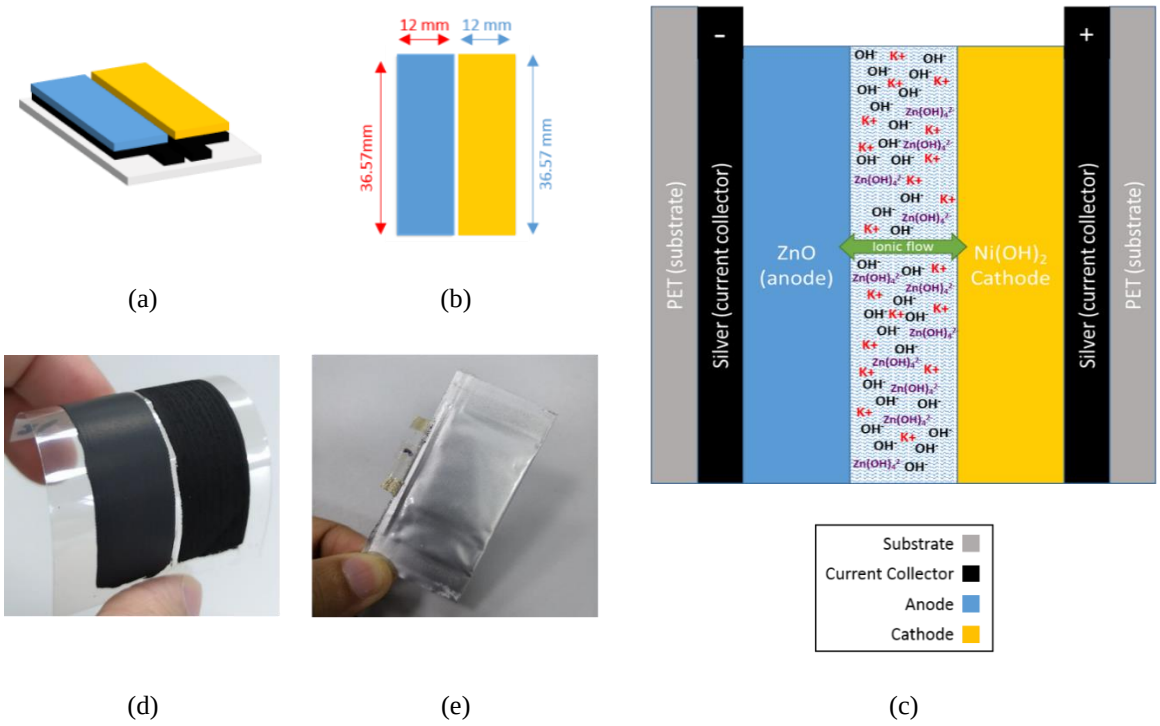


Figure 1. Clockwise from left, (a) 3D view of lateral design of the printed battery, (b) Top view of the electrode and its dimensions, (c) Printed Ni-Zn battery cross-section, (d) Fabricated electrodes and current collector on PET, (e) Fully-sealed printed battery

2.1. Materials

The PET substrate was recycled from conductive ITO film substrate (ITO400, 150µm), purchased from NejiLock, Singapore. Silver ink (CI-1036) purchased from engineered conductive materials, USA was printed on PET to form the current collector for both electrodes. The anode electrode was evaluated by varying its binder materials using either PVP or PVB. All other elements were fixed for both electrodes. Two sets of anode ink were mixed separately using two different binders, which are PVB (P110010) and PVP (average molecular weight 360000 g/mol, PVP360) purchased from Sigma-Aldrich, UK. The other components of the anode are Zn (particle size 1-5µm, GF09783018, Aldrich), ZnO (particle size<5µm, 205532, Sigma Aldrich), Bi_2O_3 nano-powder (particle size: 90-210nm, 637017 Aldrich) all purchased from Sigma-Aldrich, UK, Super P conductive carbon black (TIMCAL) purchased from KGC Resources, Malaysia and 1-ethoxy-2-propanol (25905, ACROS) purchased from Fischer Scientific, Malaysia. The composition of of the formulation is detailed in Table 1.

