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Chemical Precipitation Synthesis of Fe, Ni, and Co Doped Zinc Sulphide Nanoparticles

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Abstract: *Transition metal ions like Nickel, Cobalt and rare earth ions doped ZnS semiconductor materials have a wide range of applications in electroluminescence devices. In the present investigation nanoparticles of Fe, Ni, Co doped ZnShave been synthesized successfully by simple chemical precipitation method. Particle size and specific surface area have been calculated from XRD analyses which confirm the nano structure of the samples. Absorption spectra of Fe, Ni and Co doped ZnS were recorded and their optical transmittance are properly determined. The optical transmittance properties of the ZnS nanomaterial improved the addition of transition metal ion doping. The optical transmittance lies in visible region is one of the advantages of display devices and visible light LED'S. The molecular structure of the title compound was determined by the FT-IR analysis and the different vibrational bands confirmed the functional groups present in the sample. Current voltage graph of Fe doped ZnS were also drawn specific capacitanceof the sample were calculated. It shows enhanced electrical property of the nanomaterial. The Photo Luminescent spectra were also recorded it showed that the material has good photoluminescence property suitable for preparing optoelectronic devices. The antibacterial activity of Fe doped ZnS were also determined.*
Keywords-*Anti bacterial Activity, FT-IR, Nanoparticle, PL, Semiconductor, XRD, Zinc Sulphide*

I. Introduction

In recent times there have been extensive studies on luminescent semiconductor nanocrystals because of their potential application in future optoelectronic devices. The nanosized semiconductor crystallites could change optical properties which are different from bulk materials[1]. Transition metal ions (Ni,CO) and rare earth ions doped ZnS semiconductor materials have a wide range of application in electroluminescence devices, phosphors, light emitting displays and optical sensors. Semiconductor nanoparticles doped with transition metal ions have attracted wide attention due to their luminescence properties[2]. Various efforts have been made by the researchers to dope transition metal ions in nanomaterial, several researchers have reported the luminescence quenching in ZnS nanoparticles due to Fe and Ni doping. Generally, ZnS becomes good host material, since it is a kind of wide band gap II-IV component semiconductor materials (Eg-3.6eV), and with its energy band characteristics. It is commercially used as phosphor and thin film electroluminescence devices[3].Motivated by this, Fe,Ni and Co doped ZnS nanoparticles were synthesized by Co-precipitation method their structural and morphological studies have been analyzed by powder XRD and SEM. Their optical properties were carried by UV-vis and photoluminescence spectra. The antibacterial activity of Fe doped ZnS were studied and their Zone of inhibition were also determined. The electrochemical property of the sample helped to estimate the specific capacitance value of the nanomaterials.

II. Experimental

(i) Synthesis of Fe doped ZnS

Samples of different sized ZnS nanoparticles were prepared by simple chemical precipitation method using analytical grade Zinc Chloride, Ferric Chloride and Sodium Sulfide. 0.1M Fe doped ZnS nanoparticles were prepared by mixing 0.1M of $ZnCl_2$ (20ml) and 0.1M of $FeCl_3$ (20ml). Then, the obtained solution was continuously stirred for 1 hour, after that 90ºc heat was supplied, along with that 0.1M of NaS (10ml) was slowly added to the solution and the resultant solution was continuously stirred for another 1 hour. Finally, Orange color precipitate was obtained [4]. The obtained precipitate was allowed to evaporate at room temperature to obtain Fe doped ZnS nanoparticles in orange color powder form. The same procedure was adopted to get 0.01 M sample. But the nanoparticle shows a brown color. Change in molarity causes some change in optical property. The same procedure is applied for the doped nanomaterials preparation of 0.01M, 0.1M,0.3M,1M Fe doped ZnS. Instead of Fe various dopants like Ni and Co were added to get a green and purple color nanopowder of ZnS respectively.

III. Structural Characterizations

3.1Powder X-Raydiffraction (XRD) Pattern

The XRD Pattern of sample. 0.1M $FeCl_3$ doped ZnS is shown in figure 3.1. As expected, the XRD peaks of nanoparticles are considerably broadened due to finite size of these particles[5]. They were also compared with JCPDS file No.01-0792.

