A Review on Know-How Technologies in the Preparation of Paper Battery

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Received 11.02.2021, received in revised form 13.12.2021, accepted 18.01.2022
10.47904/IJSKIT.12.3.2022.15-21

***Abstract*- New types of thin film batteries made from bio-degradable, versatile, man-made sources along with adequate physical properties such as electronic conductivity, inter-diffusion coefficient and better energy storage capacity. Such properties are needed to replace the current technology, based on graphite as anode material as well as LiPF6, LiMnO4, LiCoO2 as electrolyte which are non-biodegradable and scarce elements. Earlier super-capacitors were discovered with higher capacitance but they had low energy storage. Therefore, modern technologies require fast charging as well as long lasting systems which are provided by paper battery. A paper battery is an advanced form of lithium-ion battery, prepared by depositing components such as single walled carbon nanotube over the cellulose substrate. This review paper focuses on the different deposition techniques as well as the properties of different components used in the paper battery. One of the most promising materials showing charge detaining properties in paper batteries is single walled carbon nanotube. Single walled carbon nanotube as the anode material, electrolyte as urea/sweat/blood which is non-toxic, bio-degradable as well as cathode in the form of lithium metal incorporated with single walled carbon nanotube are a primary constituents of a paper battery. This study also reviews the methods for construction of various constituents of a paper battery and discusses the various technologies involved.**

Keywords- paper battery, thin film battery, super capacitors, and electrochemical battery

1. INTRODUCTION

There is an increasing requirement of thin, light weight, flexible and low cost storage device due to rapid demand in electronic devices. Several types of battery technologies have been developed so far to store energy; out of which paper battery met the challenges which are developed by Stanford University in 2009. Paper battery as shown in Fig. 1 is the thinnest, light weight and flexible energy storage device, made by combining conventional sheet of cellulose-based paper and carbon nanotubes [1- 6]. It is considered to be consisting of a multiple number of cells in series, thus adding or cutting of the battery affects its output voltage [1]. The paper battery is a better alternative to the traditional battery system. It is a form of lithium ion battery where shorter length metallic single walled carbon nanotube is used in order to increase its efficiency. This feature of paper battery helps in its application in hybrid vehicles, long lasting mobile phones and other portable electronic devices.

The paper battery consists of cathode, made up of carbon nanotube fabricated over lithium metal surface, and the anode of short length metallic single walled carbon nanotube (the length of single walled carbon nanotube lying between 100-200 nm) [2]. A paper battery is flexible as well as thin shaped (thickness of 0.5-0.7 mm) energy storage device [3], made by integration of cellulose paper and carbon nanotube.

Cellulose is an organic matter, obtained from natural resources such as, from the pulp of the leaves and stems and available as paper in the market. Moreover, the use of cellulose as a covering element for paper battery could be because of it's following properties. (i) Cellulose allows the flow of charge through capillary action without any external pump. (ii) It has low shear stress and high tensile strength characteristics. (iii) Cellulose provides better film adhesion when compared with the plastics [3]. (iv) Cellulose can be recycled and reused. (v) Cellulose can be easily decomposed into the soil because of its non-toxicity. However, cellulose itself cannot conduct the flow of charge, hence it has got pores in the form of electronic pathways (due to its hydrophilicity) which is occupied by conductive fluids (in this case it is carbon nanotube ink) to transfer charges [4].

Carbon nanotube ink is considered as an adsorbent over other porous material because of its following properties. (i) Carbon nanotubes show fewer losses of conductor's electrons, even more than some metal conductors like copper and silicon. (ii) Carbon nanotube shows lesser anodic expansion and contraction than silicon, during charging and discharging. Hence, there is no problem related to cracking of solid electrolyte interface layer. (iii) Carbon nanotubes are manufactured within the scales varying from nanometers to micrometers, with weight on the nanometric scale, and it shows tensile properties similar to the bulk of steel. (iv) Single walled carbon nanotube has tensile strength of 30-45 GPa [3, 5]. (v) Single walled carbon nanotubes with helical defects show higher tensile strength (i.e. 79.9

GPa), it is because of the presence of MV defects in single walled carbon nanotube [5].

