iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH
IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 10 **Issue:** II **Month of publication:** February 2022

DOI: https://doi.org/10.22214/ijraset.2022.40242

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 10 Issue II Feb 2022- Available at www.ijraset.com

Effect of Annealing Temperature on Polypyrrole Super Capacitive Behaviour

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Abstract: ***Polypyrrole was synthesized by using potentiostatic electrodeposition method. Polypyrrole deposits on stainless steel for 25 min and it annealed at 50^0c, 70^0c, 100^0c and 200^0c, the effects of annealing temparature on polypyrrole morphology were studied by using scanning electron morphology technique. Similarly the super capacitive behaviour of polypyrrole was studied using cyclic voltammetry and charging-discharging technique. Maximum specific capacitance 62.87 F/gm is obtained at 70^0C.***
Keywords: potentiostatic, cyclic voltammetry, annealing temparature, polypyrrole, electrodeposition, stainless steel.

I. INTRODUCTION

The conducting polymers, have created a new area of research. Conducting polymers are important and interesting classes of new organic materials that have gained considerable attention in the recent years. These polymers have unique properties which have led to some commercial applications such as manufacturing of printed circuit boards, corrosion protection, electro chromic displays, electrolytic capacitors, rechargeable batteries and conductive coating for textile sensors, light emitting diodes electromagnetic shielding ,etc.[5,8-11].A polymer is a long – chain molecule that compose a large number of repeating units of identical structure. New conducting polymer structures have been developed over the past two decades with the hope of obtaining better properties than polyacetylene .In many researches it is found that polypyrrole is very sensitive to moisture, because this leads to leaching of the counter ion and thus to decrease in conductivity. Annealing makes the chain mobile to acquire a stable morphology and also to enhance the properties by elimination of moisture content. Polypyrrole is one of the most important conducting, easy to synthesize polymer. Due to enormous properties such as chemical, electrical, optical, surface morphological, etc.it used in many application like solar cell, supercapacitor, and biosensor etc. [3] .

Many of researcher studied effect of annealing temparature on polypyrrole. Rashmi Saxsena [5]has reported that polypyrrole phase was changed in range from 110^0C-170^0C, Sutar Rani[4] reported that around 100^0c water gets evaporated from material and its FTIR Spectrum recorded at different temparature are in good agreement with the TG/DTA data, Riza Ansari[6] reported that thermal treatment of PPY membrane did not lead to any improvement.

In this paper, we have synthesized polypyrrole by elctropolymerization technique using 5 sulfosalicylic acid. Then it was annealed at 50^0 c, 70^0, 100^0, 200^0 c and studied supercapacitive behaviour using cyclic voltammetry and charging-discharging techniques. Surface morphology of polypyrrole was studied with the help of SEM, XRD and Raman spectra.

II. EXPERIMENTAL METHOD

For electrodeposition of Polypyrrole we have used 99% pure pyrrole and 5 sulfosalicylic acid. In 0.1M Pyrrole added 0.01 M of 5 sulfosalicylic acid to adjust PH 2.2.We have applied 0.7 volt to the cell for 25 min, after elctrodeposition, annealed Polypyrrole samples at 50^0c,70^0c,100^0cand 200^0c for 3 hours. This electrodeposition is carried out by three electrode system, stainless steel is used as working electrode, graphite is used as counter elctrode and saturated calomel is used as reference electrode. Before deposition, we cleaned stainless steel substrate properly, then we polished Stainless steel with zero grade paper and then rinsed with acetone and ethanol ;etched in HNo3 for few second and finally cleaned them ultrasonically with distilled water. Electrodeposited polypyrrole are chemically characterized by cyclic voltammetry, charging-discharging and impedance spectroscopic method by using metrohm autolab PGSTAT 204. Confirmation of polypyrrole were confirmed with the help of XRD, Raman spectroscopy and electrochemical impedance technique.

III. RESULT AND ANALYSIS

A. XRD

XRD shows the structure of polypyrrole, figure1 a to d shows amorphous structure of polypyrrole. Peaks are attributed for steel, it is indexed by square. Hump at 20 to 26 degree shows pure amorphous nature of polypyrrole [5]. Water starts its evaporation at around 100 ^{0}c and at 200^0C it gets completely evaporated from the thin film.

