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Synthesis and Charcterization of Expanded Graphite based Nanocomposite using Phenolic Resin as a Bipolar Plate for Polymer Electrolyte Membrane Fuel Cell

Ravinder Kumar[1], Surender Kumar[2], Pawan Kumar[3]

[1] Lecturer H. E. S (II) GSSS Narnaund, Hisar, Department of Physics, Department of Secondary Education, Haryana, India
[2] Lecturer in Physics, Department of Physics S. D. Modern Public school, Hansi (Hisar) Haryana, India
[3]Lecturer H. E. S (II) GSSS Sarsod, Hisar, Department of Physics, Department of Secondary Education, Haryana, India

ABSTRACT

Expanded graphite-based nanocomposites as bipolar plates for polymer electrolyte membrane fuel cells (PEMFCs) are developed by compression molding technique. The expanded graphite is prepared by chemical intercalation of 50 +100 BS mesh particle size of natural graphite by strong acid. It is found that composite bipolar plate with a varying weight percent of resin and EG gives the high electrical conductivity, competitive mechanical properties with a bulk density of 1.55 -1.56 g/cm-3, and air tightness. The EG-based composites with 50 -60 wt % of resin are suitable for achieving desired properties of the bipolar plate as per the Department of Energy(DOE) advanced series target. The addition of 3- 5 wt % of carbon black fibre is helpful for improving the electrical conductivity with out compromising other properties of the bipolar plate. The lower value of the modulus of expanded graphite(3 -8 GPa) composite as compared to graphite-resin-based composite (>12 GPa) plates attributes that these plates are more flexible and able to withstand shock and vibration during mobile operation of the fuel cell .

Keywords: Molding, Intercalation, Resin, Expanded Graphite.

1. INTRODUCTION

Fuel cells directly and efficiently convert chemical energy to electrical energy. During the last decade, fuel cells have received enormous attention from research institutions and companies as novel electrical energy conversion systems. In the near future, they will see application in automotive propulsion, distributed power generation, and in low power portable devices (battery replacement).Polymer electrolyte membrane fuel cells (PEMFCs) are the most promising power sources for various portable electronic devices, stationary and mobile applications[1]that operate at low temperatures between 70 and 80 °C. The main components of the PEMFCs are electrolyte membrane, gas diffusion layer, platinum catalyst, and bipolar plate. The bipolar plate is one of the most important components of PEMFC, which accounts foras much as 60 -80% of the fuel cell stack weight[2]and 40 -50%of the total cost of the fuel cell stack. The U.S. Department of Energy (DOE) advanced a series of property requirements of the resin/graphite composite for the bipolar plate, in which the main properties are electrical conductivity (100 S/cm) and bending strength (25 MPa), respectively[.3]The conventional materials for producing the bipolar plate are graphite or metal. Graphite plates are preferred over the metallic plates because of their high corrosion resistance and lower density[.4]On the other hand, disadvantages are difficulties in machining and itsbrittleness.[5,6]Because of this reason, a graphite-based bipolar plate requires a thickness of the order of several millimeters and causes the fuel cell stack to be massive and voluminous[.7]These considerations have motivated researchers to develop alternative materials.

Graphite-based polymer composite bipolar plates have the potential to replace graphite plates; they offer the advantages of greater ease of manufacturing than graphiteplates[.8]However, polymer composites are associated with problems of electrical conductivity that remain to be solved; therefore, excessive graphite fillers have to be incorporated to the composites to meet the minimum requirement of electrical conductivity. High graphite loading in the polymer composite substantially reduces the strength and ductility of polymer composites, and this issue may be considered. There are some studies reported in the literature on bipolar plate made from expanded graphite (EG) for PEMFCs,9–13and more studies are available on the improvement of physical and electrical proper-ties of various polymers14–20by incorporating a few percentage of EG. Heo et al.[9]reported the development of bipolar plate made from EG by preform molding technique.