Carbon nanotube ink is prepared from shorter length single walled carbon nanotube and it is spread over the cellulose surface, in order to get a continuous and fast diffusion of charges. Moreover, surface adsorption is carried out via a pattern formation technique like wax printing [3], which creates sections of fluidic pathways on the paper to continue the capillary action of the electrolytes. The wax printing requires printing of a solid wax ink layer over the cellulose substrate. Then heating the sample for few minutes, where the time of heating is dependent on the substrate used. In order to form hydrophilic volume over the filter paper, two minutes is sufficient for the wax to penetrate the cellulose substrate when it is heated to 100°C [6, 7]. This widens the surface area of the substrate, consequently increasing the conductivity of the paper battery. This is because of the increase in a number of occupation sites for Li^+ ion diffusion over the adsorbed surface of the cellulose. Additionally, researchers have observed that on printing carbon nanotube over the paper, the paper thickens as carbon nanotube behaves as an extra layer over the paper surface, this decreases the working length and thereby reducing the weight of the paper battery by 20%.

The carbon nanotube gives the property of increasing surface area of the cellulose, where carbon nanotubes interact with the molecules at the surface of cellulose, dividing each molecule into multiple numbers (usually twice the original one). The lithium ion travel through the surface with hoping "knock off" mechanism. When there are large numbers of molecules, the occupation sites for the Li^+ ions increases, leading to crowding of ions and, hence transport of ions is significantly increased. Therefore a battery of lesser lithiation time can be modeled.

Apart from the advantages of single walled carbon nanotube, it is difficult to be used in a battery, which is designed for commercial use as the preparation of single walled carbon nanotube is an expensive process [8-10]. Single walled carbon nanotube ink adsorbed over the cellulose substrate, is used as the medium for the diffusion of charges in a paper battery. As compared to other anode materials like graphite, silicon, lithium, single walled carbon nanotube is a newer technology, therefore, much refinement is required in properties such as diameter, number of layers, electronic properties, which are essential for the development of single walled carbon nanotube based paper battery. Broad changes in the voltage as well as lack of voltage plateau while discharging are the problems associated with the battery, when single walled carbon nanotube is used over graphite as an anode. Moreover, a fraction of Li^+-ion gets consumed gets consumed instead of getting stored [11]. Cellulose gets torn out under pressure, therefore paper battery can be easily damaged as compared to the batteries having metallic base metal [8-10]. The paper battery is also hazardous to human health, as because when it is inhaled it interacts with the microphages present in the lung which is similar to asbestos fibers [10]. The Paper battery is a kind of flexible energy storage system. It is because of its property of multiplying or dividing its output voltage, paper battery can be used in small appliances such as wrist watches, Bluetooth speakers, pacemakers of heart where approximately 1.5 V of potential difference is required as well as in hybrid cars, electric rickshaws, electric scooters where 200-250V of power is required [8, 12].

A battery basically consists of cathode, anode, electrolyte, separator and current collector, where component material for one kind of battery is different from another one. Following material components are required for non-scattering flow of charges, high power capacitance as well as better energy storage:

- Shorter length metallic single walled carbon nanotube is used as an anode [11].
- Lithium metal incorporated with carbon nanotube acts as a cathode. Electron beam lithography is used for patterning the cathode [13].
- Bio electrolytes like urine, sweat, blood are used.
- Cellulose in the form of paper is used as the separator [1].