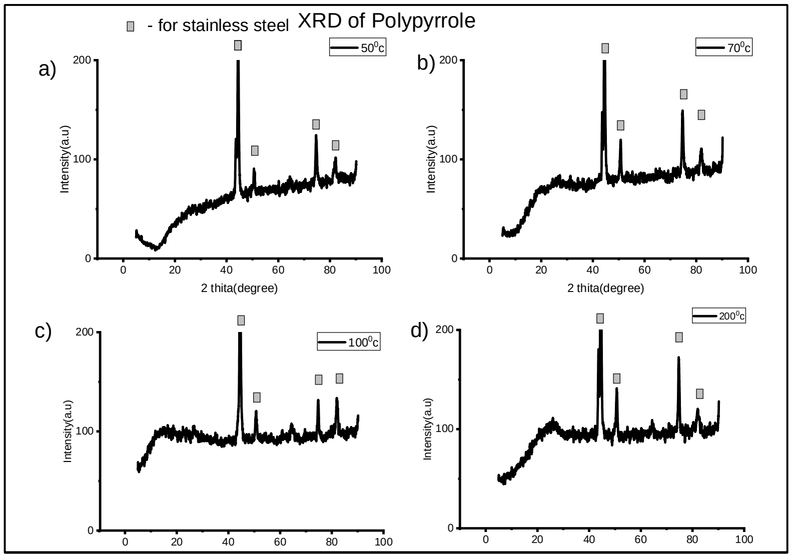

Figure 1: XRD of Polypyrrole a) 50^0c b) 70^0c c) 100^0c d) 200^0c

B. Raman Spectra

This is one of the most used technique for confirmation of material. From the above figure2 peak at 929, 933 cm^{-1} attributes the polypyrrole ring which is absent in 200^0c, peak 970, 977, 984 cm^{-1}attributes C-H Bond, peaks from 1050 cm^{-1} to 1052 cm^{-1} shows C-H plane symmetry band, the band from 1235 cm^{-1} to 1249 cm^{-1} shows C-H asymmetry plane, which is absent at 200^0c. Peaks at 1377 cm^{-1} and 1378 cm^{-1} shows C-N asymmetry plane which are greater than wave numbers obtained at 200^0c .Peaks from 1582 cm^{-1} to 1592 cm^{-1} are obtained because of the C=C backbone stretch of PPY. Around $100^{0\backslash}$c water starts its evaporation hence at 200^0c water gets completely evaporated, so polypyrrole ring vanishes slowly at increasing temparature.

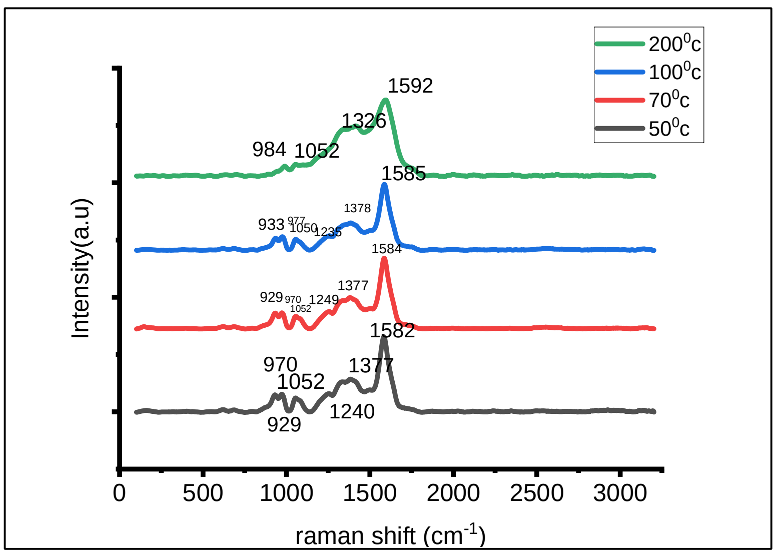

Figure 2: Raman Spectra for Polypyrrole for annealing temparature 50^0c, 70^0c, 100^0c, 200^0c

C. SEM Image

Surface morphology was studied with the help of scanning electron morphology technique. In figure3 a at 50^0c image looks like cauliflower structure and more dense structure is observed somewhat like cabbage , its size is about 2 micro meter, at 70^0c all particles are uniformly distributed and shows cloud like cluster, its average size is 480 nm, at 100^0c particles are agglomerated [3]in spherical shape and are seen distributed non uniformly, surrounded by cauliflower like structure at periphery, at 200^0 c particle structure looks like agglomeratedshape[3] and cauliflower structure but, due to reduction of water content some granular like particles are formed on it.