The electrica conductivity and flexural strength increase as the stamping pressure and curing time increase and reach the maximum value. Xiao et al.[10]reported the properties of bipolar plate made frompoly (arylene disulfide)/graphite nanosheets. In this study, graph-ite nanosheet composites are fabricated using ultrasonic irradia-tion that possesses a flexural strength of 32.05 MPa and an electrical conductivity of 158.7 S/cm. Bhattacharya et al.[11]studied the EG as an electrode material for an alcohol fuel cell and found that the EG electrode has efficient electro-catalytic activity than an unexpanded graphite electrode because of the nanochannel formed after expansion. Yan et al.[12]used E Gbipolar plates is in PEMFC stack, and it was found that the performance of the stack was excellent. Kalaitzidou et al.[13]studied morphology and mechanical properties of exfoliated graphite nanoplates -polypropylene nanocomposites fabricated by melt mixing and injection molding. Therefore, in the present investigation, efforts are paid to use the reasonable amount of EG instead of natural graphite (NG)in the polymer to obtain the requisite properties. As compared to conventional NG, EG has unique properties because it is produced from graphite flakes intercalated with highly concentrated acid, which can be expanded up to a few hundred times their initial volume; the expansion leads to the separation of the graphite sheet into nanoplates with a very high aspect ratio These have many unique properties, such as excellent electrical and thermal conductivity because of the layered structure. Meanwhile, because of porous structure, it is possible for the polymer to intercalate to produce EG/polymer nanocomposites

2. EXPERIMENTAL DETAILS MATERIALS USED

2.1. Synthesis of Expanded Graphite

Synthesis of EG. The NG flake of size -50+ 100 BS mesh was dried at 75 °C in a vacuum oven for5hto remove the moisture content. It was mixed and saturated with acid consisting of concentrated sulfuric acid and nitric acid in a ratio of 3:1 for 10 -15h to form the graphite-intercalated compound (GIC). The mixture was stirred from time to time to obtain the uniform intercalation of each flake. The mixture was carefully washed and filtrated with water until the pH level of the solution reached 7. After washing, the acid-treated flakes dried at 60 °C in a vacuum oven. GIC was rapidly expanded at temperatures between 800 and 900 °C for 10-20 s in a muffle furnace to form EG.

2.2. Development of composites:

In this study composite sample of size 60 mm × 20 mm × 4 mm was prepared by compression moulding technique with varying the ratio of resin and expanded graphite. Figure 1 shows the flow diagram of composites preparation. The expanded graphite and resin powder was mix uniformly with the help of mixture and grinder. Then the mixing composition is filled in a die mould of preferred dimension and then it is placed in a moulding press (or hydraulic press) and allows it to raise the temperature up to 100°C. The composition is pressed in between the temperature of 90-100°C. After that it is kept at 150°C constantly for 2 hour and finally the press is switched off. After normal cooling we can get the desired sample of expanded graphite bipolar plate. The Schematic diagram of formation of composite is shown below .