XRD- Particle Size Calculation

From this study, considering the peak at degrees, particle size has been estimated by using Debye Scherrer formula and found to be in the range of 21-24 nm.

$$D = 0.9\lambda/\beta\cos\theta \quad \ldots\ldots\ldots.(1)$$

Were 'λ' is wave length of X-Ray (0.1541nm), 'β' is FWHM (full width at half maximum), 'θ' is the diffraction angle and 'D' is particle diameter size. The value of d (inter-planner spacing between the atoms) is calculated using Bragg's Law[6].

$$2d \sin\theta = n\lambda \ldots\ldots\ldots(2)$$

Due to size effect the peaks broaden and then widths become larger as the particle size becomes smaller. The broadening of the peak may also occur due to micro strains of the crystal structure arising from defects like dislocation and twinning.. The XRD shows that ZnS nanoparticle has Cubic Phase[6].

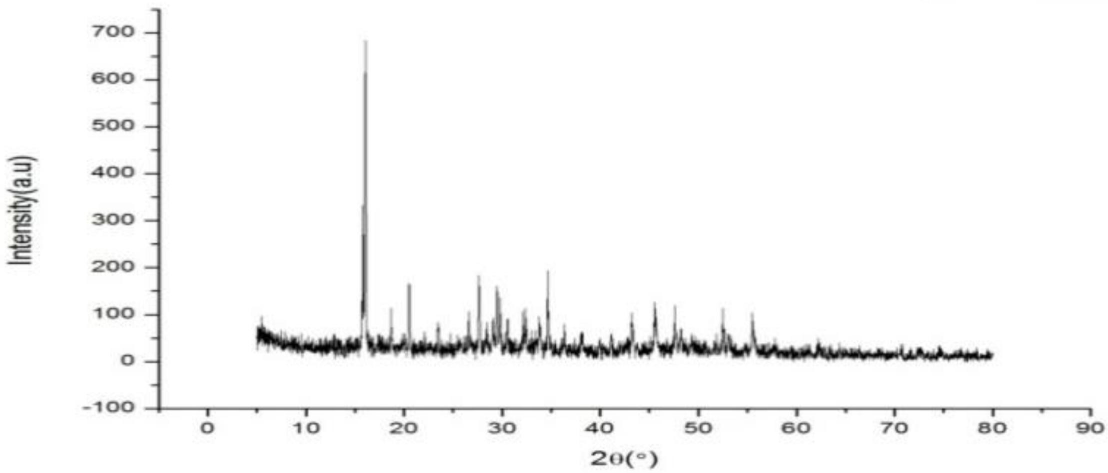


Fig 3.1 Powder XRD Pattern of Fedoped ZnS

3.2 FTIR spectral analysis

FTIR spectra of synthesized compound were recorded the range 400-4000nm employing a JASCO-FTIR 460 plus spectrometer. Following the KBr pellet technique at 300k with the scanning speed 2mm/s as shown in fig (3.2).

