Synthesized carbon material with semi-organic and aqueous electrolyte for supercapacitor application

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ABSTRACT

The supercapacitor technology is maturing to play an important role in the today's hybrid electrical energy storage systems. Supercapacitors are dominated by unique properties that can accompany other energy storage strategies due to its functional characteristics-such as quick charge and discharge ability, high power density, very long cycle life. It results in to improved system efficiency of hybrid electric energy systems with reduced environmental pollution levels. In this paper, efforts are made to construct a low-cost model of supercapacitor using synthesised carbon material obtained from the pyrolyzed scrap tyre by using both aqueous and Aloe vera gel as semi-aqueous or semi-organic electrolyte. The test results are obtained in the specially developed laboratory to investigate the characteristics of fabricated supercapacitor in terms of capacitance, internal resistance and peak current. The obtained results are superior as compare to Vulcan XC-72R with trade-off between peak discharge current and other parameters. New carbon material obtained from scrap tyres is cost effective when used along with Aloe vera gel as an electrolyte. Use of scrap tyre carbon as electrode material and Aloe vera gel as naturally available material for electrolyte is an innovation of the presented work.

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1. INTRODUCTION

The lifespan and power density of traditional energy storages like batteries are constrained. Batteries are also unable to handle abrupt and dynamic changes in loads due to their slow discharge characteristics. Additionally, drawbacks like increased thermal heating and proportionate cost for high power supply are also the main worries, therefore employing a battery as a standalone device does not offer effective transient performance [1], [2]. The electrochemical double layer capacitor (EDLC), often known as a supercapacitor, has the unique capability to supply fluctuating, transient, as well as instantaneous power demands [3]. Supercapacitors rarely experience thermal problems, and their best performance in a hybrid energy storage system maintains battery temperatures within acceptable ranges. Supercapacitor have gained an importance in the field of peak power supplies, medical equipment, pulsed lasers, space crafts, regenerative braking energy recovery in elevators [4], military equipment and hybrid energy storages comprising of batteries and supercapacitors for electric vehicles. Supercapacitor use in conjunction with batteries enhances the cycle life of batteries and reduction in on board battery sizes in hybrid electric vehicles [5]-[7]. Supercapacitor electrode materials mainly comprises of carbon nano materials in different forms such as graphene, nano tubes, nano wires which give superior characteristics in an organic electrolyte but their overall commercial costs are much higher. Most commercial supercapacitor use activated carbon obtained from the carbon precursors such as coconut shells, wood powder, and coal tar, for electrodes with active surface area ranging from 1000-1500 $meter^2.gram^{-1}$, that result in up to 500 $farad.gram^{-1}$ [8]-[10]. The developed surface area of such activated carbon materials is essentially consisting of micropores (<2 nano-meter), mesopores (2-50 nano-meter) and macropores (>50 nano-meter), which gives a specific area as high as 3500 $meter^2.gram^{-1}$. Supercapacitor cells with an organic electrolyte usually attain voltages in the range of 2-2.7 V with capacitance reaching to 100 $farad.gram^{-1}$ and aqueous electrolytes have a lower voltage range of 0.9-1.1 volts with capacitance up to 200 $farad.gram^{-1}$ [11]-[13]. It is reported that the rice husk as a precursor material to derive activated carbon material resulted in 2523.4 $meter^2.gram^{-1}$area with average pore size of 2.2 nm [14]. It is reported that the carbon material obtained from the pyrolysis of kenaf resulted in the capacitance up to 242 $farad.gram^{-1}$ [15]. Another novel green approach research has also been reported wherein biomass of bougainvillea flowers is used to derive mesoporous perforated graphene which resulted into surface area of 850 $meter^2.gram^{-1}$ with specific capacitance of 458 $farad.gram^{-1}$ [16]. Thus, appropriately pyrolyzed precursor materials followed by their activation process yields cost-effective carbon material for the supercapacitor electrodes with desired characteristics.