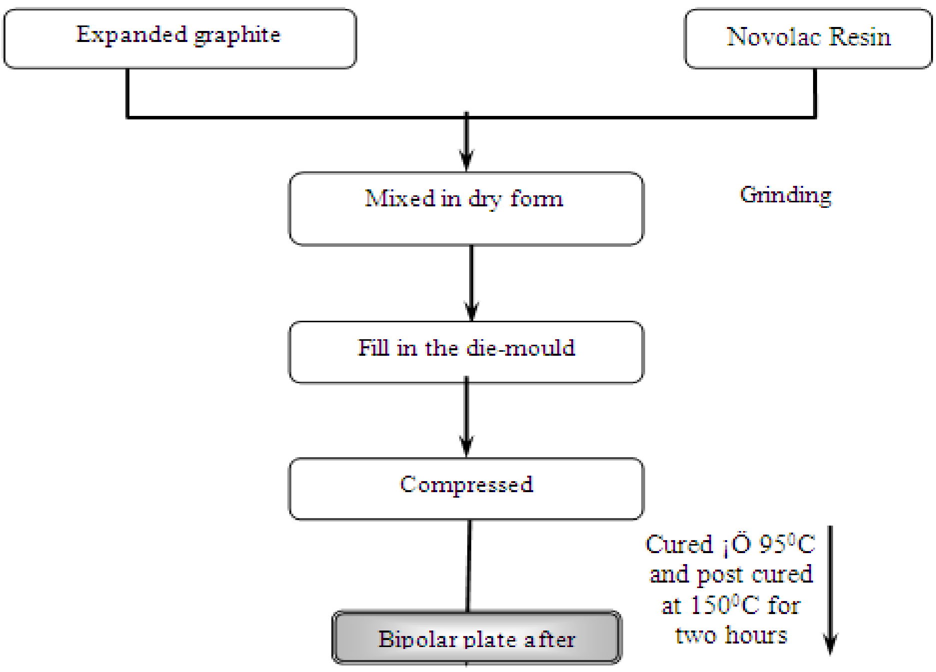

FIGURE-1 **Block diagram of composite preparation**

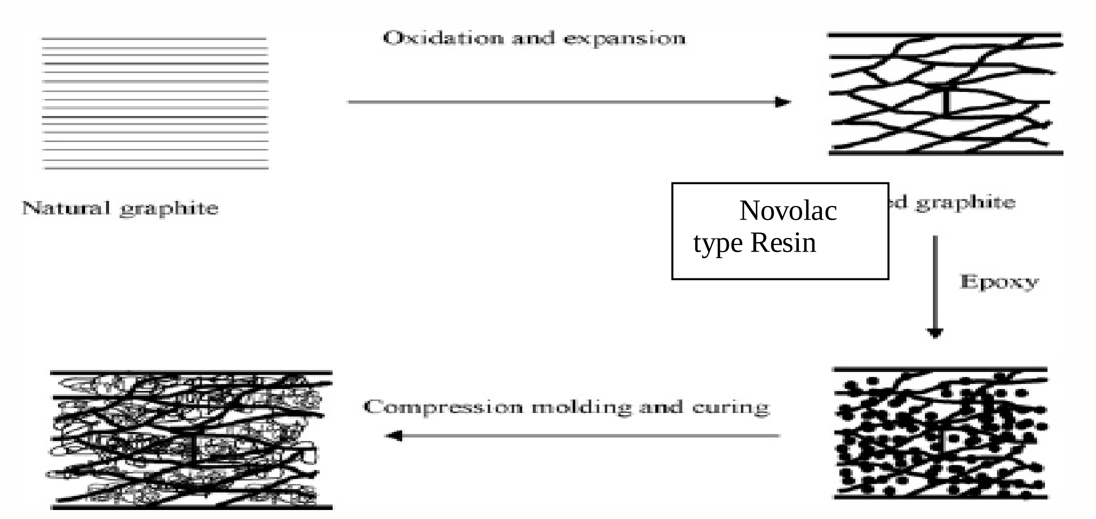

Figure 2: Flow diagram of composite preparation

3. RESULTS AND DISCUSSION

3.1 FTIR of Phenolic Resin:

Figure 3. **shows the FTIR spectra of Novolac Phenolic resin. The absorption band at 3334.7 cm^{-1} is of hydrogen bonded OH group. The peak is 2925 cm^{-1} is of $-CH_2$- stretching vibration. The peaks in between the 1455 – 1595 cm^{-1} are of C-C stretching vibration. The peak at 1235 .9 cm^{-1} is of Ar-O^- stretching vibration and peak at 1101 cm^{-1} is of CH_2-OH stretching vibration. This suggested that Novolac phenolic resin having hydroxyl and methylene group's linkages that are responsible for bonding with the expanded graphite in this study.**

