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FABRICATION AND CHARACTERIZATIONS OF ELECTRIC DOUBLE LAYER SUPERCAPACITOR BASED ON ACTIVATED CARBON ELECTRODES IN AQUEOUS ELECTROLYTE

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ABSTRACT

Super capacitor or ultracapacitor are used for many industrial as well as commercial application like for storing electrical energy. Super capacitor is a high storage capacity capacitor with capacitance much higher than normal capacitor.In this work, we report testing and comparing supercapacitor made from copper plate and aluminum foil coated with activated carbon made of coconut husk and testing which electrolyte work best. In this experiment we tested water-based electrolytes such as KCl (Potassium chloride), Na2SO4 (Sodium sulphate), MgSO4 (Magnesium sulphate) and H2SO4 (Sulphuric acid) with different electrode and using different separator materials such as thin single layer tissue paper and thick writing paper. It was found that Cu-AC (Copper-Activated carbon electrode) with 1 molar aqueous KCl as electrolyte and Common writing paper as separator material gave the best results achieving a maximum capacitance of 3.33 farad and a maximum voltage of 1volt and theoretical energy capacity of around 1.665 joule.

Keywords— EDLC supercapacitor, activated carbon, aqueous electrolytes, electrode

INTRODUCTION

Supercapacitors are recognized for over fifty years and are considered in concert of the P.E. storage systems. Electrochemical capacitors (ECs), often described electrical double-layer capacitors (EDLCs), supercapacitors, ultracapacitors, pseudo capacitances, gold capacitors, power capacitors or power caches, have involved universal research interest due to their potential applications as energy storage devices in many fields. Electrochemical supercapacitors or capacitors are novel power devices, which lie between batteries and standard dielectric capacitors in terms of energy and power densities with applications in computer power back-up, electric vehicles and power electronics. the key concentrate on developing supercapacitor materials has been on utilizing the double layer capacitance formed at the interface of the electrode and also the electrolyte. Electric double layer capacitor (EDLC) is incredibly attractive as an energy device due to its high energy density, maintenance-free long-life operation, quick charge–discharge rate, and environmentally friendly energy technology. For the applying, activated carbons (ACs) are basic materials for EDLC electrode due to their highly porous structure, large extent, good adsorption property, and high electrical conductivity. The electrochemical performances of EDLCs are associated with the pore structure, extent, and surface chemistry of the ACs. Now, ACs with high extent, suitable surface chemistry, and suitable pore structure are expected to be applied as excellent electrode materials to boost EDLC with high performance. Although this capacitance per unit area is low 10–30 mF cm2, it will be improved appreciably by the utilization of materials with high specific surface areas. the worth also has major range thanks to the presence of edge sites and use of surface functional groups to activate the carbon. a spread of carbonaceous materials with their great specific surface areas and superior conductivity provide as ideal candidates for supercapacitor devices

Capacitors which use charge-storage mechanisms, will be classified into two types: EDLC and Faradaic supercapacitor, the descriptions of which are explained within the following next section. ELDC type supercapacitor based on carbon materials such as activated carbon electrode work on the principle of charge separation at electrolyte electrode interface, [6] whereas pseudo capacitors material such as titanium, cobalt oxide, nickel oxide, vanadium oxide manganese oxide work on the principle of EDLC as well as electrochemical faradic reaction between ions and electrode materials within the suitable potential window. To develop ECs performance, especially its specific energy while retaining its basic high specific power, many researchers have focused their efforts totally on improving the properties of electrode materials. the most classes of materials applied for ECs include different styles of carbon, transition metal oxides, and conductive polymers

2Experimental details:

2.1Chemicals used:

Chemicals used were 70% H_2SO_4, Na_2SO_4 in crystalline for, KCl in crystalline form and $MgSO_4$. The chemicals were then diluted with water to make standard solution of known concentration, KCl was diluted to 1M by adding 0.7455 g of crystalline KCl to 10 ml water. Two standard solution of Na_2SO_4 were created 1M and 4M for creating 1M solution 0.7102 g of Na_2SO_4 was added to 10 ml of Water, and for creating 4M solution 5.681 g of Na_2SO_4 was added to 10 ml of water, for creating 1M solution of H_2SO_4 0.53 ml of H_2SO_4 was added to water