The desired characteristics of the supercapacitor is coupled with the selection of electrolyte materials with trade-off between high power density and high energy density. Power density is strongly reliant on the electrolyte conductivity and hence internal resistance and high energy density on the voltage window. Organic or non-aqueous electrolytes, such as acetonitrile is the most commonly used in commercial devices, due to their higher dissociation voltage. However, the resistivity of organic electrolytes is higher and therefore, limits the cell power. An aqueous electrolyte, such as sulphuric acid and potassium hydroxide [17] have higher conductivity than organic electrolytes and lower internal resistance and thus higher pulse power delivery. It is reported that the hydrogel possesses the conductivity of 0.0154 Simens-m-1 and introduction of Aloe vera gel to polyvinyl alcohol (PVA) increases the conductivity of hydrogel [18] with reported value of electrical conductivity of about 0.0063-0.0236 Sm-1at 30OC to 60 degree centigrade [19]. The chemical composition of Aloe vera gel as reported in literature is: Moisture-96.31%, pH-4.33, Sodium (milligram/100 gram) -49.65, Potassium (milligram/100 gram) -20.74, Zink (milligram/100 gram) -0.114, Cu (milligram/100 gram) -0.034, Calcium (milligram/100 gram) -71.46, Ascorbic (milligram/100 gram) -41.4, Acidity-0.1% with ascorbate acts as an electron-doner which is a conductivity enhancer [20].

This research paper is organized in 5 sections. The section 1 focuses on the literature research akin to activated carbon materials and their characteristics besides pre-cursor materials and post activation characteristics. The section 2 outlines proposed method with model of supercapacitor and rubber tyre material characteristics. The section 3 deals with the methodology used in experimental setup and stages of experimentation in the developed laboratory. The section 4 presents results obtained with various experimental trials on the developed activated carbon material for its use in fabricating the supercapacitor cell. The section 5 deals with concluding remarks on the research work.

2. PROPOSED METHOD

According to the literature review, less effort has been put into using naturally occurring materials and scrap materials in supercapacitor applications. It exemplifies the potential for using recycled rubber tyres as precursor carbon materials, which is a novel source of carbon for the development of economically viable electrode materials for supercapacitor. The open market cost of such scrap rubber tyres varies from $ 250-350 per tonne ($ 0.25-0.35 per kilogram). The disposal of abundantly available scrap rubber tyres results in serious environmental problem and pollution as rubbers are unlikely to be decomposed or biologically degradable [21]. The activated carbon derived from the scrap rubber tyre after appropriate synthesis assumes nano molecule size on ball milling and is targeted to be a viable cost-effective electrode material with novel idea of using semi-organic electrolyte i.e., Aloe vera gel for supercapacitor to target characteristic in between the aqueous and organic electrolytes.

Characterization of supercapacitor fabricated from the carbon material derived from scarp rubber tyre with aqueous and semi-organic electrolytes is one of the novel aspects of this research work as there is a research gap linked to hydrogel electrolyte in semi-organic or gel in natural or derived form. Therefore, it is proposed to develop activated nano-carbon material from the recycled scrap rubber tyre through pyrolysis, synthesis and ball milling process in the laboratory. And to fabricate supercapacitor cell by using such activated carbon nano material to investigate its characteristic in between aqueous and semi-aqueous or semi-organic electrolyte.

2.1. EDLC model

Capacitance of the EDLC can be calculated from the charge/discharge curve from the ratio of difference in state of charge at time t_1 and t_2 to the difference in corresponding voltage V_1 and V_2 [22]. Series resistance (R_S) is mainly linked to the electrolyte resistance including current collector. As per the Helmholtz theory, since electrolyte concentration is well above 0.1 mol.l^{-1} and diffusion effect resistance (R_f) is much higher than R_S so it is negligible in comparison to R_S. Also, the diffusion layer which give rise to parallel capacitance can be neglected in practical studies [23]-[25]. Commonly referred terminal-based series resistance-capacitance (RC) model i.e., internal resistance in series with its capacitance model is used normally [26]. Although, the arrangement of transmission line model is most suited for the uniform pore diameter, such as conducting polymers, metal oxides but this model fails in case of activated carbon materials as pores are not identical and uniform. In activated carbon materials, the precursor material expels unstable matter, leaving pores with diverse wall chemistries and diameter during pyrolysis of the precursor materials. Novel and high precision model in the form of vertical ladder network with different time constant is also reported. It is found that micropores and mesopores whose diameter fall in the range 0.3-50 nanometre is very difficult to model. A significant consideration is the compromise between the total capacitance of the device and a pore resistance [27]. EDLC cell capacitance, power and energy model equations are given by the following governing relationships.