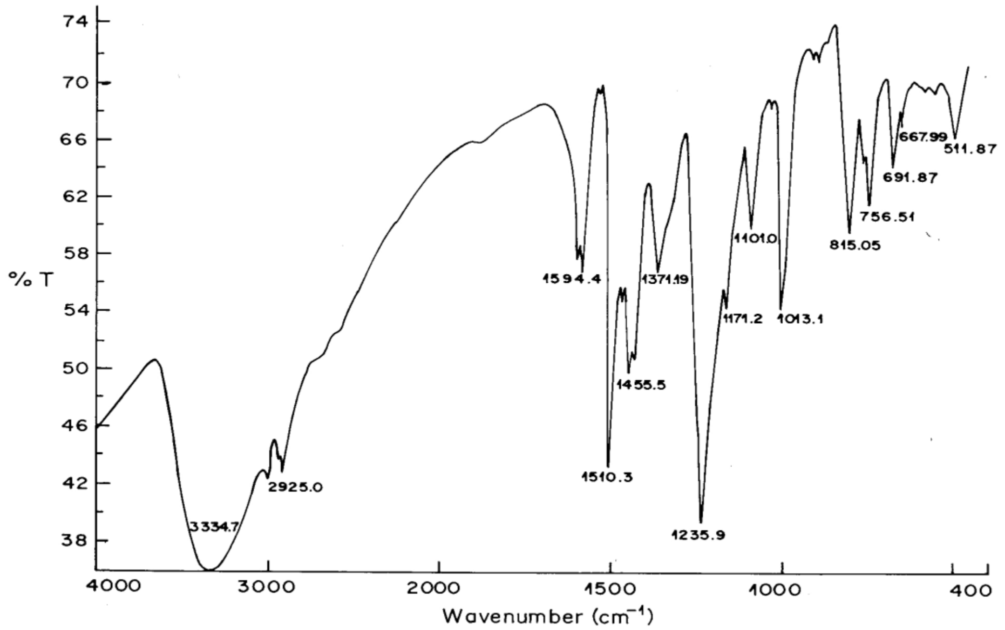

Figure 3: FTIR of novolac phenolic resin

3.2. Variation of Density with Different Properties like Resistivity, Conductivity, Hardness, Strength, Modulus.

3.2.1 DENSITY

The slightly difference in theoretical density and experimental density due to the loss of resin content in composite . Figure 4.shows the variation in the theoretical density with experimental density . The maximum theoretical density is 1.6 g/cc and minimum iss 1.4 g/cc . In this study ratio of expanded graphite and resin simultaneously increase with same volume in all cases .

The theoretical density increases from 1.4 to 1.6 g/cc in which ,in same volume content increase and porosity may decrease . This mean that at same molding pressure composite developed with different EG can possess the porosity means that these composites are less rigid.

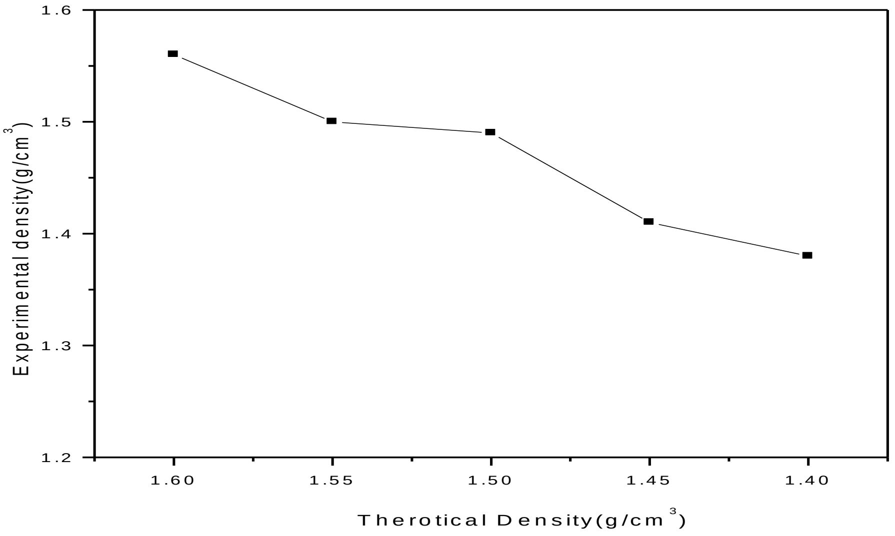

Fig 4

The variation also might be due to the experimental error as well .Fig shows the bulk density of EG composite calculated by taking the volume and weight . The Theoretical density is addition of material density , such as resin &EG taken before development of composite.