For calculating weight of chemicals needed the formula (1) and (2) bellow was used:

$$M = \frac{w}{v(Mw)} \quad (1)$$

Where M is Molarity of solution v is the volume of solvent used in l, W is the weight of solute in g, Mw is molecular weight of solute g/mol. By rearranging the formula, we get

$$w = M(v)(Mw) \qquad (2)$$

Using the formula given above we can calculate weight of the solute, in our case volume was constant to 10 ml i.e. 0.01 l

1) **2.2*Preparation of electrodes:***

2.2.1 Activated charcoal coating paste:

Activated charcoal used to coat electrode was made from coconut husk. The activated charcoal is first heated for about 5 minutes to clear off all the pores in Activated charcoal, after heating process binder solution is created to help activated charcoal stick to the electrode. Binder solution is created using Commercially available fevicol which has PVA (poly vinyl acetate) as major component. 1g of fevicol is added to a beaker followed by addition of 1.5 ml of water to help lower fevicols viscosity the solution is stirred vigorously until homogeneous mixture is obtained, after stirring 3 g of preheated activated charcoal is added to the mixture and stirred vigorously. After stirring for a while black homogenous paste is obtained. The paste will be used in next step of the process.

2) *2.2.2 Cu-AC (Copper activated carbon) electrode:*

For making Copper Activated Charcoal electrode we used commercially available copper clad board (ccb) as electrode (copper plates can also be used instead of ccb). First the ccb is cut into the shape of electrode, after cutting electrode 360 grit sand paper is used to sand the surface of ccb to remove any oxide layer (sandpaper of lower grit can damage the electrode). Water is used to get rid of any fine particles and dust, after drying activated charcoal paste is applied onto the copper side of electrode (10 mm gap should be left from either up or down to connect to power supply) the paste is then spreader evenly across the electrode. The electrode is then heated using hot air gun or on lab heat plate to evaporate water (if too much heat is applied to the electrode Binder might decompose) after drying of water from electrode the electrode is cooled to room temperature.

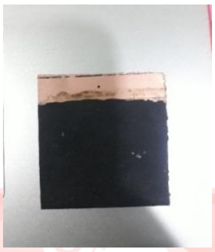

Figure 1

Figure 1 shows Copper clad board (ccb) coated with activated carbon

3) *2.2.3 Al-AC (Aluminum activated carbon) electrode:*

Process of making of Aluminum-Activated carbon electrode (Al-AC) is same as that of Cu-AC. But in making of Al-Ac aluminum foil is used instead of copper clad board

4) *2.3 Experiment procedure:*

Firstly, for making supercapacitor cell tissue paper or writing paper was placed onto one of the electrodes (Cu-AC, Al-AC), electrolyte solution was added drop wise (taking care the separator material should not tear) and the paper was allowed to absorb electrolyte solution and excess solution was removed. Second electrode was then placed over the first electrode with activated carbon side of the electrode facing the separator, gentle pressure is applied on one side

The supercapacitor cell was tested by applying a voltage by using a variable power supply and the testing voltage drop against a resistor and its discharge time.

A voltage of 0.9 v was applied initially to supercapacitor cell and charged for about 1-2 minutes; the voltage was gradually increased to 1.5v. After charging the cell the peak voltage was noted (Vmax) for calculating capacitance a known resistor of 100Ω resistance was connected across the cell to discharge the cell and a voltage meter was connected parallel to the cell for calculating voltage drop, amp meter was also connected in series with the cell to calculate peak current (Imax) and discharge current (Id)

After connecting 100Ω resistor across the capacitor started discharging and the voltage started to drop from its peak voltage. The minimum voltage after discharge was noted. The peak current and discharge current was also noted

3.Working principle:

The charge-storage mechanism of ECs (electrochemical capacitors) is predominately thanks to double-layer (DL) charging effects. But in common, further contributions of supercapacitance might also be a part of the observed capacitance thanks to the functional groups present on the electrode surface. the most important advantages of ECs are that they'll offer high powerexcellent reversibility (90–95% or higher), capability (60–120s is typical), and long cycle life. Classically they exhibit 20–200 times larger capacitance per unit volume or mass than conventional capacitors. Therefore, variety of applications of ECs are utilized in electric vehicles, electric hybrid vehicles, electric tools, electronic communication devices, mobile phones, digital cameras, pulse laser technique, uninterruptible power supply for computer and storage of electricity generated by powerplants.Electrical energy is stored in supercapacitor via two storage principle, static double-layer capacitance and electrochemical pseudo capacitance; andalso, the distribution of two kind of capacitance depend on material and stricture of electrode there are three type of supercapacitor which work on storage principle

Double-layer capacitors (EDLCs)– With activated carbon electrodes or derivatives with much higher electrostatic double-layer capacitance than electrochemical pseudo capacitance.

Pseudo capacitance – With transition metal oxide or conductive polymers electrodes with high electrochemical pseudo capacitance.

Hybrid capacitors– With asymmetric electrode, one of which exhibits mostly electrostatic and other mostly electrochemical capacitance, such as lithium-ion capacitors.

Because double-layer capacitance and pseudo capacitance both contribute inseparably to the full capacitance value of an electrochemical capacitor, an accurate description of those capacitors only are often given under the generic term. The concepts of supercapattery and supercabattery are recently proposed to higher represent those hybrid devices that behave just like the supercapacitor and rechargeable battery, respectively.

Double-layer capacitance and pseudo capacitance both contribute inseparably to the overall capacitance value of a supercapacitor. However, the ratio of the two can vary greatly, counting on the design of the electrodes and also the composition of the electrolyte. Pseudo capacitance can increase the capacitance value by the maximum amount as an element of ten over that of the double-layer by itself.

Electric double-layer capacitors (EDLC) are electrochemical capacitors within which energy storage predominantly is achieved by double-layer capacitance. within the past, all electrochemical capacitors were called "double-layer capacitors". Contemporary usage sees double-layer capacitors, along with pseudo capacitors, as a part of a bigger family of electrochemical capacitors called supercapacitors. they're also called ultracapacitors. during this project we are visiting specialize in electrochemical double layer capacitor. Electrochemical capacitors use the double-layer effect to store electric energy; however, this double-layer has no conventional solid dielectric to separate the charged ions. EDLCs store electrical energy by separating charged ions in ‘Helmholtz double layer’

Every electrochemical capacitor has two electrodes, mechanically separated by a separator, which are ionically connected to one another via the electrolyte. The electrolyte is made up of positive and negative ions dissolved within a solvent like water. At each of the two electrode surfaces originates a region within which the liquid electrolyte contacts the conductive metallic surface of the electrode. This interface forms a common boundary among two different phases of matter, like an insoluble solid electrode surface and an adjacent liquid electrolyte. during this interface occurs a very interesting phenomenon of the double layer effect.

Applying a voltage to an electrochemical capacitor causes both electrodes within the capacitor to get electrical double-layers These double-layers encompass two layers of charges: one electronic layer is within the surface lattice structure of the electrode, and also the other, with opposite polarity, emerges from dissolved and solvated ions within the electrolyte. the 2 layers are separated by a monolayer of solvent molecules, e.g., for water as solvent by water molecules, called inner Helmholtz plane (IHP). Solvent molecules adhere by physical adsorption on the surface of the electrode and separate the oppositely polarized ions from one another, and may be idealised as a molecular dielectric. within the process, there's no transfer of charge between electrode and electrolyte, that the forces that cause the adhesion aren't chemical bonds, but physical forces, e.g., electrostatic forces. The adsorbed molecules are polarized, but, because of the shortage of transfer of charge between electrolyte and electrode, suffered no chemical changes.

