

Synthesis of Nickel Oxide Nano Material by Electrodeposition for Electrochemical Capacitive Analysis

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ABSTRACT

Electrodeposition techniques is used for the deposition of nickel oxide thin film electrodes. In the present work, we report electrodeposition of nickel oxide thin film on the conducting stainless steel (SS) substrates for the application of electrochemical supercapacitor. X-ray diffraction confirms simple cubic crystal structure with polycrystalline nature of the deposited NiO sample that exhibits hydrophilic nature confirmed from the wettability study. The Scanning Electron Microscope (SEM) observed dense with cracky morphology. UV spectrum exhibits 3.55eV band gap of samples. The capacitive characteristics of the deposited thin film are investigated in 1M KOH electrolyte using cyclic voltammetry (CV). The supercapacitive properties of NiO are strongly affected by the scan rate. The maximum specific capacitance obtained is 162 F/g at 2 mV/s scan rate.

Keywords: Electrodeposition; Nickel oxide; Electrochemical Supercapacitor; Hydrophilic; UV spectrum.

1 Introduction

Nowadays, the concern of achieving high energy and getting energy storage is importance parameter on the researchers. Lot of the researcher work on the energy storage, but the efficiency of the energy storage less than the required energy. Electrochemical supercapacitor (ESc) is the one of the energy storage devices exhibiting good energy as well as power along with cycling stability. ESc is the bridges gap between conventional capacitor and battery [1,2]. The electrodes of electrochemical supercapacitors get prepared by using carbonise materials (Activated carbon, graphene oxide, Multiwalled carbon nanotubes), conducting polymers (Polypyrrole, Polyaniline) and metal oxides and their composites [3-5]. The carbon-based materials exhibiting electrical double layer capacitor (EDLCs) and metal oxides, conducting polymer composites are generally exhibiting pseudo nature [6,7]. So many metal oxides have been tried by the researchers like SnO₂[8], MnO [9], CuO [10-11], Cu(OH)₂[12], Co₃O₄[13] etc. Nickel oxide is a stable semiconductor having a wide bandgap of 3-4eV and P type conductivity [14]. Nickel oxides shows high theoretical capacitance value ~2573F, it is easily available and inexpensive materials for the preparations of the electrodes for electrochemical supercapacitors. It gets used in so many applications like LCD, LED, Solar cell and supercapacitor applications [15].

Nickel oxide/hydroxides prepared by so many methods and techniques such as chemical bath deposition (CBD) [17], SILAR[18], hydrothermal[19]. Electrodepositions is the versatile technique that is low cost, applicable in large area coating and suitable for thin-film coating, etc. This technique provides the best adherence to the metal oxide films [20]. Hence in the present work, nickel oxide thin films were



deposited on the conducting stainless steel using electrodeposition techniques and the structural, morphological and electrochemical capacitive performance for the supercapacitor application is checked.

2 Experimental details

Aqueous solution of 0.5M nickel nitrate hexahydrate (Aldrich AR grade) was prepared in double-distilled water. Stainless steel substrates were used as a current collector, before use they were ultrasonically cleaned in diluted hydrochloric acid (HCl) and raised well in deionized double distill water (DDW). In the preparation of samples, 50 ml 0.5M solution of nickel nitrate hexahydrate was used to electrodepositions, then KOH used for main the PH ~7.5 and applied the constant voltage 1V with time duration 20min for the electrode. The obtained samples were found good in adherence and uniformly.