3.2.2. THE BENDING STRENGTH

The bending strength of EG composite are decreases with decreasing density because of the resin % in the matrix which is the called as percolation .The resin %wt in composite get losses while compression in moulding press due to which there is variation in the density .Because of that, bonding with EG (Expanded Graphite) is loose with density variation .There are different composition for each sample of desire density makes the sense of decreasing bending strength.The resin content is in the ratio of 45% with that of EG 55%and for the different therotical density i.e..from 1.4 to 1.6 g/cc.

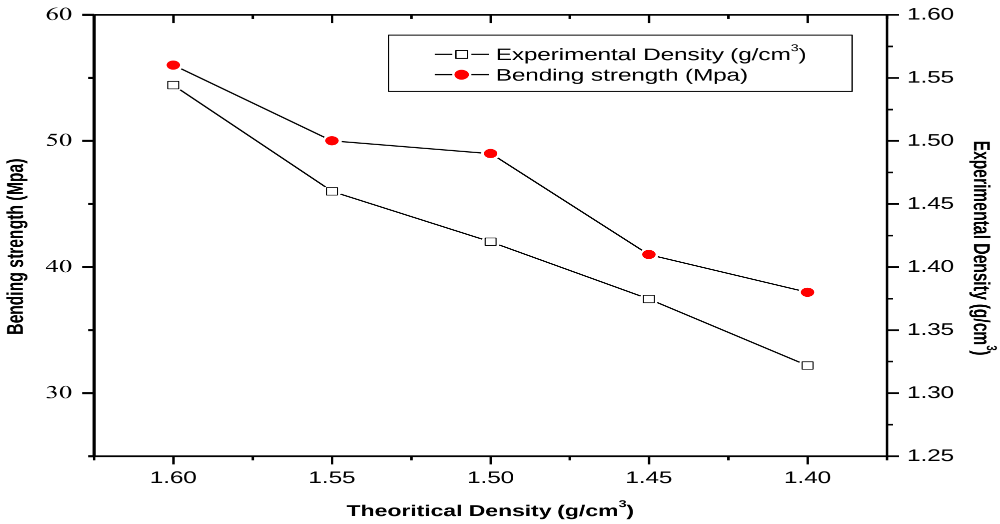

Fig5

3.2.3 SHORE HARDNESS

The shore Hardness of EG composite are due to the bonding b/w resin and EG ,which makes the composite hard after heat treatment .From fig.6. it is clear that , the hardness of EG composite is decrease with density