The amount of charge within the electrode is matched by the magnitude of counter-charges in outer Helmholtz plane (OHP). This double-layer phenomena stores electrical charges as a conventional capacitor. The double-layer charge forms a static field of force within the molecular layer of the solvent molecules within the IHP that corresponds to the strength of the applied voltage.

The double-layer serves approximately as the dielectric layer in a conventional capacitor, with the thickness of a single molecule. Thus, the standard formula for conventional plate capacitors (3) can be used to calculate their capacitance

$$c = \varepsilon \frac{A}{d} \qquad (3)$$

Accordingly,capacitance *C* is greatest in capacitors made from materials with a high permittivity *ε*, large electrode plate surface areas *A* and small distance between plates *d*. As a result, double-layer capacitors have much higher capacitance values than conventional capacitors, arising from the extremely large surface area of activated carbon electrodes and the extremely thin double-layer distance on the order of a few angstroms (0.3-0.8 nm), of order of the Debye length

The amount of charge stored per unit voltage in an electrochemical capacitor is primarily a function of the electrode size. The electrostatic storage of energy in the double-layers is linear with respect to the stored charge, and correspond to the concentration of the adsorbed ions. Also, while charge in conventional capacitors is transferred via electrons, capacitance in double-layer capacitors is related to the limited moving speed of ions in the electrolyte and the resistive porous structure of the electrodes. Since no chemical changes take place within the electrode or electrolyte, charging and discharging electric double-layers in principle is unlimited. Real supercapacitors lifetimes are only limited by electrolyte evaporation effects.

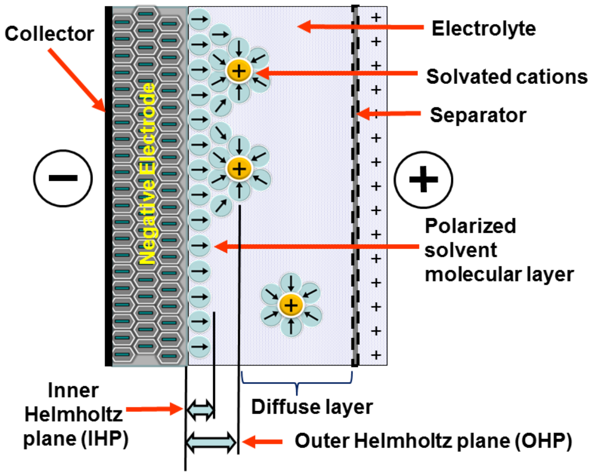

Figure 2

Figure 2 shows Double layer effect in super capacitor

4. RESULTS AND DISCUSSION:

Discharge method is used for calculating the capacitance of supercapacitor. Discharge method calculates the capacitance using discharge time of a given capacitor connected across some known resistance

Formula (4) is used to calculate capacitance using discharge method:

$$c = \frac{2t(v_2 - v_1)^2}{R(v_2^2 - v_1^2)} \qquad (4)$$

In the above equation:

t = capacitor discharge time across resistance R

R = Discharge resistance

V2 = Voltage of capacitor before discharge

V1 = Voltage of capacitor after complete discharge

C = capacitance of capacitor

CU-AC = Copper electrode coated with Activated carbon

AL-AC = Aluminium electrode coated with Activated carbon

11 X 7 Al-AC = 77 cm^2 area Aluminium electrode coated with Activated carbon

(25:57) Cu-AC = 25:57 ratio of Binder to Activated carbon Cu-AC

4.1 Observation:

Capacitor type	Discharge Resistor (R) (Ω)	V2 (V)	V1 (V)	Discharge time (Seconds)	Electrolyte used and concentration (M)	Capacitance (F)
CU-AC	100	0.55	0.032	52.18	1 M KCl	0.92884
AL-AC	100	0.52	0.10	72.6	1 M Na2SO4	0.9836129
AL-AC	100	0.09	0.00	13	4 M Na2SO4	0.26
AL-AC	100	0.17	0.05	56	1 M MgSO4	0.610909
CU-AC	100	0.936	0.00	16.09	1 M KCl	0.3218
AL-AC	100	0.25	0.13	45.22	1 M KCl	0.2856
Al-AC	100	0.25	0.13	45.22	1 M KCl	0.2856
SST-AC	100	0.25	0.09	41.32	1 M KCl	0.3888941
11x7 Al-AC	100	0.40	0.07	138	1 M KCl	1.9378
(25:57) Cu-AC	100	1	0.30	309.6	1 M KCl	3.33