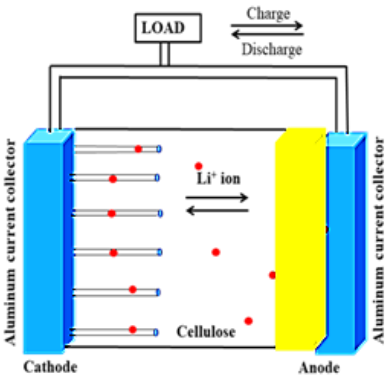


Fig. 1 Schematic of a paper battery [11]

2. CARBON NANOTUBE

Carbon nanotubes are basically one-dimensional structure forming a hexagonal network of carbon atoms belonging to sp^2 bonding family [14]. Carbon nanotubes can be prepared either through chemical vapor deposition method or electric arc discharge method or laser ablation method. Chemical vapor deposition method is found to be an effective method for synthesizing various forms of carbon nanotubes as per literature [15-17]. The length of carbon nanotube can be of the order of few micrometers or hundreds of nanometers and its diameter vary in the

range of few nanometers. The carbon nanotubes can be classified as single walled carbon nanotube and multi walled carbon nanotube depending upon the sputtering (transformation) time of chemical vapor deposition process. Carbon nanotubes are known because of its properties such as high tensile strength in the order of 10's of GP, lightweight and high electronic conductivity [3, 5]. Carbon nanotubes can be further classified into metallic and semi-conducting carbon nanotubes with an energy band gap inversely proportional to its diameter, a property that is a direct result of parent graphene. Furthermore, carbon nanotubes are considered to be as semi-metal, zero band gap semiconductors. This could be because of the fact that the band gap between the valence band and conduction band is too small to be considered as a semiconductor and there is no overlap of conduction and valence bands to be fully recognized as a metal [3, 14].

3. DIFFERENT DEPOSITION TECHNIQUES

3.1 Electrophoretic Deposition

The preparation of carbon nanotube suspension for electrophoretic deposition is carried out by chemical oxidation of carbon nanotube in a concentrated solution of HNO_3 and H_2SO_4 mixed in the ratio of 1:3 by volume. The chemical oxidation of carbon nanotube method is carried out by refluxing 1 g of raw carbon nanotube into 40 ml of acid mixture at 120^0C for 30 min. Followed by washing of the oxidized carbon nanotube with distilled water, yielding 50% by wt. of neutral oxidized carbon nanotube. Furthermore, the ultra-sonication and centrifugation (3000 rpm for 15 min) of oxidized carbon nanotube takes place. Therefore, a well dispersed carbon nanotube suspension will be prepared [18].

In order to establish kinetics of deposition process, there are a number of conditions upon which electrophoretic deposition of carbon nanotube depends: variation of field strength, carbon nanotube concentration and deposition time. Materials such as 316L stainless steel are used for building the electrodes of electrophoretic deposition cell. The electrodes are degreased by acetone, followed by distilled water. The electrodes within a cell bath are kept parallel to each other, maintaining a sufficient distance between them. Further, sonication of carbon nanotube for 15 minutes is carried out within an ultrasonic bath. TTi 1906 computing multimeter is used for recording the current flowing through the electrode as a function of time. To the end, the coated samples were removed from the electrophoretic deposition cell to minimize any drag between the wet coating and the remaining suspension. The surface morphology and thickness of the carbon nanotube film were characterized by field emission gun SEM. Atomic Force Microscopy with a pico SPLE was used to calculate the local inhomogeneous packing density [19-20].

3.2 Deposition around a Microfiber via Evanescent Light

The deposition of carbon nanotube around the microfiber through evanescent light has been done using experimental setup. This setup consists of a bare standard single mode fiber, placed on two fiber holders and fixed on two translation stages. The fiber ends are connected respectively to erbium doped fiber amplifier (EDFA) and power meter. The fiber is being tapered in such a way that the tapered diameter becomes 6 μm.

The deposition of carbon nanotube around the microfiber takes place through the immersion of microfiber into a carbon nanotube dispersed DMF on a slide glass. The EDFA amplifies the light of wavelength 1560 nm from an optical power of 10 dBm to that of 13 dBm, emitted by a laser diode. The amplified light is injected into the microfiber. The power meter is used to detect the start of deposition of carbon nanotube around the microfiber, hence calculating the deposition time. High pressure carbon monoxide is used for fabrication of carbon nanotubes. Carbon nanotube has absorption peaks at 1175, 1325, 1425 nm. This absorption band included 1.55μm wavelength ranges. Saturable absorption can be expected at the wavelength for passive mode locker. In this deposition method microscope Raman spectroscopy is used to ensure the existence of carbon nanotube [20-22].