$$C_{DL} = k\,\varepsilon o\,\frac{A}{D} \qquad E = \frac{1}{2}CV^2 \qquad P_{max} = \frac{V^2}{4\,ESR}$$

where,
k - dielectric constant, ε_o - permittivity, A - electrode surface area, D - electrodes distance
V - voltage, C_{DL} - capacitance of electrochemical double layer, Ce = interface capacitance
R_S - series resistance and R_f - diffusion effect resistance, which is normally neglected

Figure 1 shows EDLC cell model with formation of capacitance given by the following relationship and Figure 2 depicts diffused capacitance along the pore boundary in an activated carbon material used in electrodes.

$$\frac{1}{Ccell} = \frac{1}{C_+} + \frac{1}{C_-} \qquad C_{cell} = \frac{Ce}{2}$$

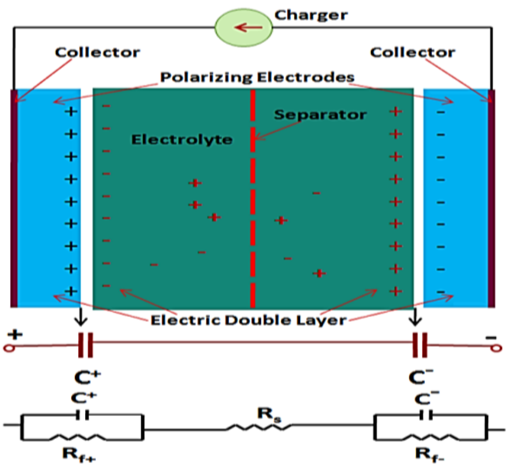

Figure 1. Supercapacitor (EDLC) cell

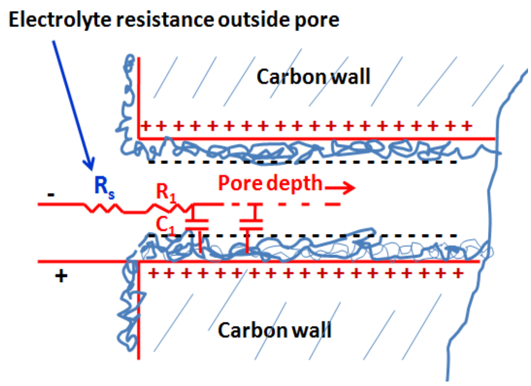

Figure 2. Diffused capacitance foramtion in cabon pore of an activated carbon material

2.2. Rubber tyre chemical analysis and ball milling process

The solid residue after pyrolysis of scrap tyre contains carbon black and the mineral matter. Only few numbers of studies have been reported to investigate the pyrolysis of waste tyres in the laboratory for obtaining the carbon materials. These studies are based on pyrolysis in a nitrogen atmosphere using a fixed-bed batch reactor at 300-720 degree centigrade [28]. Elemental analysis of the carbon obtained from scrap tyre gives 71-80% of Carbon, 7% of Hydrogen, 5.4% of Iron, 0.4% of Nitrogen, 2.8% of Sulphur, 2.3% of

Zink, 1.3% of Calcium, Zink-oxide -2%, 8% of ash [29], [30]. Such activated carbon materials can be ball milled to obtain cost-effective carbon nano materials. This method is also treated as creation of functional materials by mechanochemical synthesis. Ball milling grinds carbon material or carbon nano tubes into extremely fine powder depending up on the milling duration, drum's revolving speed, size and type of the balls. At certain processing conditions, the particles can be milled as small as 100 nano-metre. This process has been employed to transform carbon nano tubes into smaller nano particles [31]. It is reported that the unique carbon nanostructures including carbon nanotubes and carbon onions are synthesized by high-speed ball milling of steel balls under local high temperatures induced by the collision-friction energy resulting in phase separated unique carbon nano material [32]. It is reported that the supercapacitor capacitance is increased on grinding the metal oxide and carbon materials up to twelve hours. It is also reported that the iron oxide materials take the form of 6-7 nano-metre particle size after 14 to 28 hours of ball milling [33].