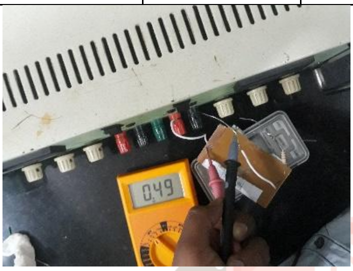

Figure 3(a)

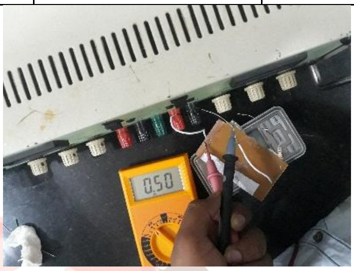

Figure 3(b)

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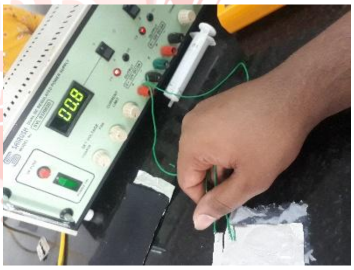
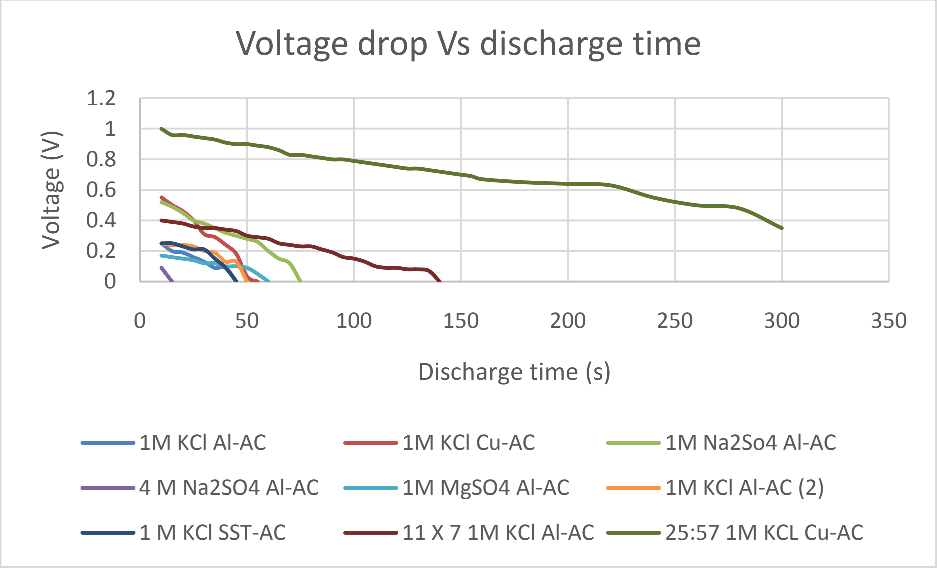


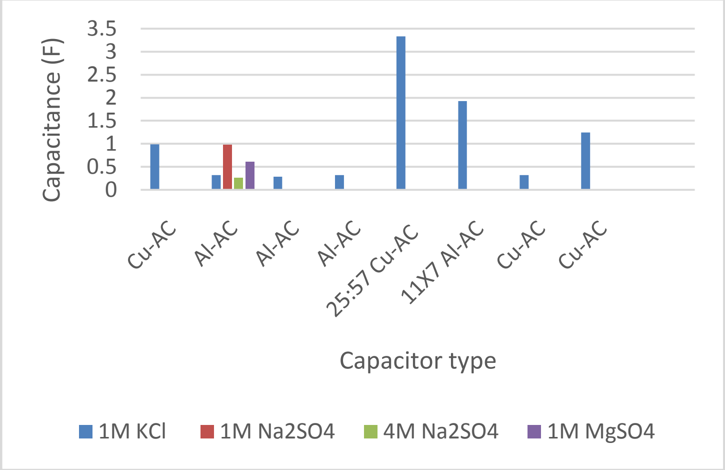
Figure 3(d)