The cathode nickel (II) hydroxide electrode used PVB (P110010) purchased from Sigma-Aldrich, UK as its binder. The cathode ink comprised of $Ni(OH)_2$ (average particle size: 5.2µm, 283622 Aldrich), Zn (GF09783018) both purchased from Sigma-Aldrich, UK, conductive carbon black (Cabot Black Pearl 2000), purchased from Cabot China, and 1-ethoxy-2-propanol purchased from ACROS, Malaysia as the solvent. These items were mixed with PVB binder. Details of the cathode formulation is shown in Table 1.

Table 1. Electrode ink material composition

Electrode	Materials	Weight (g)	
		PVB-based	PVP-based
Anode	ZnO	2.922	2.922
	Zn	1.402	1.402
	TIMCAL SP	0.292	0.292
	Bi_2O_3	0.5845	0.5845
	PVB	0.3507	0
	PVP	0	0.3507
	1-ethoxy-2-propanol	3	5
Cathode	$Ni(OH)_2$	4.910	4.910
	Zn	0.292	0.292
	Cabot BP2000	0.292	0.292
	PVB	0.3507	0
	1-ethoxy-2-propanol	5	9

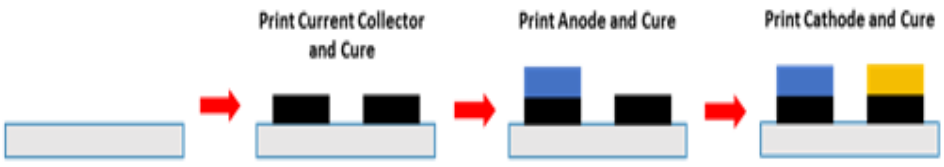
2.2. Fabrication methods

The solid powders (zinc, zinc oxide, carbon black and Bi_2O_3) were dry-mixed for 1 minute using ARE 310 planetary centrifugal mixer at 2000 rpm. All the electrode inks were mixed using. In a separate container, the solvent and binder were mixed at intervals of 20 minutes with 20 minutes rest in between. Next, this mixture is combined with the powder mixture and further mixed for 20 minutes. Then, the ink is ready to be printed. The same mixing procedure is used for both binder sets of anode and cathode.

The silver ink was manually screen-printed on PET and dried in Contherm Thermotec 2000 batch oven at 120°C for 30 minutes. The screen mesh used is a polyester mesh with a mesh count of 70 th/inch. Similar mesh size was used for both anode and cathode printing. Next, the PVB-based ZnO anode ink was screen-printed on one of the current collectors and dried in the oven at 100°C for 1 hour. Once the printed anode was dried, the PVB-based $Ni(OH)_2$ cathode ink was printed on the other side of the current collector and dried with similar temperature and time settings. The printing and drying steps were repeated for PVP-based anode and PVB-based cathode ink. The overall electrode printing and drying process are illustrated in Figure 2.

The printed anode and cathode are next cut using scissors and separated. A Whatman filter paper with a pore size of 11 µm and a thickness of 180 µm is used as a separator to avoid the anode and cathode shorting each other. Next, the three layers, anode, separator and cathode are assembled together and placed in a battery-grade aluminum pouch case. The aluminium pouch is then partially seaded and injected with the aqueous 4M potassium hydroxide (KOH) that was mixed with ZnO until saturation. Next, the pouch is fully sealed and is now ready for further evaluation. The pre-assembled electrode and fully sealed printed zinc battery is shown in Figure 1(d) and Figure 1(e). Several samples were produced for testing.