2.1 Characterizations of prepared electrodes:

Weight of the deposited sample (mg/cm²) was measured by the gravimetric weight difference method using Tapson 100 Ts 8-digit microbalance. The internal geometry of the NiO i.e. crystal parameters of the sample were studied by using X-ray diffraction pattern carried out by X-ray diffractometer (XRD)(Rigaku D/max2550Vb 18 KW with CuK_α,k = 1.54056 Å) in the range of diffraction angle 2θ from 20° to 80°. Morphological studies carried out using a SEM JEOL JSM-6360 scanning electron microscope. The analysis of optical absorption carried out by using UV-VIS spectrophotometer in the wavelength range 400–1200 nm. The cyclic voltammetry study of the sample was carried out using an electrochemical analyzer (CHI 600AD, CH Instruments, USA) with standard three-electrode cell having platinum wire as a counter electrode and saturated Ag/AgCl as a reference electrode. The study of the wettability of the samples was observed using Holmarc 8.0 contact angle meter.

3 Result and Discussion

3.1 X-ray analysis

Fig.1.Shows the XRD pattern of prepared NiO electrode exhibit orientations along different planes (111), (200), (220), (311), and (222) indicating polycrystalline nature of the nickel oxide. Same polycrystalline nature was observed by Sajid Hussian et.al [21]. The observed ‘d’ values were properly matching with standard ‘d’ values taken from JCPDS data card no. 47-1049 of nickel oxide shown in table 1. X-ray diffraction study confirms a simple cubic crystal structure of the as-deposited samples. By using (200) plane of the diffraction pattern, the crystallite size of the NiO sample was instigated by Debye Scherrer’s relation [22]. The observed grain size is 163 nm incomparable with the grain size reported by *et al* Inamdar [23].

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

Where, ‘D’ is the crystallite size, ‘λ’ is the wavelength of the X –ray, ‘β’ is the half width of full maxima, ‘θ’ is the diffraction angle.

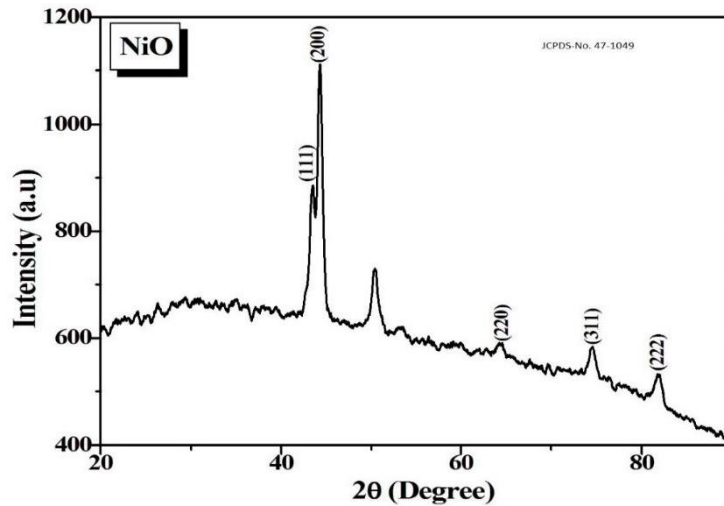


Fig.1: XRD pattern of the nickel oxide.

3.2 Morphological SEM Analysis

The surface morphology was investigated by SEM JEOL JSM-6360 scanning electron microscope. Fig.2 represents the SEM images of A to D at different magnifications. The SEM images indicate the formation of a compact, uniform, dense muddy like with crackly porous surface morphology of NiO. The crackly morphology of the NiO similarly observed by anamika et.al[24]. This exclusive building of image provides a high-specific capacitance due to easy access of electrolyte ions interactions with NiO during faradic redox processes.