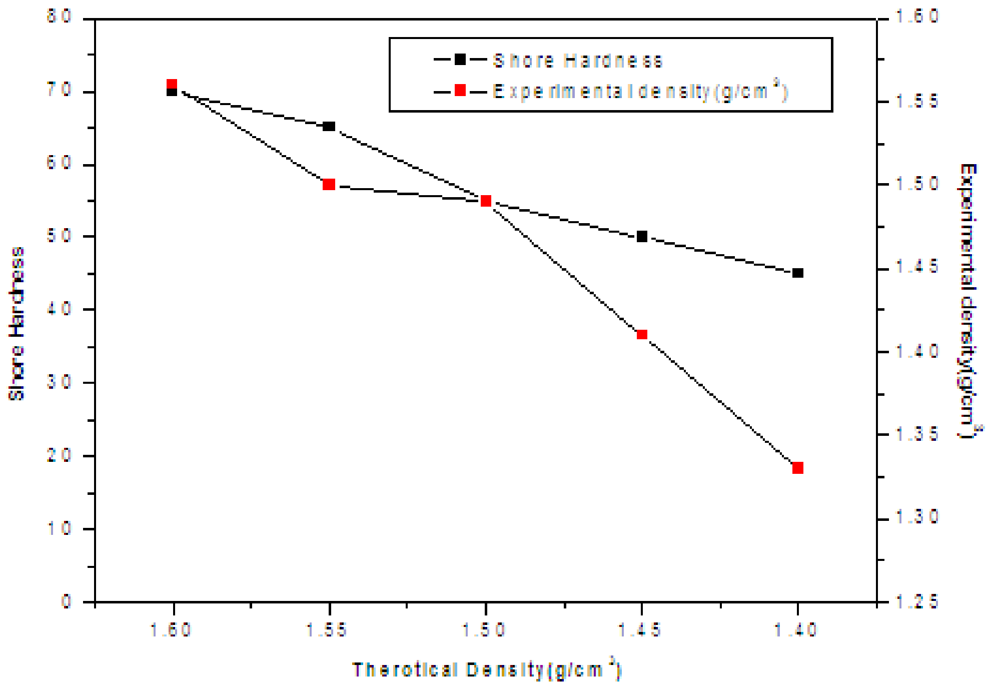

FIG.6

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3.2.3 Conductivity

The conductivity of the composite is decrease with decrease in density . The varation shownn in fig.7. due to the reason that conductig phase of EG composite has large conductiviy and resin insulatin material .The bonding b/w the two makes conductivity decreases with density.

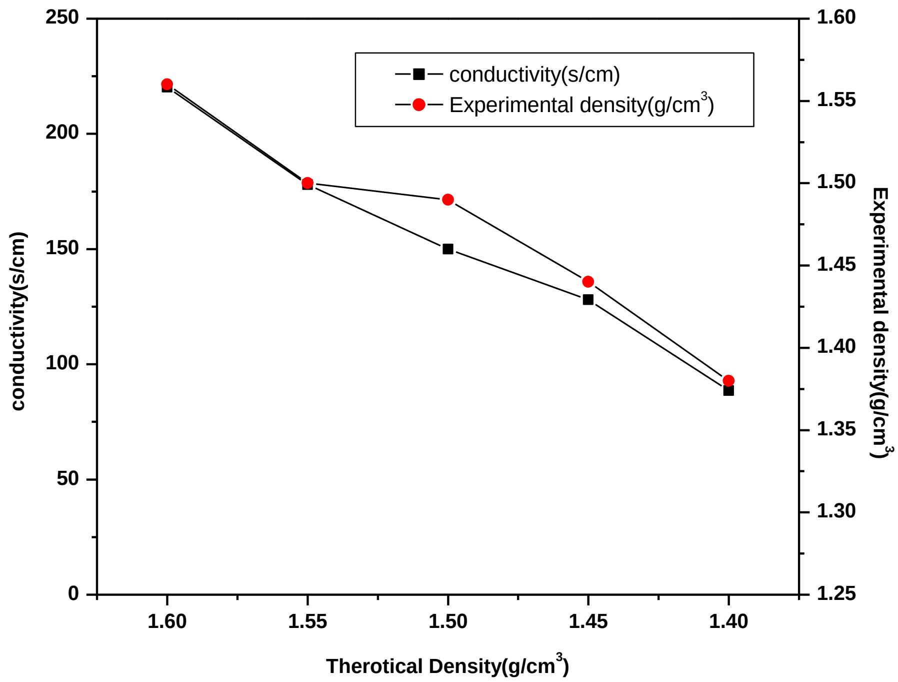


Fig 7

3.2.4 MODULUS Modulus of EG composite is decreases with density due to the varying resin content. This is due to the reason that the surface area of expanded graphite is large, which makes the large volume and hence decreases the modulus.

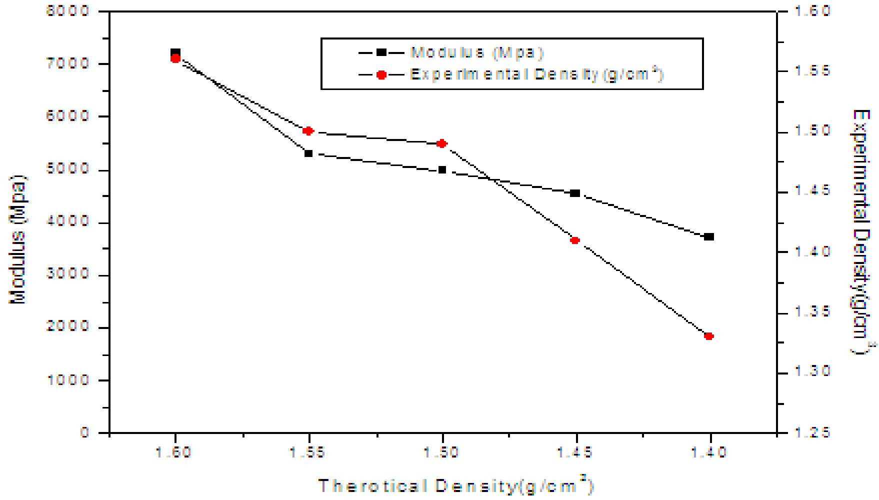


Fig 8

SEM micrograph of expanded graphite Nanocomposites

Fig9 (a) shows SEM micrograph of EG phenolic resin composites in which the most of the EG particle are bonded with resin matrix .in some region due to lower content of resin some properties are separated from each other.