(a)



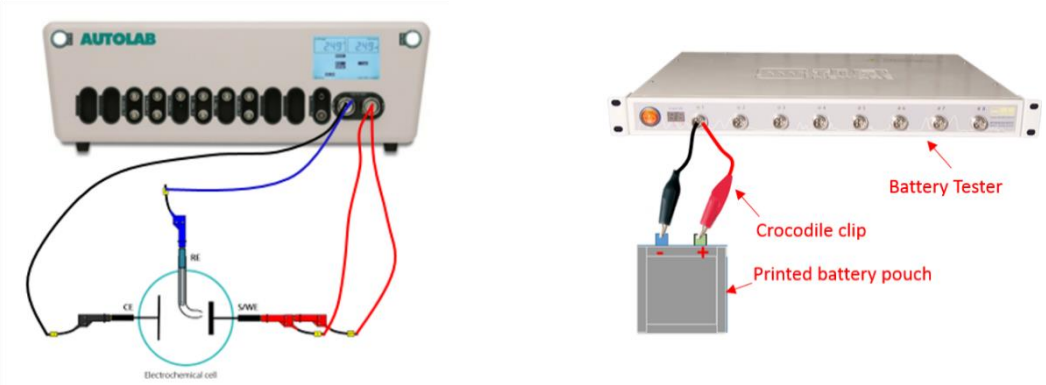
(b)

Figure 2. These figures are, (a) Top view of anode and cathode, (b) The electrode printing process. First, the silver current collector was first screen printed and dried in the oven, Next, the ZnO anode was printed on one of the silver current collectors. The second current collector is covered with nickel (II) hydroxide

2.3. Experimental setup for electrochemical characterization and battery testing

Prior to assembly and packaging, each pair of the electrode was tested using half-cell cyclic voltammetry (CV) [24] in order to validate its redox reactions. Half-cell CV measurements were made using the three-electrode setup using an Autolab potentiostat (PGSTAT 302N) as shown in Figure 3(a). The reference electrode (RE) is Hg/HgO (1M NaOH) and counter electrode (CE) is a graphite rod. Three different working electrodes (WE) were tested, printed PVB-based ZnO, PVP-based ZnO, and PVB-based $Ni(OH)_2$. The electrodes are immersed in 4M KOH+ZnO electrolyte and the CV was run at a scan rate of 10mV/s. Different potential windows were used for different working electrodes namely i) -2V to -0.5V for ZnO [25], ii) -0.2V to 0.6V for $Ni(OH)_2$ [26]. Next, scanning electron microscope (SEM) and x-ray diffraction (XRD) analysis were performed in order to validate the presence of ZnO and $Ni(OH)_2$ on the electrodes as well as to observe the morphology of the printed electrode. The electrode's surface morphology was captured at 10000x for ZnO and 1000x for $Ni(OH)_2$..

Once the individual electrodes are proven to be satisfactory, the electrodes are packaged in a pouch to form a battery as shown in Figure 1(e). Next, to test the functionality of the battery, two-electrode CV was performed by using the anode as the WE, while RE/CE is connected to the cathode using the potentiostat. The same electrolyte and scan rate were used for this test with the scanning window set to 0V to 2V. This scanning potential window was fine-tuned until the best redox peak is obtained. Next, charge-discharge analysis was conducted in order to evaluate the performance of the printed zinc battery using a battery tester (Neware BTS4000-5V20mA-8CH). The negative terminal was connected to the current collector with the ZnO electrode, while the positive terminal is connected to the current collector with $Ni(OH)_2$ electrode as shown in Figure 3(b). Another charge-discharge analysis was done by reducing the amount of the binder by 50% for both electrodes. This is to study the effect of reducing the binder amount on battery performance.



(a) (b)

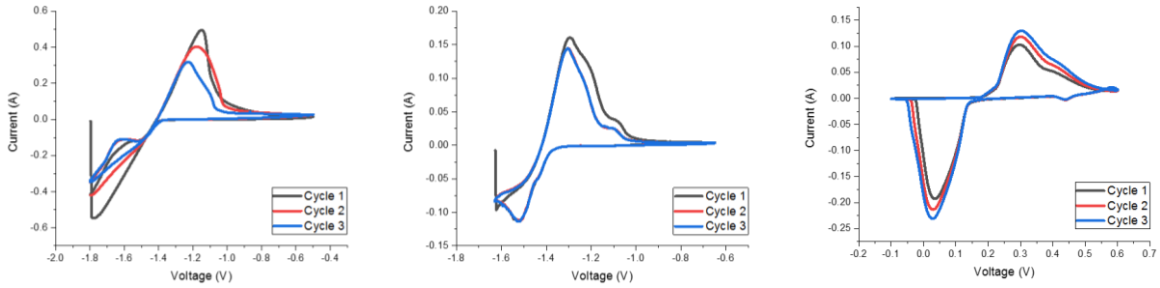
Figure 3. These figures are, (a) Three-electrode setup for cyclic voltammetry for printed electrodes half-cell redox reaction analysis, (b) Experimental setup for charge-discharge test using battery tester