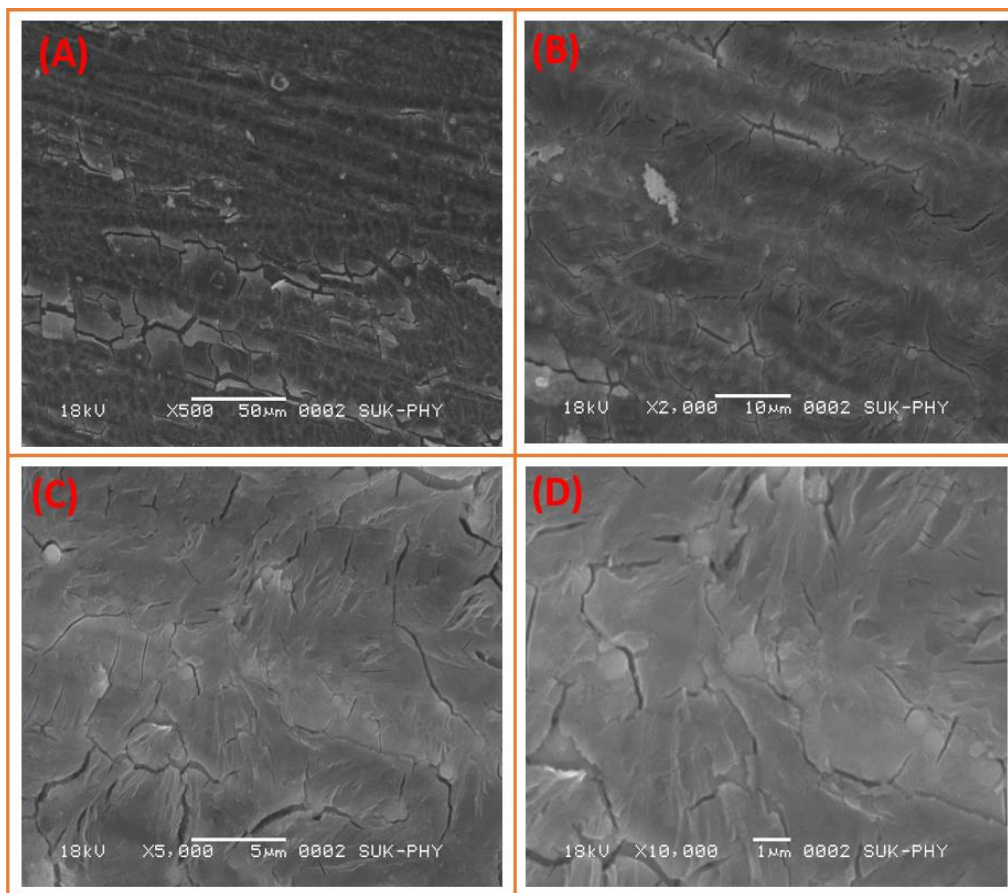
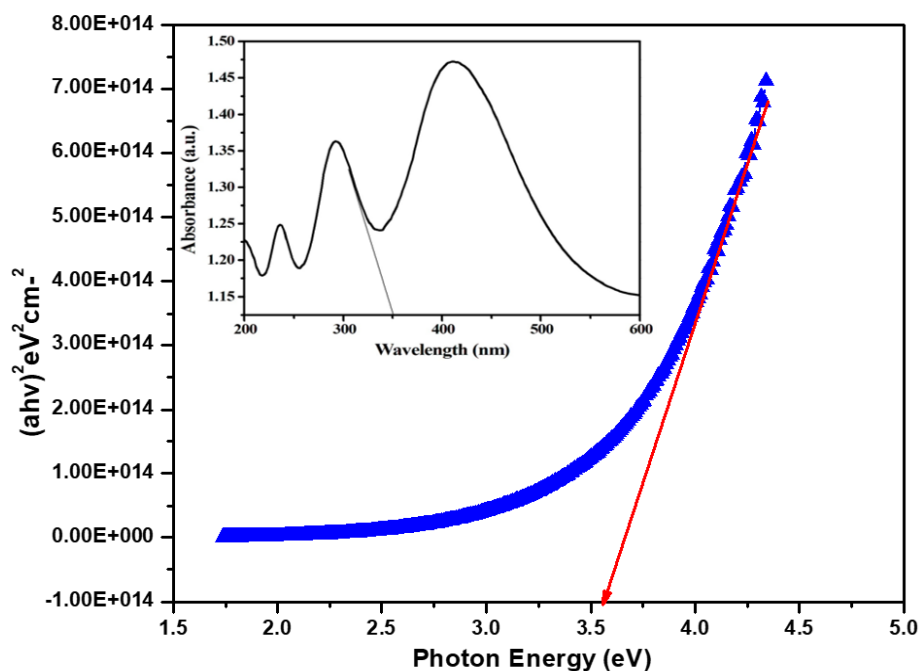