3.3 Electroless Plating

The Electroless deposition method requires pre-activation of carbon nanotubes for its initiation. Pre-activation of carbon nanotube is carried out by ultrasonically dispersing the carbon nanotube in a solution of 0.1M $SnCl_2$/ 0.1M HCl for 30 min. Moreover, the carbon nanotube aggravated with Sn^{2+} ion is introduced in an aqueous solution of 0.0014M $PdCl_2$/0.25 M HCl for another 30 min. Then, the sensitized carbon nanotube is transferred from the solution to the electroless plating bath. The whole procedure takes place in a room temperature and the samples are washed periodically after each step. Transmission electron microscopy technique is used to analyze the nickel based carbon nanotube [20, 23-24].

3.4 Electroplating method

The electroplating method is carried out through preparation of a solution containing dark color suspension by mixing carbon nanotube and nano diamonds in a deionized water at a weight ratio of 1:10:100. Moreover, the suspension is mixed with a Cu electroplating solution in a ratio of 1:100.

Electron beam evaporation is being used for depositing a layer of titanium and copper on silicon wafer, which acts as an adhesion and electroplating

seed layer respectively. The electrodeposition method is carried out by creating a window of area 1 cm^2 in a thin polymer layer over the diced silicon piece. Furthermore, DC and PPR currents are applied by a Wafer Power Technology power supply. On using PPR method, the forward pulse current density, J_f with 50 mA/cm^2 was applied with power ON time, T_{fON}= 50 ms. The reverse current density, J_r of 10ma/cm^2 and power ON time, T_{rON} varying in the range from 10-75 ms The forward as well as reverse pulse OFF time were both maintained at 100 ms. The films resulting from co-deposition of Cu/Carbon nanotubes were examined by scanning electron microscopy [20, 24-26].

3.5 Immersion Plating

Prior to immersion plating process, carbon nanotubes are purified in a bath containing 3 parts of nitric acid and 1 part of sulfuric acid for 30 min at 80^0C. Followed by 2 hours of cooling and then flushed in distilled water bath for 1 day. Further, they were filtered using a filter paper of 0.45 nm pore size and then dried in a baking furnace. The modification of carbon nanotube with individual dispersant takes place at constant temperature, pH value and ultrasonic treating time. UV-visible spectrophotometer is used to analyze the characteristics of a modified carbon nanotube and zeta potential for carbon nanotube dispersion as well as surface potential of modified carbon nanotube.

Table 1: Comparison of electrode and reagent used in different deposition techniques

Deposition technique	Electrode (substrate) material	Reagent Used	References
Electrophoretic Deposition	316L stainless Steel	Acetone and distilled water	[19-20]
Deposition around a micro-fiber via evanescent light	Microfiber	DMF	[20-22]
Electroless plating	Nickel based carbon nanotube substrate	.1M $SnCl_2$/ 0.1M HCl, 0.0014M, $PdCl_2$/ 0.25M HCl	[20, 23-24]
Electroplating	Silicon wafer	Carbon nanotube, Nano diamond	[17, 21-23]
Immersion plating	Zn electro-plated carbon nanotube substrate	Nickel based solution	[20, 24, 27]

Zinc electroplated glass substrates were taken and zinc layer was displaced by nickel in order to form carbon nanotube/nickel composite. The glass substrates were first activated and sensitized and then electroplated with zinc. Electroplating of zinc takes at a voltage of 4 V and a current of 0.5 mA for 5 min. Then, the zinc electroplated glass substrate was dispersed into solution containing carbon nanotube and nickel. After immersion, a layer of carbon nanotube/nickel composite is formed which act as a carbon nanotube field emitter. The field emission characteristics of carbon nanotube/nickel were measured inside a chamber with 5×10^{-6} torr vacuum diode structure with a spacer height 150μm [20, 24, 27]. Table 1 shows the comparison of electrode and reagent used in different deposition techniques.