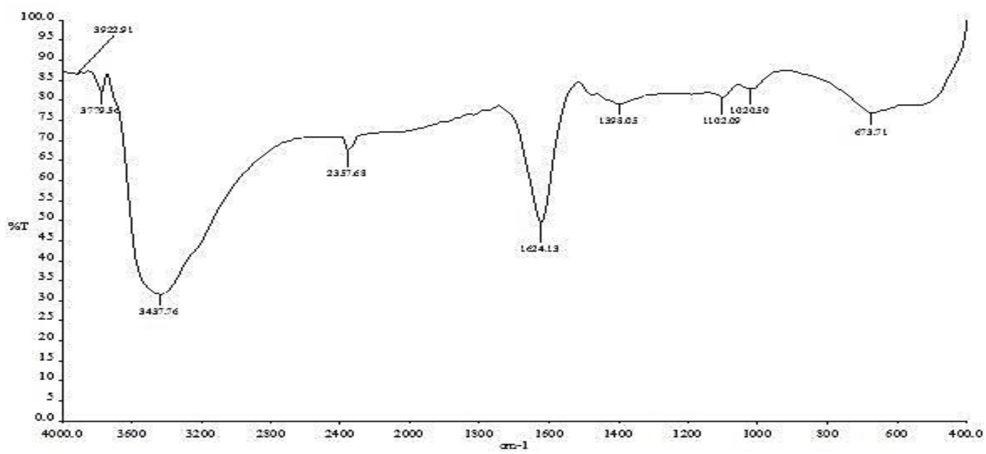


Fig 3.2 FTIR Spectrum of Fe doped ZnS

IV. Optical Properties

4.1 UV- Vis spectroscopy

UV-Vis spectrum of Fe ,Ni and Co doped ZnS Optical transmittance and absorption spectra of the Fe, Niand Co doped ZnSwere recorded using a Lambda 35 UV-Vis NIR spectrophotometer in the UV-Vis region.The UV-Vis absorption spectra of Fe, Ni, Co doped ZnS shows the absorption band at 300nm,220nm and 380 nm, respectively and after that it shows less absorption, I.e. (highly transparent). In the transmittance spectra of Fe doped ZnS shown in fig (4.2(a)), and Ni doped ZnS shown in fig (4.2 (b)) and Co dopedZnS shown in fig (4.2 (c)) closely agree with the absorption spectra. It has a large optical transmittance window in the region 380 -1100nm. There is no remarkable absorption band found in between. Wide transparency region is the important criteria of nanomaterial. Hence, it can be used in optical nano device fabrications. The sample is completely transparent in the visible region, which is mandatory for using in the electrochemical sensors and high sensitivity optical sensors for the detection[8]. The optical energy band gap of the given[9] Fe, Ni and Co doped ZnS of the nano materials is as shown Fig(4.3 a, b, c) and tabulated in the table 4.2.

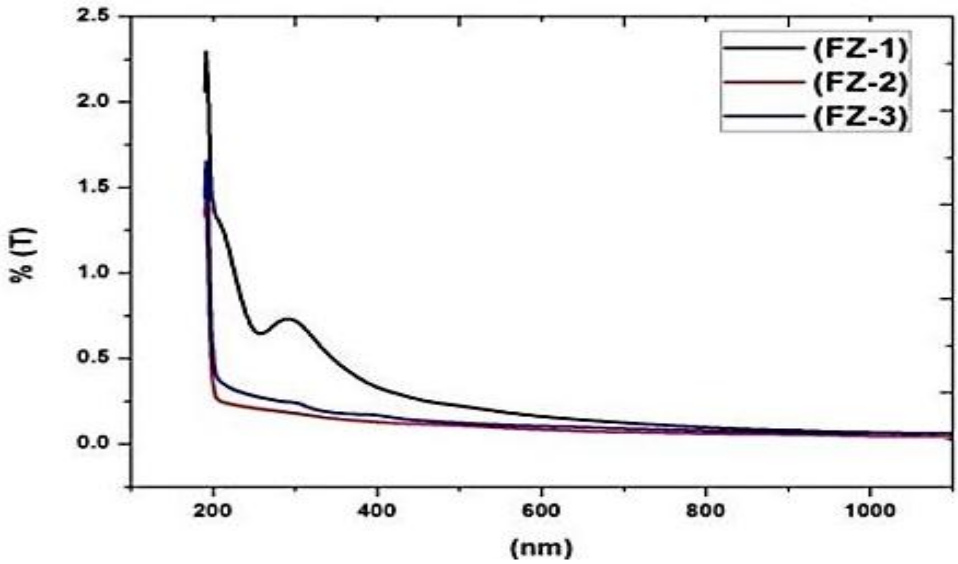
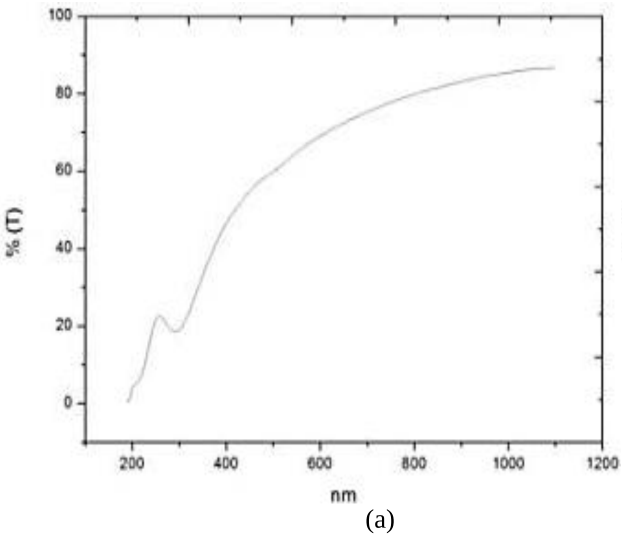


