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Fabrication and Analysis of EDLC Supercapacitors

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Abstract:- Supercapacitors with extremely high properties have been evolved and dominated the market so well that they surpass batteries and fuel cells. They hold high specific capacitance and energy density by making use of material which increases the surface area and thus specific capacitance. Thus, supercapacitor's fabrication using different electrolyte in gel form is presented. High dielectric materials like barium titanate and aluminium oxide are used in other capacitors to analyze them too. Firstly, emphasis on graphene synthesis is done and then gel electrolytes are used to make supercapacitor. Moreover, cyclic voltammemtry and charging discharging curves are produced.

Keywords:- *Supercapacitor, Gel Electrolyte, Super Dielectric Material, Graphene, Cyclic Voltammemtry.*

I. INTRODUCTION

Electric Double Layer capacitors are those class of supercapacitors in which graphene coated aluminium electrodes (to increase the surface area, for more charge accumulation) and electrolytes like H_2SO_4, KOH, H_3PO_4,NaOH etc. in gel form(sandwiched between two Al electrodes) are employed. The merging of the unique properties of graphene with new device concept and nanotechnology can overcome some of the main limitations of traditional electronic systems in terms of greatest linearity, energy density and power density. Graphene is reported to have unique properties like high electron and hole mobility, high surface area, zero band gap and carriers confinement to a one atom thick layer only making it extremely flexible and transparent. There are several methods to synthesize graphene like Solution-based reduction of graphene oxide, Chemical vapor deposition (CVD), Micromechanical exfoliation of graphite and Epitaxial growth on electrically insulating surfaces. The unfussiness and straightforwardness of chemical method is adopted by most of the researchers. Gel Electrolytes employing Poly Vinyl Alcohol are generally used to make supercapacitors. The intention of using electrolyte in gel form is to make good contact with graphene electrode. This scarcely modifies graphene transparency . The various electrolytes such as and similar to H_3PO_4, H_2SO_4, KCl, NaCl, NaOH and KOH can be used. H^+ ion's ionic radius in the H_3PO_4/H_2SO_4-PVA electrolyte is very much smaller than that of the ions (Na^+, K^+, OH^-, Cl^-) from the base used in the gel electrolyte . The H+ ions can diffuse conveniently and speedily between the layers of graphene . Although, ions, such as Na^+ or K^+ which are heavier, could only reach the surface of the graphene electrode during charging and the discharging while H_3PO_4 can generate more free ions than NaOH or NaCl with the same molar concentration. For electrolytes having a low ion concentration, the supercapacitor's capacitance enhances with the ion concentration in the electrolyte

II. MATERIALS AND METHODS

Graphite fine powder (Extra pure), Ortho Di-Chloro Benzene (ODCB) with CAS: 95-50-1 were used without any dilution or distillation. The other reagents like Trichloroethylene (TCE), Sulphuric acid (H_2SO_4), Hydrogen Peroxide (H_2O_2), Electrolytes (KOH, H_2SO_4 and H_3PO_4), PVA, aluminium Sheet, Methanol and De-ionized (DI) water were used. Techniques like ultrasonication, centrifugation, spin coating, XRD, UV-VIS Spectroscopy and cyclic voltammemtry were used.

III. EXPERIMENTAL PROCEDURE

The procedure to carry out the experiments would be discussed in this section.

A. Graphene Preparation

- A simple chemical approach is used to obtain stable homogenous dispersion of graphene [16]. In a typical process, graphite (0.1 gm) was mixed with ODCB (100ml) in a glass beaker.
- The mixture was sonicated in an ultrasonic bath (frequency: 33 KHz & power: 150 W) for 12 hours at 50-60°C. Then the sample in dispersion form was kept in vial for overnight and the supernatant was transferred to another vial.
- This dispersion was next subjected to 30 minutes centrifugation at 7000 rpm to remove the unexfoliated part of graphite.
- It was observed that the heavier particles got settled down and were removed from the dispersion. The dispersion was then heated to reduce its volume by 50% so as to make it more viscous and thermodynamically stable.
- This solution was preserved for further processing. The n-type oxidized silicon wafer and pyrex glass pieces (2.5 x 2.5 cm^2) were cleaned by organic solvents TCE and methanol and then rinsed in DI water for ten minutes.
- Then the samples were cleaned in a mixture consisting of H_2SO_4 and H_2O_2 in the ratio of 3:1 for 10 minutes (Piranha cleaning) and then rinsed in DI water for 15 minutes.