Fig 2: SEM Micrograph of NiO thin film with different magnifications

3.3 UV-Visible Spectroscopy

The UV-VIS spectroscopy carried out of the NiO thin film electrodes. In this Fig.3 the absorbance peak observed at $\sim 410\text{nm}$. Further Using the data of spectroscopy estimates the band gap energy of the NiO, observed direct band gap from this graph is $E_g = 3.55\text{ eV}$. The same band gap energy observed by Boschloo and Hagfeldt et.al [25] It is evaluated that the transmittance is High in UV and poor portion in Visible portion.



3.4 Wettability Study

To study the wettability property of the NiO electrode which is necessary for the intercalation of the electrolyte's ion to electrode, contact angle measurement was carried out. Fig.4 sample exhibits hydrophilic nature showing contact angle 65° which is less than 90° . Same hydrophilic nature was observed by Devasthali et.al [26]. The contact angles between the electrolyte and electrode is less than the 90° is clearly indicates that the prepared electrode is porous nature materials due to electrolytes diffuse in nano materials.

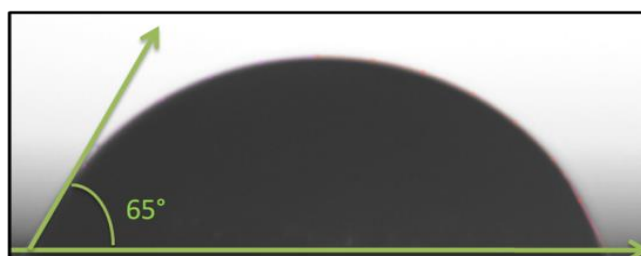


Fig.4. Wettability of the NiO electrode for contact angle.

3.5 Cyclic voltammetry study

Fig. 5(A) Shows the CV curves of prepared NiO electrode in the potential range of -1.8 to 0.8 V/S in aqueous 1M KOH electrolyte at different scan rates. The observed cracky morphology of the NiO more suitable for the diffusivity of the electrolytes ions in the cracks and inhaances of the capacitive

behaviour. A pair of cathodic and anodic peak in CV curves indicates the pseudocapacitive characteristics, which mainly governed by Faradic redox reactions.