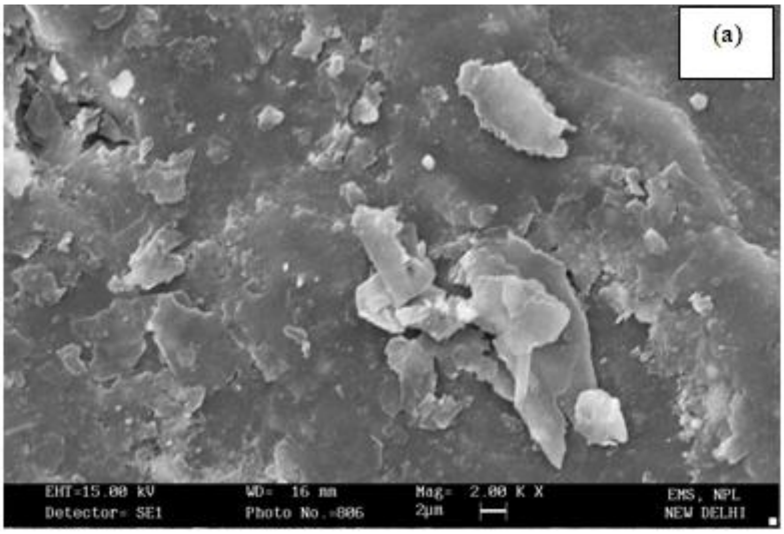
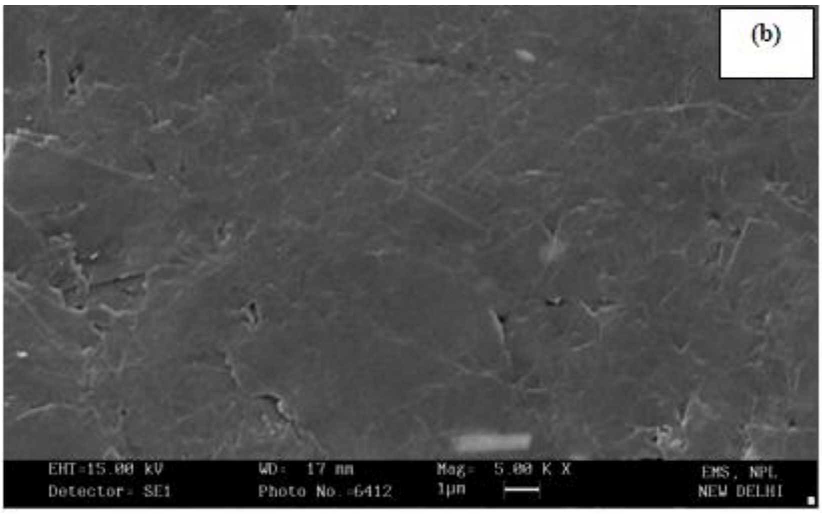


Figure 9(b) shows micrograph of –50+100 BS mesh based composites perpendicular to molding direction. Micrograph does not show any visible gap between the continuous phases of composites.



I-V performance of expanded graphite nanocomposites

Figure 10 compares the I-V characteristic of unit fuel cell prepared with (a) pair of commercial schunk composite bipolar plates of density 1.90 g/cm^3 and (b) EG based composite bipolar plates of density 1.50 gm/cm^3. I-V performance is evaluated in identical condition of both the types of plate in unit fuel cell. From the I-V performance, it is found that EG based composite bipolar plate performance is similar with the commercial schunk composite plates. In the both cases maximum power density is more than 600 mW/cm^2 corresponding to current density 1650 mA/cm^2 for EG based plates and 1400 mA/cm^2 for schunk composite plate.