3. METHOD

The activated carbon materials that are now on the market have a high surface area but a low mesoporosity, which results in a limited capacitance because the electrolyte is not readily accessible. Additionally, poor electrical conductivity and low electrolyte accessibility cause high internal resistance, which reduces power density. In order to address the shortcomings of currently available activated carbon materials, the potential of recycled rubber tyres, which are widely available at a very low cost, has been chosen as a precursor carbon source for generating new activated carbon nanomaterials. Hydrogel as semi-organic material as electrolyte has not been reported in the application of supercapacitors as on date. Therefore, the potential of a semi-organic electrolyte, such aloe vera gel, is aimed to be used to obtain supercapacitor characteristics in between aqueous and organic electrolyte. A stack typed construction of supercapacitor is prepared for research and characterization with available laboratory equipment. The main considerations towards the development of supercapacitor by using new activated nano carbon material from the scrap rubber tyre is to achieve higher value of capacitance, pulse value of discharge current and cell voltage. This innovative research is a step towards healthy ecosystem and development of cost-effective supercapacitor, which may find its application in hybrid energy storage system to supply short duration pulse power.

3.1. Laboratory set-up

A separate mini-laboratory is set up for the supercapacitor research work which comprises of Muffle-furnace (1200 degree centigrade capacity with auto control), Nitrogen gas cylinder with regulator and controls, Horizontal ball milling machine with steel balls (1 kilogram capacity), magnetic stirrer with hot plate, hot press machine, digital weighing machine with least count 0.001 gram, Auto range inductance-capacitance-resistance (LCR) meter, cylindrical reactor, battery charger (bosch), battery tester, direct current regulated power supply, silica and glass pots, separator materials, substrate materials, different chemicals and solutions. Typical instrumentation used in trials in the laboratory are shown in Figure 3. A specially designed stainless steel (SS)-310 grade sealed horizontal type cylindrical reactor with 3-millimetre thickness, 10-inch length, 4-inch diameter with 10-millimetre diameter inlet and outlet pipe for connecting nitrogen gas pipe is fabricated in the workshop. Figure 4 depicts this cylindrical reactor arrangement for the pyrolysis of raw scrap tyre material in a muffle furnace.

Figure 3. Instrumentation for Laboratory testing

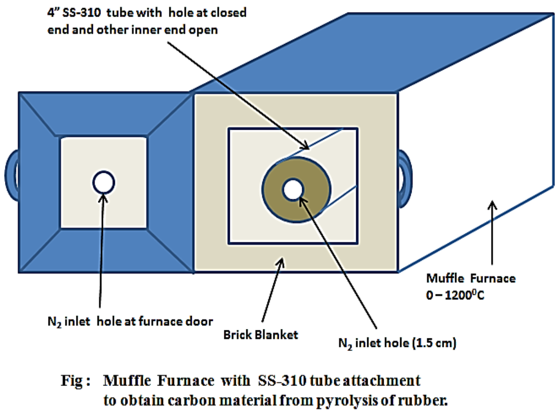

Figure 4. Furnace with SS-310 reactor for pyrolysis

3.2. Nitrogenated pyrolysis and synthesisation

The nitrogenated carbon exhibits pseudocapacitive behaviour with an increase in capacitance which is almost double as that of plain porous carbon. Further, nitrogenated carbon resulted in improved cyclic stability due to retention of its high capacitance even after widespread cycling. Most importantly, the nitrogenated carbon show battery-like characteristics [34]. The adsorption of nitrogen molecules affects the available surface area for the chargeable double-layer formation in the micropore region but do not affect larger pores significantly. This structural and chemical modification of the nitrogenated carbon serves as a control mechanism to access the active molecules in the micropores, thus, increasing its capacitance while cycling. The nitrogen adsorption isotherms lead to the development of the porosity by the activation process [35].