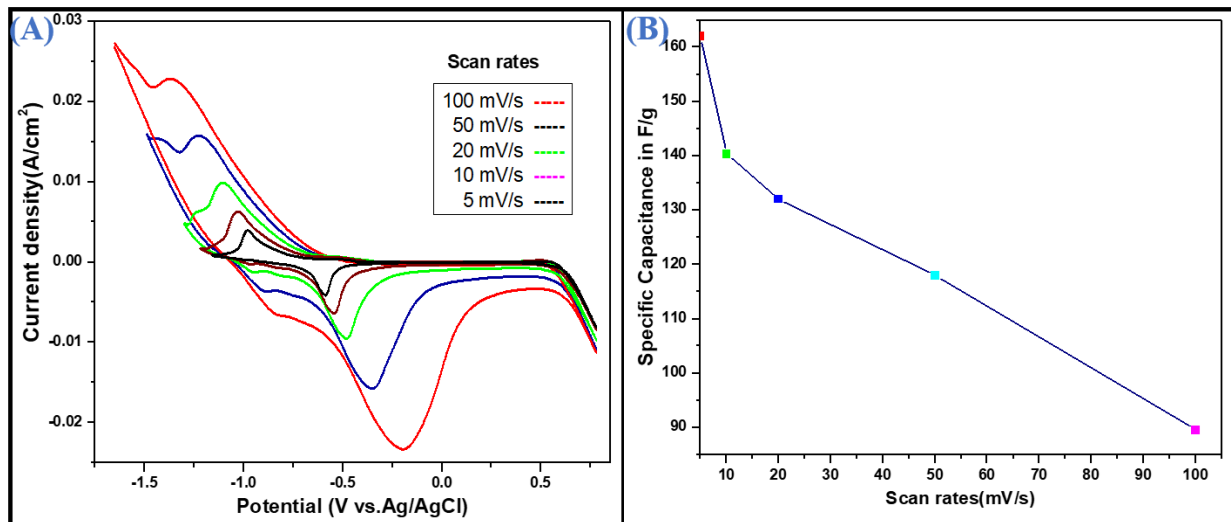


Fig.5: Cyclic voltammetry curves.

Specific capacitance (SC) of the nickel oxide electrode was calculated by using CV using following relation.

$$\text{Specific capacitance (Sc)} = \frac{\int i * dt}{2 * M * \vartheta * V} \text{ F/g}$$

Where, $i * dt$ is be the current, M is be the active electrode loaded mass, ϑ is be the scan rate and V is be the potential window of the electrode. The variation of SC values with the scan rate is given in Table 2. Here it is observed that, with increase in scan rate SC value decreases, indicating the reversible nature of the electrode. Fig.5 (B). show the deposited nickel oxide electrode material cannot exhibit the complete redox transition at a higher scan rate due to the improper diffusion of the NiO electrode.

3.6 Stability study

Electrochemical stability is one of the most important parameters for liquid supercapacitors. Here stability curve of the NiO electrode was carried by repeating the CV cycles at a potential scan rate of 100 mV/s in 1M KOH. Fig.6 shows the variation in SC with several number of cycles. Here it is observed that the initial value of SC 89.56 F/g, decreases up to 600 cycles then it remains steady indicating a 42% decrease in capacitance value. This decrease in capacitance value is due to the slight degradation of the electrode material at a high scan rate. Prepared NiO electrode exhibit better electrochemically stability above 600 cycles.

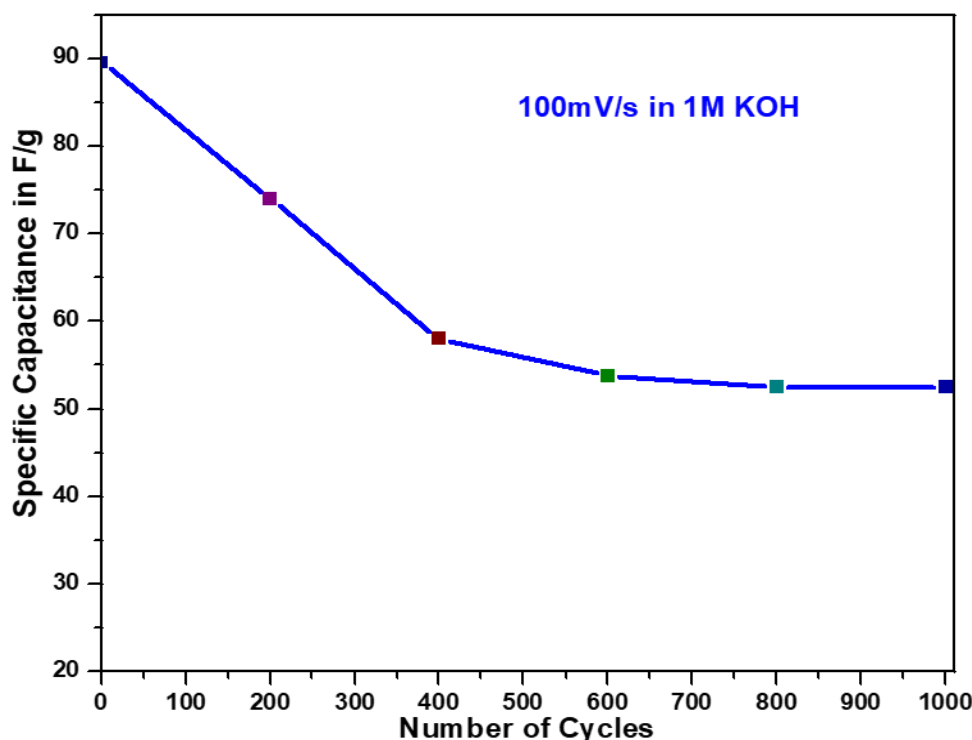


Fig.6: Stability curve of NiO electrode.