4 CARBON NANOTUBE INK PREPARATION TECHNIQUES

The preparation of carbon nanotube ink begins with the separation of carbon nanotube inside a liquid through the process of sonication [28]. Shortening of nanotubes as well as causing defects are the two end products of sonication. The shortening of nanotubes decreases the conductivity with the decrease in electronic pathways within a network of carbon nanotube. In addition to that, the inherent conductivity is negatively affected by the defects. However, the overall process of dispersion can be undergone through the process of sonication. After sonication, centrifugation takes place to obtain a well dispersed carbon nanotubes from bundles and agglomerations. To this end, filtration of nanotubes ink takes place from the bundles in order to prevent from clogging through the printer nozzle [28-31].

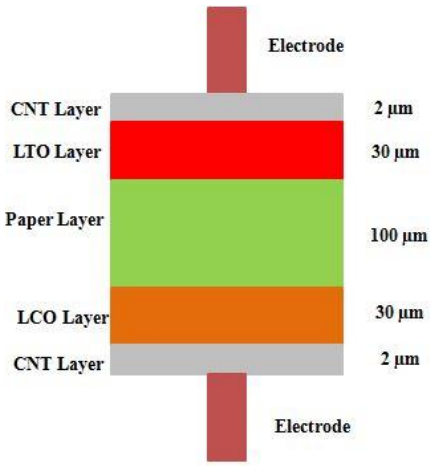
Another method has been derived for multi walled carbon nanotube. Since both single walled carbon nanotube and multi walled carbon nanotube belong to a particular class of nanomaterial i.e., carbon nanotube and shows some common behavior like working conditions, dissolution properties, this method can also be used for single walled carbon nanotube.

The preparation of carbon nanotube ink begins with the purification of multi walled carbon nanotube (single walled carbon nanotube for this paper) samples [42]. Followed by the fragmentation of the samples in a ratio of 3:1 H_2SO_4/HNO_3 at 140˚C for 30-60 minute simultaneously as well as washing the samples with de-ionized water to pH-7. Then the samples are centrifuged for 10 minutes at 3000 rpm and then the deposit produced are carried to the centrifuge rotating at 20000 rpm for 15 minutes, where the deposit taken out was ultrasonically dispersed in distilled water with special dispersant [29, 32-35].

Both the methods can be used for the preparation of carbon nanotube ink, but the first one is not specific about the concentration of the samples, the working conditions of the process and it is associated with the reduction in conductivity of the samples. Whereas the second one is taken from a paper where researcher has carried out the process for multi walled carbon nanotube; this issue can be covered as the above mentioned condition may fluctuate a little in case of single walled carbon nanotube.

5. PREPARATION OF A PAPER BATTERY

Paper battery structure as shown in Fig. 2 describe the details different layer of thickness of a battery and materials used in anode, cathode and electrolyte. A Xerox paper of size according to the desired length of the battery is taken. Then, the specially formulated ink of metallic single walled carbon nanotube is conformably coated over the cellulose paper through a simple Mayer rod [4]. A small volume of ink being ejected out from the nozzle requires low surface tension over its surface. The adsorbed carbon nanotube on the cellulose surface increases the surface area of the cellulose paper [36]. Moreover, the electrolyte i.e. sweat/blood/urine is applied over the cellulose. Then, the exposed cellulose surface is laminated with a thin film. Furthermore, a current collector specifically, of aluminum metal is connected to the battery [36-37]. The current collectors are pretreated to reduce corrosion rates and improve adhesion properties [37]. The single walled carbon nanotube electrode should be aligned in the direction of ionic diffusion, thus the accessibility of electrodes interior to the electrolyte is significantly increased [38]. A cellulose paper infiltrated with the solution of EC, and DEC is placed in between the electrodes to act both as separator and electrolyte [38].