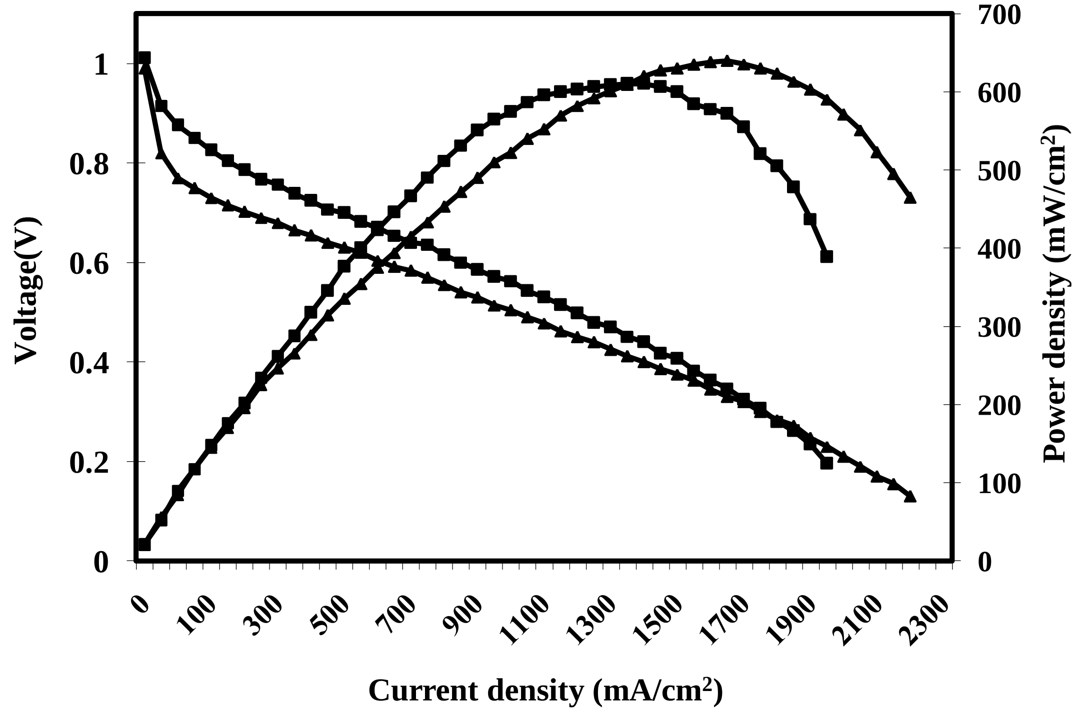


Fig 10I-V performance of expanded graphite nanocomposites

4. CONCLUSION

FTIR Spectra of Novalac phenolic resin suggest the hydroxyl and methylene group that is responsible for linkage between resin and expanded graphite(EG) that make the nanocomposite. There are some variation in theoretical and experimental density might be due to experimental error. Bending strength decrease with decrease density. The nanocomposite of resin and EG have more hardness but this hardness decrease with decrease in density. The conductivity decrease with density due to insulating resin. Modulus decrease with density due to large surface area. This study bring out the role of bulk density of nanocomposite on mechanical and electrical property o

Table 1 summaries the properties of EG composite bipolar plate with Department of Energy(DOE) advanced series of target of bipolar plates [14, 18]. It is found that, properties are satisfied with 50 wt % of EG in composite with bulk density of ~1.5 g/cm^3.

Table 1: Comparative properties of Expanded graphite bipolar plate with DOE target

Properties	DOE -2010 target	Composites with 55 wt % EG
Bulk density (g.cm^{-3})	1.90	1.56
Flexural strength (MPa)	25	54
Modulus (GPa)	-	5.6
Electrical Conductivity (S/cm)	100	150
Air Permeability (MPa)	0.5 MPa	No leak 0.78 MPa
Shore hardness	50	50

This infers that lower value of density with competitive properties of bipolar plate is really helpful in reducing weight and volume of fuel cell stack

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