Sample scrap rubber tyre of lorry is selected for the pyrolysis in furnace reactor. A 50-gram scrap rubber tyre is taken for the pyrolysis in the reactor. The reactor's inlet pipe is connected to the nitrogen gas cylinder through gas pressure regulator for nitrogenated pyrolysis in the reactor and to remove oxygen. The outlet pipe of the reactor is connected to the exhaust system. Furnace timer is set for 800 degrees centigrade. It took 50 minutes to reach to 550 degrees centigrade and 70 minutes to reach to 800 degrees centigrade. Sample scrap tyre material is pyrolyzed for two hours at 800 degrees centigrade and allowed to cool for several hours. Final char residue of 50-gram scrap rubber tyre found to be 16 grams. This char residue is ball milled for 12-14 hours to reduce material particle size to nano form. The ball milled material is then heated in the reactor for an hour at 800 degrees centigrade in a nitrogenated atmosphere. The balance residue after second stage heating of ball milled carbon was found to be 13 grams. This derived carbon material possesses impurities which could not be dispensed with during the pyrolysis process. In order to minimize these impurities, filtration and several steps of synthesisation is done on 5 gram of derived carbon in a 100-millilitre hydrofluoric acid (40%) and 5 millilitre nitric acid and stirred it for 8 hours. Then it was filtered by using Watman filter paper no. 42 and thereafter, water wash was done three times followed by filtering every time and then finally ethanol wash was done with 50 millilitre ethanol. The filtrate of the carbon powder was given heat treatment at 200 degrees centigrade for 1 hour, which yielded 4.2 grams of final residue of carbon.

3.3. Fabrication of supercapacitor cell

Electrode were made by pasting a slurry of newly derived carbon material (tyre carbon) on the current collector or substrate of SS-310 steel mesh (100 wires per inch) using propanol as binder and a cell is formed using porous polyethylene as separator. The fabricated electrode was given mild heat treatment to make it completely dry. Battery type separator such as polyethylene has 50-65% porosity possessing mean pore diameter of 0.15 micron with electrical resistance of 25-55 milliohm-sq.cm. Therefore, in this research work, carbon paper and polyethylene are used as separator for the fabrication of stack type supercapacitor. Based upon the characteristics, the Aloe vera gel electrolyte is considered to be a suiting material which is in semi-aqueous form to display the characteristics of aqueous and organic electrolyte combinely. The commercially available Aloe vera gel with 97% moisture/water and 3% of alcohol-based matters selected as semi-aqueous electrolyte for making supercapacitor module. After the fabrication of supercapacitor by using derived tyre carbon material, an Aloe vera gel available with given chemical composition is selected for obtaining higher conductivity and enhanced voltage window of the fabricated supercapacitor. Figure 5 show developed supercapacitor module housed in a container filled with Aloe vera gel as semi-organic electrolyte. Figure 6 show the test set up for constant current charging and discharging of fabricated supercapacitor.