Figure 3(e)

Figure 3(a) and 3(b) shows Cu-AC capacitor after charging, Figure 3(c) shows Al-AC capacitor after charging, Figure 3(d) shows Al-AC capacitor in charging, Figure3(e) shows Cu-AC electrode after usage

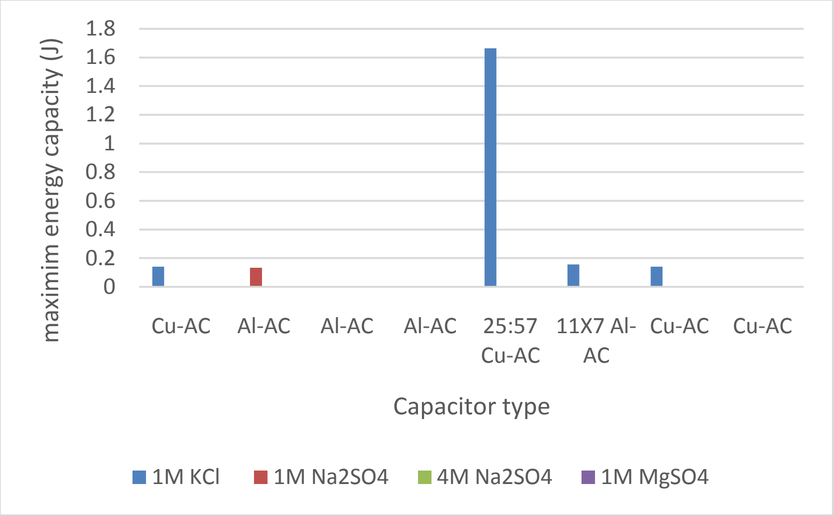


4(a)Voltage drop vs Discharge time Graph of different supercapacitor cell



4(b) Capacitance Vs capacitor type

Capacitor type	Electrolyte used and Concentration (M)	Internal resistance (Ri)Ω	Power loss (Watt)	Maximum energy Wmax (joule)	Effective energy Weff (joule)	Maximum power Pmax (watt)	Effective power Peff (Watt)
Cu-AC	1M KCl	12.33	0.0217	0.1404	0.1400	6.133 X 10^-3	3.0667 X 10^-3
Al-AC	1M Na2SO4	10.5	0.0168	0.1329	0.12806	6.438 X 10^-3	3.219 X 10^-3
Al-AC	4M Na2SO4	9	9 X 10^ -4	1.053 X 10 ^-3	1.053 X 10^-3	2.25 X 10^-4	1.125 X 10^-4
Al-AC	1M MgSO4	4.2857	3.35 X 10^-3	8.827 X 10^-3	8.063 X 10^-3	1.685 X 10^-7	8.42 X 10^-4
Cu-AC	1M KCl	23.63	3.7055	0.14096	0.14096	9.26 X 10^-3	4.63 X 10^-3
Al-AC	1M KCl	4	3.6 X 10^-3	8.925 X 10^-3	6.511 X 10^-3	3.906 X 10^-3	1.9531 X 10^-3
11X7 Al-AC	1M KCl	5.44	0.329665	0.155029	0.1471814	7.352 X 10^-3	3.676 X 10^-3
25:57 Cu-AC	1M KCl	11.532	0.0424	1.665	1.51515	0.0216	0.010839