- The samples were dried in oven at 100 °C for 30 minutes and the above dispersion solution was deposited on cleaned samples by spin coating at a rate of 2000 rpm for 30 seconds. The coated film was dried at 90 °C for 15 minutes in the oven.

- This process was repeated seven times to obtain the desired thickness of the film. Finally the prepared samples were sent for XRD and UV-VIS spectroscopy.

B. Gel Electrolyte Preparation

- In order to prepare gel electrolyte, PVA (3.5 gm) was dissolved in DI water (50 ml) via ultrasonication for 30 mins and then heating it for 10 minutes at 80-90°C.Similarly KOH(2.8 gm) and DI water(50ml) solution was prepared by ultrasonicate this mixture for 30 minutes.

- After that KOH solution (9 ml) was added in (25ml) PVA gel and ultrasonicated it for 1 hour. In an another approach to get better results (1 gm) PVA and (0.2 gm) KOH in (10 ml) DI H_2O were dissolved after sonicating it for 30 minutes. Further investigations were done by using (0.2 M) of H_2SO_4 and H_3PO_4 for the same ratios of DI water (10 ml) and PVA (1 gm).

- This time PVA was added slowly in order to get uniform gel and was ultrasonicated for 45 minutes. This time a better quality of gel was formed. Different analysis were done on these gel electrolytes depending upon the various parameters like ultrasonication time, Coating or number of layers deposited on electrode using spin coating, thin layer consideration etc.

C. Electrodes Preparation

- This shape of electrode was chosen so as to take contacts easily for measurements. The electrodes were cleaned using organic cleaning and then Pirahana cleaning.

- In organic cleaning, TCE was taken in beaker and the electrodes were boiled in it for 10 minutes. Then these electrodes were rinsed in methanol (3 minutes) and swaping with tissue was done to remove the stains or marks on them. Then wash them with DI water (5 minutes).

- Next Piranha cleaning was done using H_2SO_4 and H_2O_2 in 3:1 ratio. All the electrodes were kept in this mixture for 2 minutes and then washed with DI water 4 times. Then these electrodes were kept in the oven to dry at 150°C for 30 minutes. As prepared graphene was coated on all the cleaned electrodes using spin coating using d.c. motor at 160 rpm. Electrodes were kept in oven to dry at 150°C for 2 hours.

D. Making Supercapacitors

In this section, the process of making supecapacitor using already prepared graphene, gel electrolyte and aluminium substrate is presented. The following are the steps to make supercapacitor.

- Take out the graphene coated Al electrodes and as prepared gel electrolyte.

- Now apply gel on one electrode and spin coat it for 30 seconds at 200 rpm using spin coater.

- Only a thin layer of gel electrolyte is to be applied on the electrode and it should be kept in mind that the gel electrolyte should cover whole surface area of graphene coated Al electrodes.

- Now the second electrode is to be kept over the electrolyte coated electrode as shown in figure.

- Seal this fabricated supercapacitors with tape so that the electrolyte do not leak.