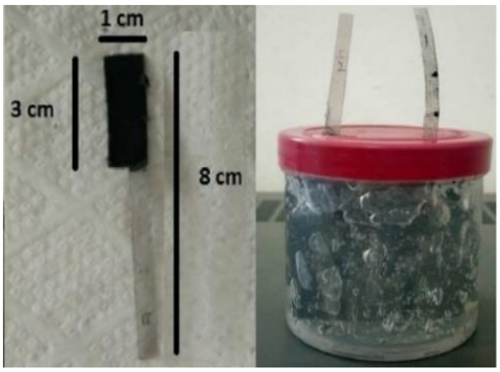

Figure 5. Supercapacitor prototype

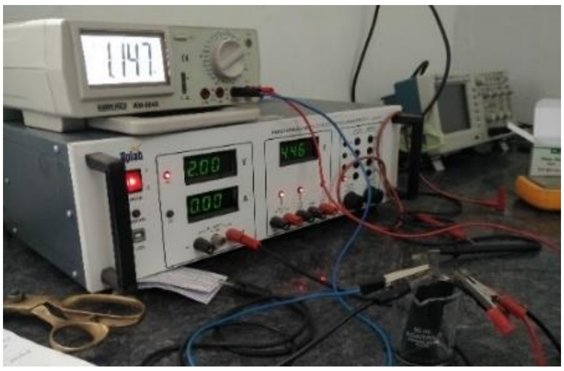

Figure 6. Supercapacitor charge and discharge

4. RESULTS AND DISCUSSION

Results obtained from the newly developed novel carbon material (tyre carbon) from the scrap rubber tyre with aqueous and semi-aqueous electrolytes are corelated and compared with the results of commercially available activated carbon materials such as Vulcan-XC72R. Characterization is also done in the laboratory on cyclic voltammetry to confirm capacitive behaviour of new material. Supercapacitor prototypes of tyre carbon with aqueous sulphuric acid and semi aqueous Aloe vera gel were developed for parametric evaluation.

4.1. Cost comparison

A cost of activated carbon available in the market is about $ 15-16 per kilogram. The estimated plain cost of derived carbon material comes out to be $ 1.6 per kilogram. By considering the doubling effect to account processing cost, the estimated cost of activated new carbon material comes out to be $ 3.2 per kilogram which is almost 4-5 times lesser than the cost of activated carbon materials available commercially. This finding is one of the take away of this research work.

4.2. Capacitor discharge current and capacitance: Aloe vera gel as electrolyte

Obtained results of the supercapacitor developed from tyre carbon are presented in the Table 1. It is seen that the cell internal resistance for tyre carbon is marginally more than Vulcan-XC72R. This pronounced effect was anticipated in the case of activated carbon materials as it has already been reported about higher values of internal resistance. Further this internal resistance is very much low for series-parallel combinations of the cells. Through experimentation, it is found that 2V is acceptable charging voltage for Aloe vera gel-based electrolyte.

From Figure 7 it is seen that the rate of discharge of the tyre carbon supercapacitor is sluggish in comparison to Vulcan-XC 72 carbon material which depicts somewhat battery like discharge which is desirable for sustain energy output. It can be concluded that the nitrogenated synthesized tyre carbon material with its ball milling processing could be the potential reason to achieve higher pulse current and capacitance. Different modules of electrodes of tyre carbon material were developed in different sizes and the corresponding capacitance was calculated from respective discharge curve. Figure 8 depicts that the nature of the curve is exponential thereby confirming that the increasing electrode area rapidly increases the supercapacitor capacitance for tyre carbon. However, beyond certain limit this rise restricted due to fabrication issues associated with it.

Table 1. Performance parameters of developed supercapacitor

Carbon Material	Pulse Current (milliampere)	Internal Resistance (ohm)	Capacitance (farad)	Electrode area (centimetre^2)
Tyre Carbon	30.5	24.9	0.85	3
Vulcan-XC72R	24	18	0.5	3

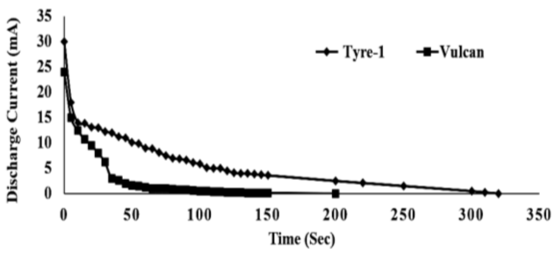

Figure 7. Pulse discharge current of supercapacitor

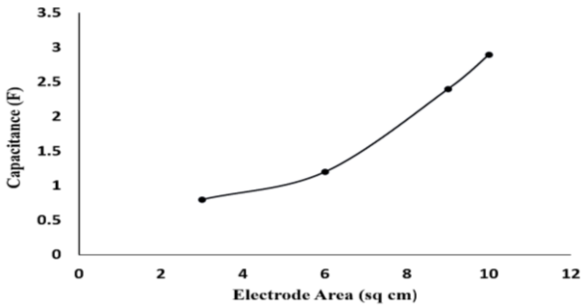

Figure 8. Capacitance Vs. electrode area (tyre carbon material)