3. RESULTS AND DISCUSSION

The half-cell measurement results using the three-electrode setup for three different andoes: PVP-based ZnO, PVB-based ZnO and PVB-based $Ni(OH)_2$ are shown in Figure 4. The CV curve for PVP-based ZnO as shown in Figure 4(a) shows a less pronounced reduction peak as compared to PVB-based ZnO as shown in Figure 4(b). This shows that the printed ZnO electrode with PVB as binder performed better than PVP-based ZnO. The CV curve of PVB-based ZnO is also more stable since cycle 2 and 3 are consistent. The PVB-based $Ni(OH)_2$ as shown in Figure 4(c) also shows pronounced redox peak current. Thus, both PVB-based ZnO and $Ni(OH)_2$ can be used for printed zinc battery development.

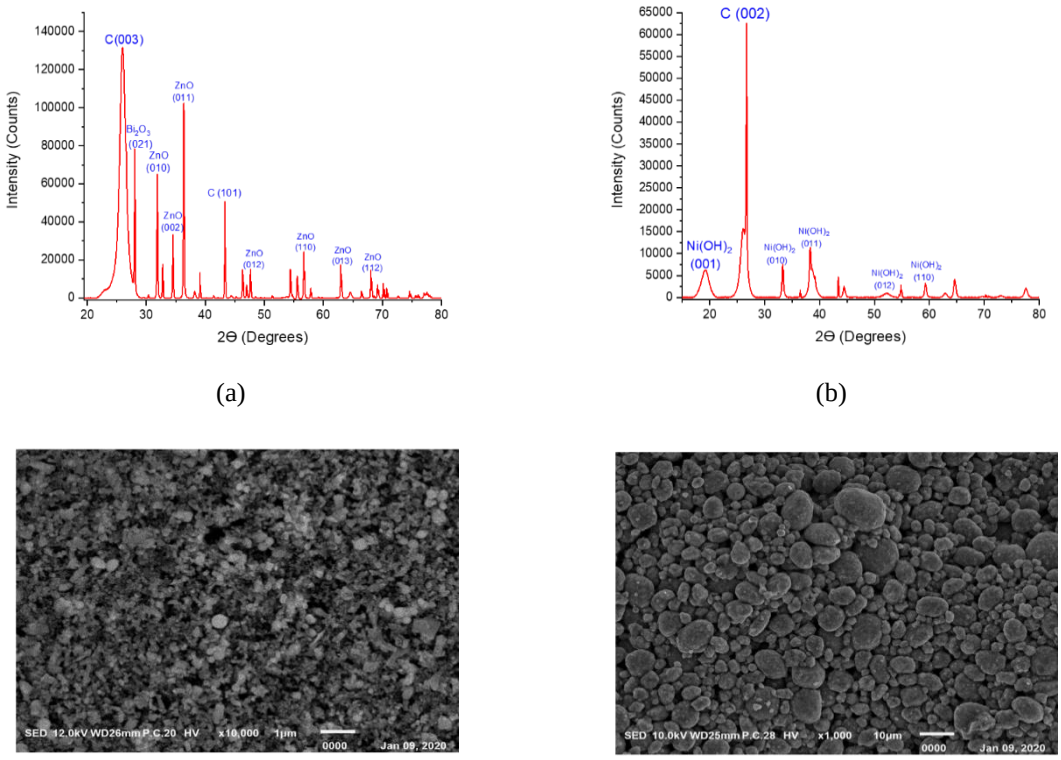
The next experiment performed was XRD and scanning electron microscopy (SEM) for the ZnO and $Ni(OH)_2$ electrode samples. Figure 5(a) shows the XRD measurement results for the anode which indicates the presence of ZnO and carbon peaks. The XRD performed on the cathode shows distinct $Ni(OH)_2$ peaks that can be seen in Figure 5(b). This shows that the electro-active materials were successfully transferred to the sample. SEM image illustrated in Figure 5(c) show that the ZnO anode has smaller particle sizes which are less than 1 um in size. The $Ni(OH)_2$ SEM image shown in Figure 5(d) show much larger particles with average sizes of 5 µm. This indicates that the ZnO has higher surface areas compared to the cathode. The structure of anode materials is also crucial for longevity of battery cycle life. Utilization of