5(a) graphical representation of energy capacity of different capacitor

5. Formula used to calculate capacitor parameter:

Internal resistance (Ri):

$$R_i = \frac{\Delta v}{I_d} (5)$$

Power loss (Pl):

$$P_L = R_i I^2 \qquad (6)$$

Maximum energy capacity (Wmax):

$$w_m = {}^1/_2\, C \cdot v^2 (7)$$

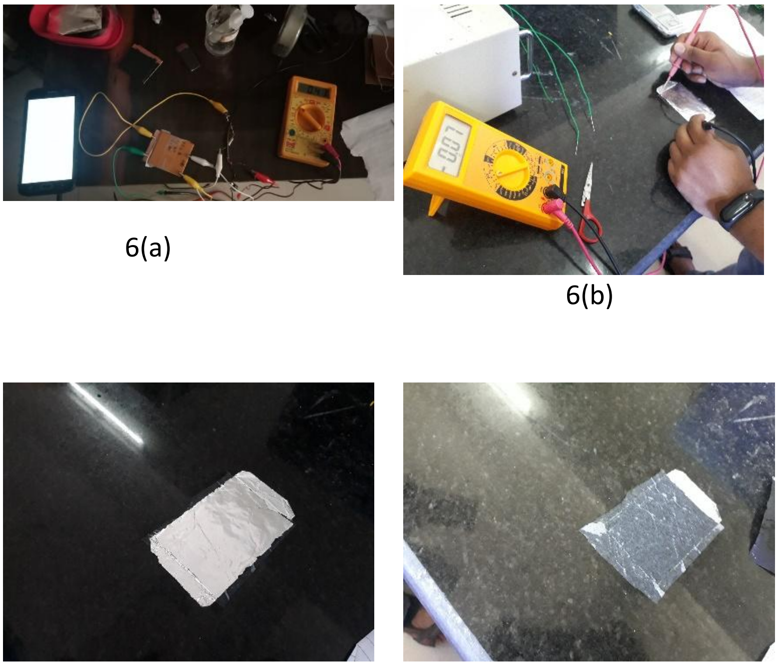
Effective energy capacity (Weff): $w_e = \frac{1}{2} c(v_2^2 - v_1^2)$ (8)

Maximum power (Pmax):

$$P_m = \frac{1}{4}\frac{v^2}{R_i} \qquad (9)$$

Effective power (Peff):

$$p_e = \frac{1}{8}\frac{v^2}{R_i} \qquad (10)$$



6(a)

6(b)

6(c)

6(d)

Figure 6(a) shows discharge of 25:57 Cu-AC, figure 6(b) shows Al-AC capacitor after discharge, figure 6(c) and 6(d) shows Al-AC capacitor

- 1M Sulphuric acid (H2SO4) was also used as electrolyte but electrode started reacting with sulphuric acid and hydrogen gas was produced. Degradation of electrode can be avoided by coating aluminium electrode with copper by the method of electroplating, as copper is less reactive towards sulphuric acid
- Resistivity of electrode material can be altered by changing binder to activated carbon (AC) ratio. In this experiment we used 1:3 ratio of binder to activated carbon, binder used in this experiment was Fevicol which has PVA (Poly vinyl acetate) as major component
- Experiment can be done using organic solvent instead of aqueous solvent which can be used with high voltage
- Different membranes can be used such as Kapton, Cellulose acetate, In this experiment we used single sheet of tissue paper and thick writing paper
- Thin sheet of tissue paper tends to tear during experiment causing unwanted arcing which causes thin aluminium foil to tear and degradation of electrode
- Thick writing paper performed quite well during test and was durable
- Graphene can be used instead of Activated carbon as intercalation in graphene would help increase the storage capacity by a lot
- [4Carbon nano tubes (CNT) can be used to increase capacitance
- Increasing area of the capacitor cell increases the capacitance significantly
- Better binder can be used instead of PVA (poly vinyl acetate) to decrease internal resistance and increase the effective power
- [9] Activated charcoal made of different material than coconut husk such as rotten carrots can be used to increase the porosity of activated carbon

CONCLUSION

By testing different electrode on electrolytes and comparing results we found out that 25:57 Cu-AC electrodes with 1M KCl as electrolyte and writing paper as separator material was the best performing supercapacitor cell, having capacitance of 3.33 F and max energy capacity of around 1.665 joules having discharge time of 5 minutes. The second-best Capacitor cell was 11 X 7 Al-AC which had capacitance of 1.937 F and max energy capacity of 0.155 J

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