4.3. Discharge current and capacitance: tyre carbon with aqueous electrolyte

Table 2 presents the results for the aqueous electrolyte material sulfuric acid for different concentrations which are generally used in electrical energy storage devices like battery. It is seen that the pulse or peak current and capacitance of the supercapacitor increases with molar concentration of sulfuric acid. As expected, the internal resistance is much lower than the semi-aqueous electrolyte such as Aloe vera gel due to higher conductivity of the aqueous electrolyte with cell voltage window of 1.3V.

Table 2. Performance parameters of tyre carbon material with sulfuric acid as aqueous electrolyte

Concentration of acid (mole)	Maximum pulse current (milliampere)	Internal resistance (ohm)	Capacitance (farad)
2	31	4.42	0.8
3	43	3.81	1.1
4	54	3.32	2.35

Figure 9 shows the discharge curves for aqueous electrolytes of various concentrations. From Figure 9, it is observed that the peak discharge current in the case of aqueous electrolyte such as sulphuric acid is increasing with the concentration of sulfuric acid. However, it is not advisable to increase concentration of sulfuric acid due to thermal instability of the cell. It is also observed that the current discharge rate of the aqueous electrolyte is somewhat faster as compared to the Aloe vera gel used in supercapacitor with tyre carbon material.

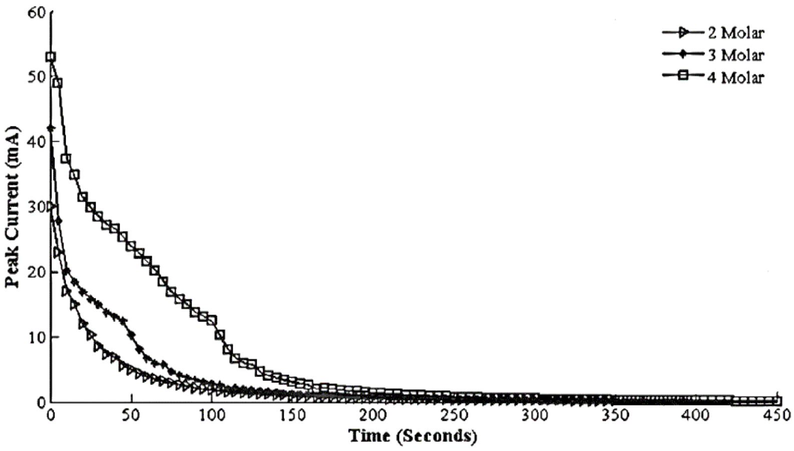

Figure 9. Discharge curves for aqueous electrolytes of various concentrations

5. CONCLUSION

Supercapacitor is coming up as good device, suitable for hybrid energy storage pack or standalone energy storage pack in few applications. It is seen as a device with highly porous carbon and compatible electrolyte. However, there is a need to look beyond conventional porous carbons and electrolytes. Use of scrap materials and naturally available materials in this device is rarely seen. Efforts in those directions are required. This paper is one such attempt. Characterization of supercapacitors using scrap tyre carbon materials with Aloe-vera gel as semi-organic material and sulphuric acid as aqueous electrolyte has been studied separately and it is the main finding of presented research work. The maximum voltage attained by the developed prototype was found to be 2 volt with semi-organic material and 1.3 volt with aqueous electrolyte. Activated carbon extract from scarp tyre is found to be less than 10%, but such carbon can solve issue of scrap tyre disposable. Carbon extracted from scrap rubber tyre is found to be possessing better characteristics than other activated carbon materials available in the market. It is concluded that the materials used in developed supercapacitor module proved to be cost effective as compared to other carbon materials with enhanced characteristics of the module. Cost of the proposed carbon is less than 20% as compared to commercially available carbon grades used in supercapacitor applications which is the notable finding of this research work.

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