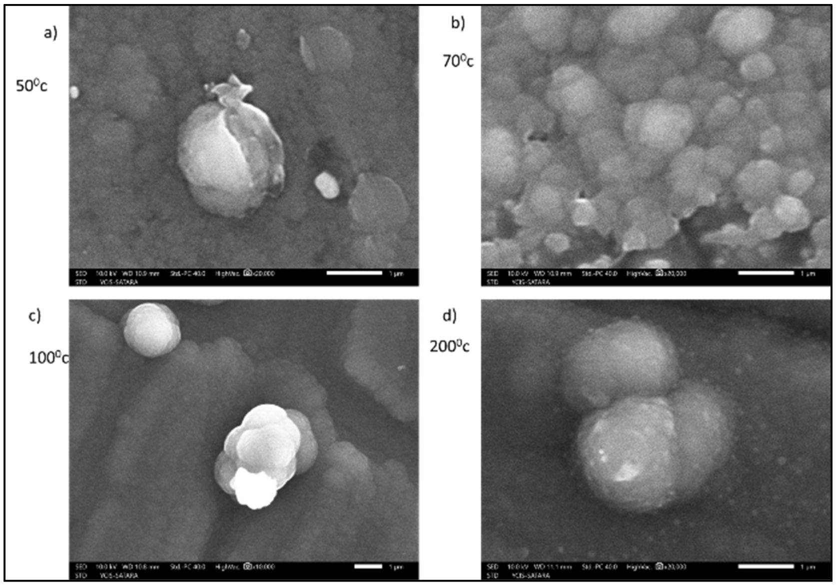

Figure 3:- SEM Image for a) 50^0c b) 70^0c c) 100^0c d) 200^0c

D. Electrochemical Characterization

1) *Cyclic Voltammetry:* Cyclic Voltammogram is one of the technique which is used to study supercapacitive behaviour of material. Using this technique we have studied supercapacitive behaviour of Polypyrrole in 0.5 M of Na2So4 at voltage range from -0.5 V to 0.4 V at 2mV/sec.

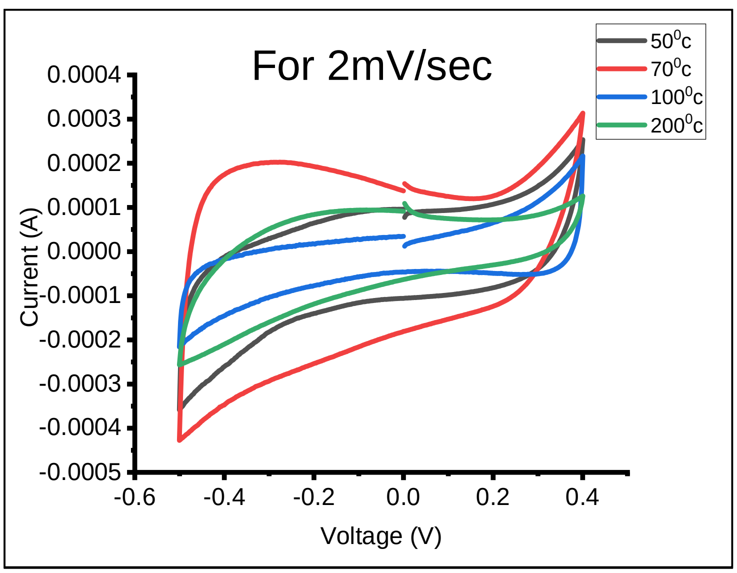

Figure 4(a): Cyclic Voltammogram curve of polypyrrole for 50^0c, 70^0c, 100^0c,200^0c in $0.5MNa_2So_4$

figure4(a) shows temparature at 50^0 and 70^0 , area under the curve increase, while at increasing temparature greater than 70^0 area under the curve reduces, so maximum capacitance is 62.87 F/gm at 70 degree for scan rate of 2mV/sec. At increasing temparature from 100^0 C to 200^0C polypyrrole shows poor capacity performance.