nanoscale zinc oxide could increase the contact surface area between electrode and electrolyte thus improving the efficiency of the electrode, its volumetric capacity, and provides enhanced cycle stability [19].



(a) (b) (c)

Figure 4. The half-cell reaction cyclic voltammogram for, (a) PVP-based ZnO anode, (b) PVB-based ZnO anode, (c) PVB-based $Ni(OH)_2$ cathode. Each of the anodes were printed on a silver current collector



(a) (b)

(c) (d)

Figure 5. These figures are, (a) XRD measurement results for anode ZnO, (b) SEM image of anode ZnO, (c) XRD measurements for nickel hydroxide cathode, (d) SEM image for nickel hydroxide cathode showing the larger grain sizes compared to ZnO

Next the electrodes are packaged in the pouch cell and battery functionality tests are conducted. The charge discharge analysis was first performed using the battery tester and its results is as shown in Figure 6. At least 5 consistent cycles with nominal voltage of 1.56V can be observed. The discharge capacity at the 2nd cycle was recorded as 1.3407 mAh or equivalent to 0.153 $mAh.cm^{-2}$. The coulombic efficiency at the 2nd cycle is 94.71% which can be obtained by calculating the discharge capacity over the charge capacity.

Despite having a fairly good charge-discharge curve, it was observed that after several cycles, the cathode current collector turns to black, while the separator turns yellow as shown in Figure 7(a). The printed battery lifetime also ends at this point. It is hypothesized that the current collector turns black is due to a chemical reaction which causes silver (white) to change into silver oxide (black) when exposed to the electrolyte. The yellow stain at the separator shows the presence of silver hydroxide in the electrolyte. The stain occurs when silver hydroxide dissolves into the electrolyte and then is stuck at the separator. To study this phenomenon further, an electrochemical study was done using three-electrode CV or half-cell reaction using an electrode covered only with silver ink. The CV results are shown in Figure 7(b). When comparing Figure 7(b) to Figure 4(c), both curves look similar. This confirms instead of a Ni-Zn battery; the device performs as an Ag-Zn battery. This is probably due to the fact that silver is more dominant and has higher tendency to reduce as compared to $Ni(OH)_2$. The behavior of zinc silver is also confirmed by looking at the nominal voltage of the charge-discharge curve, which is between 1.5V-1.6V as mentioned before.