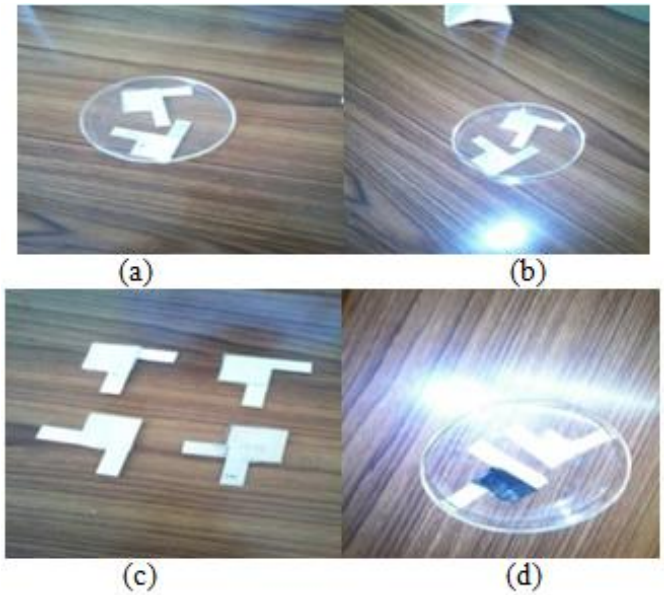

Fig 1:- (a-c) Images of EDLC Supercapacitors , (d) $BaTiO_3$ and SDM capacitors

IV. THEORY AND CALCULATION

Various CV curves at different scan rates are analyzed and depending upon the analyzed results some conclusions have been drawn. The curve so obtained through CV measurement depicts the storage capacity or capacitance of the supercapacitor. The area under curve, scan rate, average current, weight of the active material, and the voltage window are certain parameters which are to be noted and calculated from CV curve to know capacitance, energy density and power density of supercapacitor. Using the following equation one can calculate capacitance as

$$C = \left(\frac{\int Idv}{\left(\frac{dv}{dt}\right) * M * V} \right) \qquad 1$$

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Where C= Specific Capacitance, dv/dt=scan rate, M=weight of the active material on one electrode and V=voltage window (1V).Charging Discharging curves depicts the time taken by the capacitor during charging and discharging action. One can use these graphs data to find out the specific capacitance. One can calculate Specific Capacitance using the equation involving charge discharge curves

$$C = \left(\frac{I}{\frac{dv}{dt} * M}\right) \qquad 2$$

Where I= constant current during charge discharge, dv/dt=scan rate and M=weight of one of the active electrode material. Also one can calculate and have estimate of the energy density and power using the following equation

$$E.D = \frac{1}{8} CV^2 \qquad (3)$$

where E.D= Energy Density inWhKg-1, C=Specific Capacitance(F/g) and V=Voltage Window.

$$P.D = \frac{E.D}{V} . \frac{dv}{dt} \qquad 4$$

Where P.D=Power Density in KWg^{-1}, V=Voltage Window, dv /dt=scan rate and E.D=Energy density.

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V. RESULTS AND DISCUSSIONS

As depicted in table I various observations are noted during the experimental process of synthesizing graphene .Apart from the chemical reaction of these organic solvents on graphite chemistry , there are some physical changes that occurred .When comparing NMP and ODCB ,it is finally observed that ODCB was found to give better results due to its high surface tension ($36.01 mJ/m^2$) and is a high boiling solvent with boiling point at 180°C.Also it can be visualized from the table above that it provides better results as is confirmed from XRD ,UV-VIS and SEM results. The sheet resistance and thus resistivity is calculated using four probe method with V/I ratio observed 3.5317 ohm. The formula for calculating resistivity is $\zeta = R_s \times t$, where Rs=4.53xV/I (4.53 is the constant used for equidistant probes of Four probe apparatus), t is the thickness of the material coated, ζ is used to denote resistivity. Conductivity is the reciprocal of resistivity. Moreover, electrical conductivity of graphene made from ODCB is calculated as 1.5×10^6 S/m with sheet resistance equal to 16 ohm/square. The weight of graphene calculated is 0.0011g.

Parameters	NMP	ODCB
Sonication(50Hz,150 watt) for 4 hours	No results obtained	Graphite remains unexfoliated
Sonication(50Hz,150 watt) for 12 hours	No satisfactory XRD results obtained	XRD confirms formation of GO
Colour change	Turned reddish-brown after 12 hours	Turned into Grayish solution after 12 hours
When kept overnight after centrifugation	No homogenous solution observed	Homogenous solution observed as no further settling down of particle occurred
After 15 days	Turned black again	Remained grayish and as it is even after 1 month
UV-VIS	near about peak observed as mentioned in literature	UV- VIS also proved the formation of GO

Table 1:-Differences Observed Using NMP and ODCB Solvent

Figure 2 demonstrates the XRD results of natural graphite powder in which diffraction peaks corresponds to crystallographic orientations (002), (100) and (101) etc. at angles 2θ = 26.4°, 42.17° and 44.96° respectively (Standard RRUFF Data Book).The formation of homogenous dispersion of graphene in ODCB can be explained through solvent-graphite interaction. Sonication leads to the separation of graphite layers and once the graphite sheets are separated, solvent molecules (ODCB) penetrate inside the interatomic layers of graphite and adhere to graphite layers. This leads to the formation of sonopolymer. Moreover ODCB is known to decompose during sonication to liberate chlorine which can easily penetrate inside graphite layers. With increasing sonication time the formation of polymer bounded graphite sheets increase as well as distance between the graphite layer also increases and exfoliation of graphite takes place.