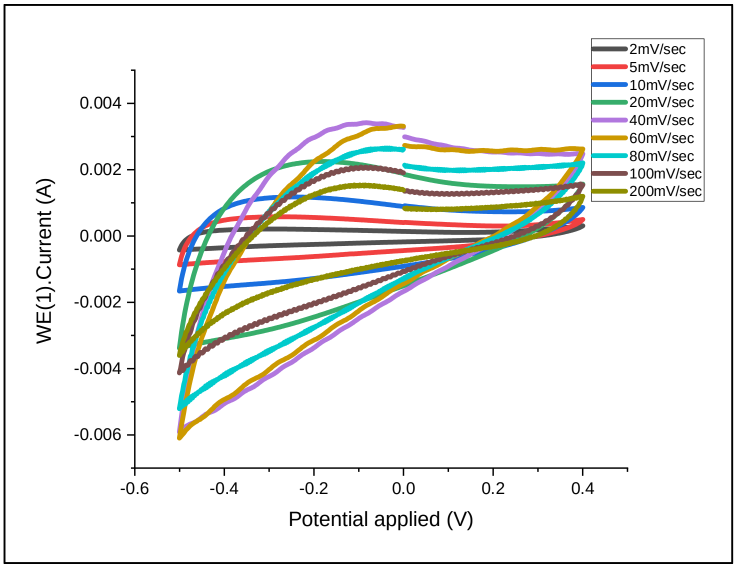

Figure 4(b): scan rate wise cyclic voltammetry curve of polypyrrole at 70^0c in 0.5Na2So4

The figure 4(b) for 70^0c shows that at increasing scan rate area under the curve increases, for low scan rate electrons enter inside pore of film, so specific capacitance is larger at low scan rate. For fast scan rate with increase in scan rate, specific capacitance (Cs) decreases of all polypyrrole films. Ideally capacitive behaviour of the film is observed from CV curves which depict, increase in current with increases scan rate.

2) *Charging- Discharging Curve:* Figure 5 shows the charging-discharging curve for polypyrrole at 50^0, 70^0, 100^0, 200^0C in 0.5 M of Na2So4 at 0.5 mA current. Voltage window for charging- discharging curve is 0 to1 V, it represents pseudocapacitve behaviour of polypyrrole. Maximum specific capacitance from charging-discharging curve is 21.66 F/g. The charging-discharging curve shows asymmetry Nature and maximum energy density and power density are 10.57wh/kg and 292.88W/kg respectively.

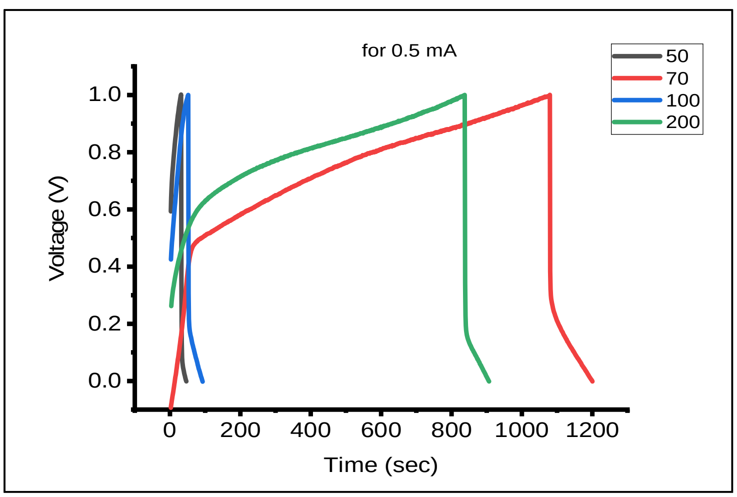

Figure 5 charging discharging curve of polypyrrole for 500c, 70^0c, 100^0c, 200^0c

IV. CONCLUSION

Effect of annealing temparature on polypyrrole and its characterization is successfully studied, its supercapacitive was studied in 0.5M of Na2SO4 from cyclic Voltammetry. Film annealed at 70^0c shows maximum capacitance (62.87 F/gm). At 100^0c evaporation of water starts and at 200^0c water completely gets evaporated which is observed in raman spectra and some granual particle is form around film and is observed with the help of SEM. Due to low porosity at other annealing temparature film capacitance reduces.

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