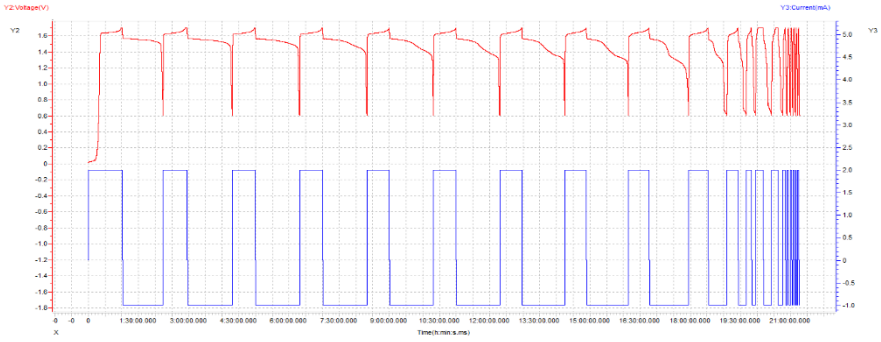
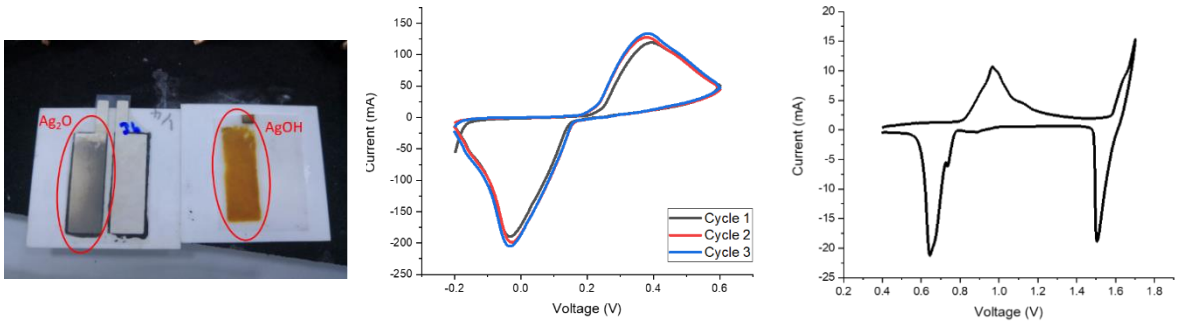


Figure 6. Charge discharge curve of printed zinc battery

The full cycle, two-electrode cyclic voltammetry with ZnO as its anode and $Ni(OH)_2$ as its cathode was also conducted and its results are shown in Figure 7(c). It can be seen from the CV results that redox reaction occurs between the pair of electrodes. The peak current is observed between 0.4V to 1.7V, which confirms that the developed printed battery is functioning and matches the behaviour of zinc silver battery.



(a) (b) (c)

Figure 7. These figures are, (a) Image of the silver current collector turns to silver oxide (black) and silver hydroxide (yellow), (b) Half-cell reaction cyclic voltammogram of silver, (c) Full-cell reaction (two-electrode) cyclic voltammogram for printed zinc battery

4. CONCLUSION

A printed zinc battery was successfully fabricated and tested. The electrodes were formed using silver ink with either zinc or nickel hydroxide. Two different binders were tested when mixing the ink, namely PVB and PVP. It was found that the PVB binder delivered better electrochemical performance than PVP at the anode. XRD and SEM measurements were obtained for both electrodes to determine their material composition and surface area. The functionality of the printed battery was verified using cylic

voltammetry (both two and three electrode) tests as well as charge-discharge analysis. The nominal voltage recorded for the developed printed zinc battery is 1.56V with a discharge capacity of 1.3407 mAh, which also equivalent to 0.153 $mAh.cm^{-2}$. Based on the experiments, it was found that silver ink is not a suitable current collector for $Ni(OH)_2$ as a printed battery cathode as it will dominate the electrochemical reaction at the cathode due to its high reduction potential. Alternatively, printed current collector based on stainless steel or a high and inert carbon-based current collector such as graphene can be considered for future development.

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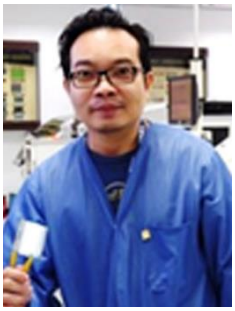
BIOGRAPHIES OF AUTHORS

Muhamad Aiman Nazri is an MSc. candidate in International Islamic University Malaysia. He received his bachelor degree in Mechatronics Engineering, International Islamic University Malaysia (IIUM) in 2017. He is also pursuing his Masters in Electronics Engineering in IIUM and has more than 3 years of industrial experience. His research interests are related to printed electronics and printed zinc batteries design and fabrication.