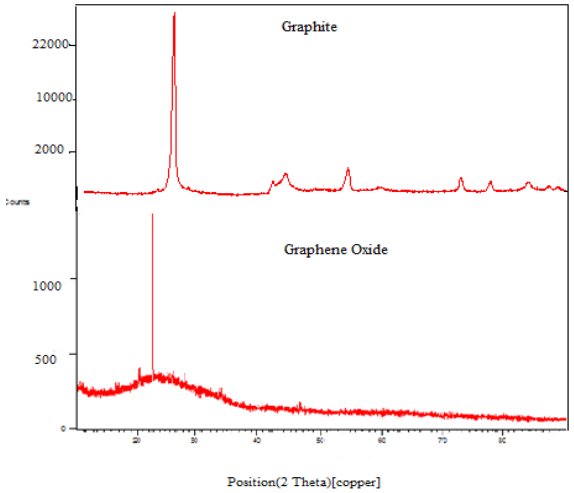

Fig 2:- Comparison of Natural Graphite and Exfoliated Graphite

The figure clearly shows that the most intense peak (002), is at 2θ = 26.4° corresponding to d-spacing of 0.335 nm of the original graphite disappeared after exfoliation, while an additional peak at lower angle at 2θ = 11.3° appeared corresponding to the (001) diffraction peak of graphene oxide (GO) and the d-spacing also increased to 0.784 nm. Another intense peak at 22.8° shows the formation of graphene [1, 17]. These results are in close agreement with results reported earlier in literature. The grain size of nano particles of graphene at different diffraction peaks varies from 32 to 84 nm as calculated from Scherrer's formula. These results also show the crystalline nature of the exfoliated graphite.

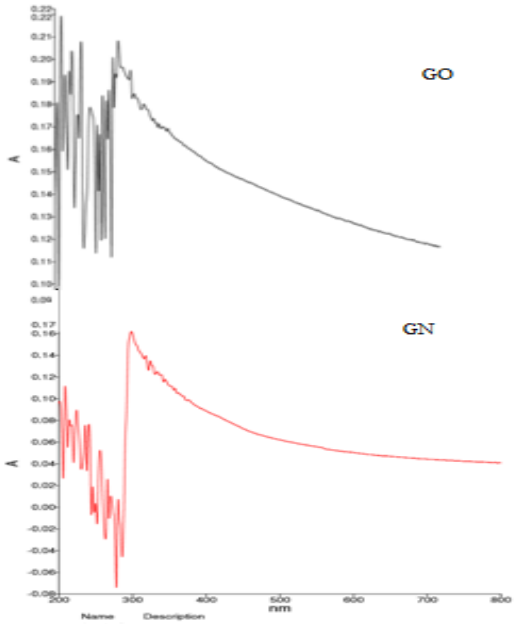

Fig 3:- UV-VIS Comparison of Graphene obtained from NMP(GN) and ODCB(GO)

The XRD results of ODCB dispersion thus prove the successful synthesis of GO and graphene nano particles/sheets. The figure 3 shows the absorbance peaks at various wavelengths in the case of ODCB suspension and in NMP. The GO and graphene formation with sonication method can also be described using UV-VIS spectroscopy studies for the optical absorption range from 200 nm to 800 nm as shown in figure 3. The figure 3 shows the results obtained from ODCB suspension [1, 18]. A sharp peak at 210 nm can be seen and one more peak around 226 nm with a little bit less intensity of absorption peak is also observed due to Π-Π* bondings of the C-C aromatic rings [19]. The absorption peak around 300 nm is shown in comparison and with further increase in wavelength the absorption decreases this confirms the exfoliation of graphite in NMP too.