.Fig. 2 Paper battery structure [13]

6. SIGNIFICANCE OF BIO-ELECTROLYTE IN A PAPER BATTERY

In a paper battery sweat/ blood/ urea (bio-electrolyte) is used as an electrolyte. The evolution of H_2F gas during lithiation is a major cause for the degradation of electrolyte [39] (H_2F is obtained when $LiPF_6$ is combined with H_2O). In paper battery, there is no such compound which would lead to the evolution of H_2F gas. Therefore, there is a significant improvement in the cycle life of a paper battery. Moreover, after the first cycle, the coulombic efficiency of delithiation of 77% is obtained and the rest 23% by irreversible capacity, where, 10-12% (varying with battery's temperature) of the 23% (the irreversible capacity) [37, 40] is used up for the growth of solid electrolyte interface, during the intercalation of Li^+ ions into the anode. The amount of irreversible capacity utilized depends on graphite's surface area and layer formation condition [39]. Whereas, in paper battery there is a very less deposition of anions over the electrodes, thus solid electrolyte interface layer formed over the electrode is relatively thinner than the Li-ion battery. Therefore decrease in efficiency of the cycle life of paper battery due to irreversible capacity is very less.

7. ADVANTAGES OF A PAPER BATTERY

The paper battery is a better alternative to the traditional battery system. It is a form of lithium ion battery where shorter length metallic single walled carbon nanotube is used in order to increase its efficiency. Apart from this, there are following advantages which have been discussed in the form of following points. (i) The cellulose used as a cover of paper battery, is reusable and recyclable material [41]. (ii) It does not get overheated even under extreme conditions because of its low resistance. (iii) It is very thin, light weight and flexible [1, 41]. (iv) Paper battery is considered to be consisting of a multiple number of cells in series. Therefore cutting the battery into two halves decreases the output voltage to half of the original one [1]. (v) Output voltage of the battery can be varied by varying the carbon nanotube concentration. (vi) The carbon nanotube used in the paper battery helps in the accumulation of charges in between its carbon lattice as well as flow of charge through variable hoping mechanism, makes it a Super-capacitors. Therefore, paper battery shows the property of both i.e., as a super-capacitor as well as a battery [3, 5, 11]. (vii) Lithium incorporated in carbon nanotube, reduces its toxicity. (viii) Due to the intrinsic rough and porous surface of the single walled carbon nanotube electrode surface, the solid electrolyte interface layer covering the electrode surface is non-linear, hence covering the maximum surface area. This helps in better intercalation of ions within the electrode and achieving high-power performance. (ix) Bio-electrolytes are applied over the cellulose surface, so there is no leakage problem, under accidental damage.

8. ABBREVIATIONS

Symbols	Abbreviation
$LiPF_6$	Lithium hexafluorophosphate
$LiMnO_4$	Lithium permanganate
$LiCoO_2$	Lithium Cobalt Oxide
HNO_3	Nitric Acid
H_2SO_4	Sulphuric Acid
HCl	Hydrogen Chloride
$PdCl_2$	Palladium(II) Chloride
H_2F	Hydrogen Fluoride
H_2O	Water

9. CONCLUSION

In this paper, we have reviewed a single walled carbon nanotube based thin film battery (paper battery) and discussed the increase in efficiency as well as the cycle life of a battery as a result of deposition of carbon nanotube ink over the cellulose surface. A flexible energy storage system which find its application in a diversified field i.e., it can be used in technologies as small as wrist watches as well as have a capability to be used in electric vehicles when cells are parallelly assembled. Paper battery is a thin and versatile energy storage system which can be easily fitted within small devices such as oximeters, watches, pacemakers of heart etc. Apart from its thickness, paper battery is eco-friendly as its components are either found from natural resources (such as cellulose, bio-electrolytes) or they are man-made (such as single walled carbon nanotube), therefore it is flexible to be used in most technologies. Moreover, the output voltage of paper batteries multiplies on subsequent addition of cells; therefore they can also be used in electric cars, rickshaws, bikes etc. Deposition of carbon nanotube over the cellulose surface significantly increases the conductivity of the surface. The single walled carbon nanotube used as an anode decreases the potential window between the electrode and the electrolyte. The bio-electrolyte used over the ionic compounds prevents degradation of electrolyte, hence increasing the life of a battery. On the other hand, the cost for the preparation of carbon nanotube through the chemical vapor deposition technique is quite high. Therefore, the overall cost of the paper battery becomes high.

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