4 Conclusions

The prepared nickel oxide nano material electrode by electrodeposition method from the nickel nitrate and it shows excellent characteristics of the electrochemical capacitive behaviours and more suitable for charge storages. The NiO sample shows polycrystalline nature of nano grains. The nanomaterials of the nickel oxide show highest band gap energy $\sim 3.55\text{eV}$ with 410nm wavelength exhibits highest absorbance. Nickel oxide shows hydrophilic nature favourable for capacitive property. Cyclic voltammetry study exhibits the pseudo nature of the electrode. The specific capacitance exhibited by the electrode in 1M KOH at 5mV/Sec is 162F/g and cycling stability of the electrodes is 42% capacitance decreases up to 1000 cycles.

Conflict of Interests

Authors declare that they have no conflicts of interests.

References

- [1] Fang He, Kaiyu Liu, Jianjian Zhong, Shuirong Zhang, Qi Huang, Cheng Chen, One dimensional nickel oxide-decorated cobalt oxide (Co₃O₄) composites for high-performance supercapacitors, *J Electroanal. Chem*, 749 (2015) 89-95.
- [2] Limin Chang, Fang Ren, Cuimei Zhao, Xiangxin Xue, Synthesis of Co(OH)₂/Ni(OH)₂ nanomaterials with excellent pseudocapacitive behavior and high cycling stability for supercapacitors *J. Electroanal. Chem*, 778 (2016) 110.
- [3] Qinglan Zhao, Xianyou Wang, Chun Wu, Jing Liu, Hao Wang, Jiao Gao, Youwei Zhang, Hongbo Shu, Supercapacitive performance of hierarchical porous carbon microspheres prepared by simple one-pot method, *Journal of Power Sources*, 254, (2014), 10-17.
- [4] W.X. Chen, H. Zhang, Y.Q. Huang, W.K. Wang, *J. Mater. Chem.* 20 (2010) 4773- 4775.
- [5] D.R. Dreyer, S. Park, C.W. Bielawski, R.S. Ruoff, *Chem. Soc. Rev.* 39 (2010) 228-240.
- [6] M.M. Sk, C.Y. Yue, *J. Mater. Chem. A* 2 (2014) 2830-2838.
- [7] E. Frackowiak, K. Metenier, V. Bertagna, F. Beguin, *Appl. Phys. Lett.* 77 (2000) 2421e2423.
- [8] Qinqin Zhao, Lisha Ma, Qiang Zhang, Chenggang Wang, Xijin Xu, SnO₂-Based Nanomaterials: Synthesis and Application in Lithium-Ion Batteries and Supercapacitors, *Journal of Nanomaterials*, 850147, (2015), <https://doi.org/10.1155/2015/850147>.
- [9] Yogesh Kumar, Seema Chopra, A.Gupta, Y.Kumar, S.J.Uke, S.P.Mardikar, Low temperature synthesis of MnO₂ nanostructures for supercapacitor application, *Materials Science for Energy Technologies*, 3, (2020), 566-574.