Electrode	Dielectric/Electrolyte	Capacitance at 100Hz	Areal Capacitance
Aluminium	Air	6.3pF	1.008 pF/cm^2
Aluminium	Paper	22pF	3.52 pF/cm^2
Aluminium	Al_2O_3	225 pF	36 pF/cm^2
Aluminium	$BaTiO_3$	117 pF	18.72 pF/cm^2
Graphene Coated Al	Air	51 pF	8.16 pF/cm^2
Graphene Coated Al	Al_2O_3+NaCl+H_2O	15 µF	2.4 µF/cm^2
Graphene Coated Al	PVA+KOH(1M) gel	11.35nF	1.816 nF/cm^2
Graphene Coated Al	PVA+KOH(0.2M)	6.5 µF	1.04 µF/cm^2
Graphene Coated Al	PVA+H_3PO_4(0.2M)	9 µF	1.44 µF/cm^2
Graphene Coated Al	PVA+H_2SO_4(0.2M)	435µF	69.6 µF/cm^2
Graphene Coated Al	PVA+H_2SO_4(0.2M) (Gel coated on single electrode)	2949 µF	471.84 µF/cm^2

Table 2:- Capacitance of Different Capacitors Measured Using LCR Meter.

The table 2 shows the capacitance measured through LCRQ 6018 Bridge meter of different Capacitors. It can be seen that the lowest capacitance observed was in the case of air capacitor and the highest in case of Super dielectric Material. However, 5 more capacitors were measured using LCR meter by making use of graphene for active electrode material. Here, PVA and electrolyte gel(H_2SO_4(0.2M), H_3PO_4 (0.2M) and KOH(1M,0.2M) was used to realize the supercapacitor concept. Further, capacitor's can be checked and its capacitance can be measured using CV analysis which is discussed in next section.

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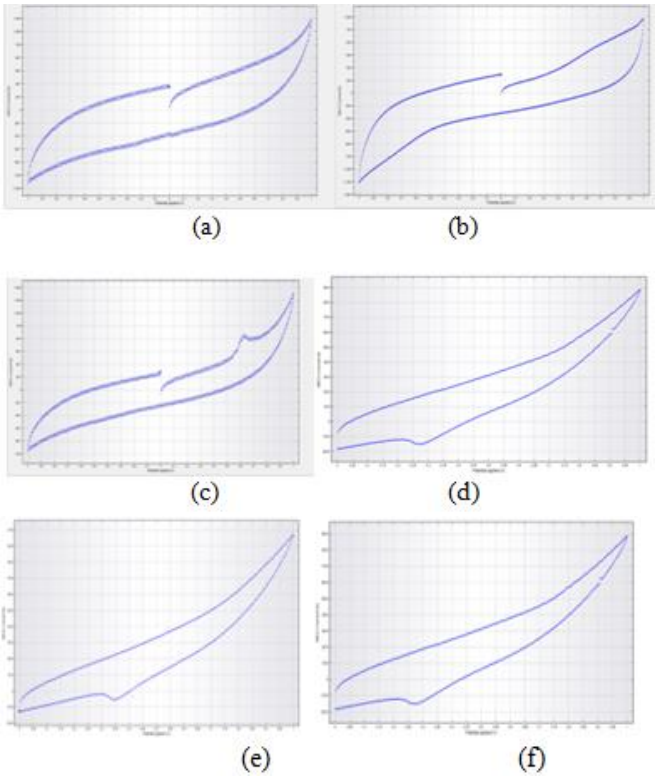

Fig 4(a-f):- CV characteristics of KOH, H_3PO_4 and H_2SO_4 supercapacitors at different scan rates.