Anis Nurashikin Nordin is a Professor in Electronics Engineering Department, International Islamic University Malaysia. She has a BS degree in Computer and Information Engineering from International Islamic University Malaysia in 1999. She obtained both MS and PhD degrees in Computer Engineering from the George Washington University, Washington, DC, in 2002 and 2008, respectively. Her research area covers personalized healthcare devices, MEMS, BioMEMS, SAW devices, printed electronics, and microfluidics.



Lai Ming Lim is the Principal Engineering Services Engineer of the Manufacturing Technology & Innovation Group at Jabil. He has more than 25 years of experience in industry with expertise in printed electronics, advanced assembly, rework development, etc.

Idris Mansor is the Lead Services Engineering Engineer attached to Manufacturing Technology & Innovation Group at Jabil. Specializes on the advanced assembly particularly on SMT related process development/ characterization and deployment, 3D MID, ACA alternative solution, advanced rework of the miniature packages up to the large packages development and critical BU support. He has almost 27 years field experience and majority of the years spent in EMS. 7 years attached with Cisco system as Project Manufacturing Lead managing SEA Contract Manufacturing, global collaboration with Product Operations team i.e. USA, Canada, Italy, Holland, Japan, India & China. Has experience as a Mechanical Designer in automotive industry Proton car rear light, also as a M&E Consultant on construction of Gurney Plaza & Prangin Mall in Penang.

Mohd Yusuf Tura Ali is the Professional Engineering Services Engineer of the Manufacturing Technology & Innovation Group at Jabil. He has more than 20 years of experience in industry with expertise in Advanced Assembly and he is an SMT equipment specialist.

Muhammad Irsyad Suhaimi is an Associate Engineering Services Engineer of the Manufacturing Technology & Innovation Group at Jabil. He graduated from the International Islamic University Malaysia (IIUM) with Bachelor of Mechatronics Engineering in 2017, and he is currently pursuing his Masters in Electronics Engineering in IIUM as well. He has more than 3 years of experience in industry with expertise in printed electronics, Advanced Assembly, and piezo electronics.

Raihan Othman completed his tertiary studies at the University of Malaya. He did his BSc in Physics. His MSc project was on plasma enhanced chemical vapour deposition (PECVD) silicon nitride. He later worked on zinc-air cell for his Ph.D. He joined IIUM in 1997 as a matriculation teacher and currently, is an academic staff of the Department of Science in Engineering, Kulliyyah of Engineering, IIUM. He served the faculty as the Deputy Dean (Student Affairs) from 2003 until 2008. He is currently the Head, Department of Science in Engineering. Dr. Raihan and his co-workers have patented their i nnovative work on MCM-41 as separator for electrochemical cells, and, Zn/MCM-41/air cell as humidity sensor.

Shahrul Razi Meskon received his B.E. and M.Sc. in Materials Engineering from International Islamic University Malaysia in 2008 and 2011, respectively. Also, he received his Ph.D. in Engineering, specializing in rechargeable Ni-Zn thin-film battery, from the same university in 2018. His research interests include electroplating, printed electrode, electrode analysis, and battery separator. He is currently working in a printing company. He may be reached at: s_razi1803@yahoo.com

Zambri Samsudin is the Senior Advanced Technology Manager of the Manufacturing Technology & Innovation Group at Jabil. He is heading the Asia team developing Strategic Capabilities to support the USD25.3B enterprise. His team is deploying innovations (adhesives, advanced assembly, and printed electronics which includes the flexible as well as the stretchable electronics) for the scalable manufacturing of medical, industrial, energy, consumer, aerospace, automotive, and defense products. Graduated from Swansea University with Bachelor of Engineering (Mechanical), Zambri is a technology, operations, sourcing, and business solutions strategist for Jabil. With more than 29 years of global experience in electronics manufacturing, new process development, material handling, automation, additive manufacturing, and printed electronics, Zambri leads business and technical strategic initiatives for the company. He has demonstrated ability to develop & execute management and engineering strategies at discrete and enterprise level, over a wide span of technical challenges and commercial initiatives.