- [10] TS Ghadge, AL Jadhav, BJ Lokhande, Synthesis and electrochemical study of ruthenium influenced copper oxide electrodes prepared by self anodization, *Journal of Alloys and Compounds* 824, (2020), 153860
- [11] SV Kambale, AL Jadhav, RM Kore, AV Thakur, BJ Lokhande, Cyclic Voltammetric Study of CuO Thin Film Electrodes Prepared by Automatic Spray Pyrolysis, *Macromolecular Symposia* 387 (1), (2020), 1800213.
- [12] TS Ghadge, AL Jadhav, YM Uplane, AV Thakur, SV Kamble, B.J.Lokhande, Controlled synthesis, structural, morphological and electrochemical study of Cu (OH) 2@ Cu flexible thin film electrodes prepared via aqueous–non-aqueous routes *Journal of Materials Science:Materials in Electronics*, 1-14,(2021).
- [13] S. K. Meher , G. R. Rao ,Ultralayered Co3O4 for High-Performance Supercapacitor Applications, . *Phys. Chem. C*, 115, 31, (2011),15646–15654.
- [14] B.R. Cruz-Ortiz, M.A. Garcia-Lobato, E.R. Larios-Durán, E.M. Múzquiz-Ramos, J.C.Ballesteros-Pacheco, Potentiostatic electrodeposition of nanostructured NiO thin films for their application as electrocatalyst, *J. Electroanal. Chem.* 772 (2016) 38-45.
- [15] A. A. Yadav, Preparation and electrochemical properties of spray deposited α -Fe₂O₃ from nonaqueous medium for supercapacitor applications, *J. Mater. Sci.: Mater. Electron*, 27 (2016) 1866-1872.
- [16] AI Inamdar, AC Sonavane, SM Pawar, YoungSam Kim, JH Kim, PS Patil, Woong Jung, Hyunsik Im, Dae-Young Kim, Hyungsang Kim, Electrochromic and Electrochemical properties of amorphous porous nickel hydroxide thin films, *Applied surface science*, 257,22, (2011),9606-9611.
- [17] G.S. Gund, C.D. Lokhande, H.S. Park, Controlled synthesis of hierarchical nanoflake structure of NiO thin film for supercapacitor application, *Journal of Alloys and Compounds*, (2018), doi: 10.1016/j.jallcom.2018.01.166.
- [18] S. D. Dhas , P. S. Maldar,, M.D. Patil, A.B. Nagare,M.R. Waikar, R.G. Sonkawade, A. V.Moholkar, Synthesis of NiO nanoparticles for supercapacitor application as an efficient electrode material, *Vacuum*,181,(2018),1096-46.
- [19] J.L. Yin, J.Y. Park, A Nickel Foam Supported Copper Core/Nickel Oxide Shell Composite for Supercapacitor Applications, *Microporous and Mesoporous Materials* (2014), doi:http://dx.doi.org/10.1016/j.micromeso.2014.08.016.
- [20] S. Husain, F. Rahman, Nasir Ali and P.A. Alv, Nickel Sub-lattice Effects on the Optical Properties of ZnO Nanocrystals *Journal of Optoelectronics Engineering* 1.1 28 (2013) 28.temperature-dependent morphological and electrochemical properties of copper hydroxide thin film electrodes obtained by anodization of copper 51 (21),(2016) 9879.
- [21] A.I. Inamdar, Young Sam Kima, S.M. Pawar , J.H. Kimb, Hyunsik Ima, Hyungsang Kim,Chemically supercapacitors, *Journal of Power Sources*, 196 (2011) 2393.
- [22] A.C. Sonavane, A.I. Inamdar, P.S. Shinde, H.P. Deshmukh, R.S. Patil, P.S. Patil, Efficient electrochromic nickel oxide thin films by electrodeposition, *Journal of Alloys and Compounds*, 489, (2010),667–673
- [23] Boschloo, Gerrit, Hagfeldt, Spectro electrochemistry of nanostructured NiO, In: *Journal of physical chemistry b*, 1089-5647, 105, 15, (2001),3039-3044.
- [24] A. S. Devasthali, S. G. Kandalka, Preparation and characterization of spray deposited nickel oxide (NiO) thin film electrode for supercapacitor,*IOSR Journal of Computer Engineering (IOSR-JCE)*,2, (2015),47.