In figure 4 (a-b) Supercapacitors (made using KOH with 1 M and 0.2 M concentration) CV curve at scan rates at 500mv/s and 300mv/s are shown. Similarly, figure 4(c-d) demonstrates the CV curves for H_3PO_4 at 100 mv/s and 200 mv/s. It can be observed that a small deviation of staircase curve is obtained on the left of graph which confirms the formation of double layer.The figure 4(e-f) shows the area under the CV curve for H_2SO_4 gel electrolyte with molar concentration 0.2 M used to make supercapacitor(SC5). This time it was observed that the CV curve couldn't be traced at scan rates higher than 50mv/s. And for the smallest scan rates only 10 mv/s and 20 mv/s the curves are obtained. Above 50 mv/s the curve sweep behaves abruptly and a broadened curve shape without any appreciable area is obtained. This is due to instability of Sulphuric acid.

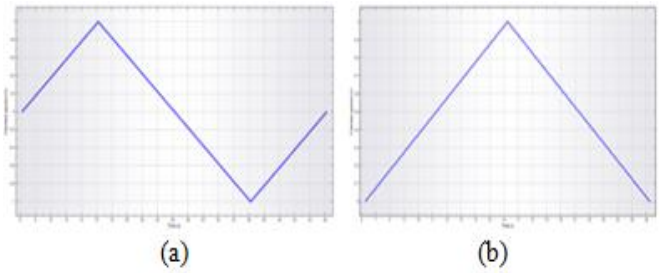

Fig 5 (a, b):- Charging Discharging curve for KOH and H_2SO_4 Supercapacitors at 100 mv/s

The specific capacitance calculated at different scan rates for different gel electrolyte capacitors are noted. With SC2 at 300mv/s 3.97 F/g, at 500mv/s 2.245 F/g and 1.5495 F/g obtained. Similarly SC 3 at 100mv/s shows 7.15 F/g, 4.74 F/g at 200mv/s, 3.429 F/g at 300mv/s and 3.09 F/g at 400mv/s specific capacitance.SC 4 when measured at100mv/s produced 8.424 F/g and 4.0055 F/g at 200mv/s. While SC5 generated high specific capacitance at low scan rates i. e 80.45 F/g at 10mv/s, 51.71 F/g at 20mv/s and 8.34 F/g at 50mv/s respectively. It can be observed that the specific capacitance of SC 5 is exceptionally high as compared to gel electrolyte supercapacitors. And the lowest capacitance is observed in case of SC 2 with scan rate 1000 mv/s. The only difference in analysis found is that SC 5 shows result at lower scan rates (10mv/s to 50mv/s) comparing other supercapacitors formed.

Supercapacitor	Specific Capacitance(F/g)	Energy Density ($WhKg^{-1}$)	Power Density (KWg^{-1})
1 (KOH 1M)	3.97 F/g	0.4962	0.14886
2 (KOH 0.2M)	7.15 F/g	0.8937	0.08937
3($H_2SO_4$0.2M)	8.424 F/g	1.053	0.1053
4(H_2SO_4 S.C)	80.45 F/g	10.05	0.1005

Table 3:- Comparison of Different Supercapacitors in Terms of Specific capacitance, Energy Density, and Power Density.

One can easily compare the difference in specific capacitance, energy density and power density of various supercapacitors as shown in table 4.6. The highest energy density is found to be 10.05 $WhKg^{-1}$ in SC5 and SC2 found to have the highest power density of 0.14886 while SC2 has the lowest energy density of 0.4962 and SC3 possessing the lowest power density.

VI. CONCLUSION

The graphene synthesized possess the sheet resistance of 16 ohm/sq while the resistivity calculated is found to be $6.410312e^{-5}\Omega$/m. The conductivity of the film obtained is 1.5×10^{-6} S/m. Based on the work done and practically achieving the results is concluded that supercapacitors made up of H_2SO_4 gel electrolyte among all other gel supercapacitors possess more charge storing ability with high specific capacitance of 80.45 F/g and areal capacitance of 471µF/cm^2 with energy density 10.05 $WhKg^{-1}$. However, the power density of H_2SO_4 is 0.1053 KWg^{-1} lower than

KOH supercapacitor. The super dielectric material concept is also analysed and it is noted that SDM capacitors possess second most highest areal capacitance of 2.4 $\mu F/cm^{2.}$ .To summarize one can conclude that graphene is both quantitatively and qualitatively different from any other material conventionally used in electronic applications. The idea of using it to make active electrodes of supercapacitor results into production of highly efficient device. Further modifications can lead to achieve better results.

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