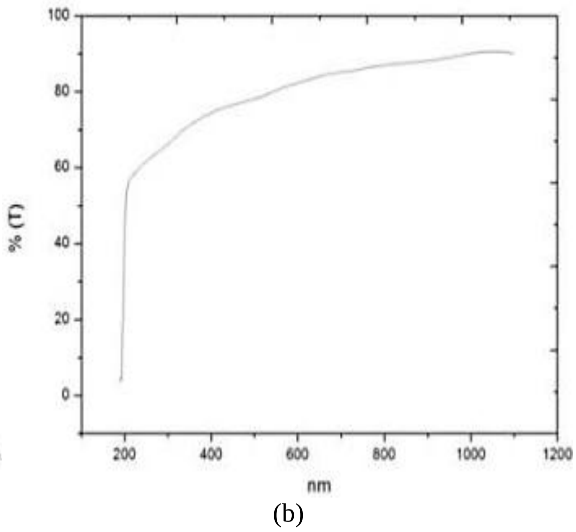
Fig.4.1 Absorption spectra

Table 4.1

Nano particles	Optical transmitance window(nm)	Energy gap(ev)
$FeCl_3$ doped ZnS	300-1200	4.7
$NiCl_4$ doped ZnS	360-1200	6.1
$CoCl_2$ doped ZnS	380-1200	6.2



(a)



(b)

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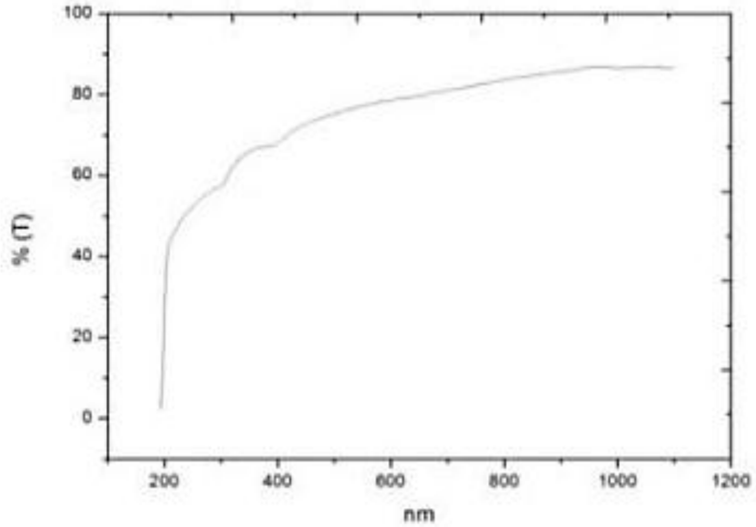
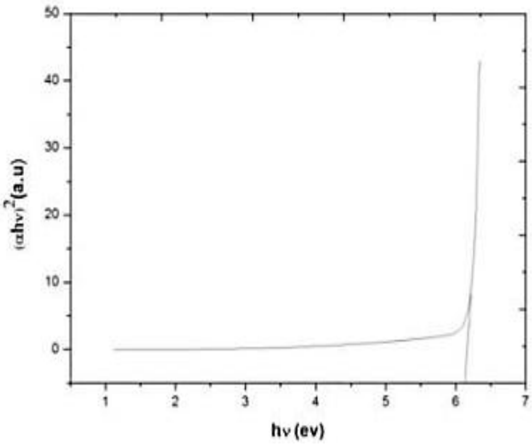
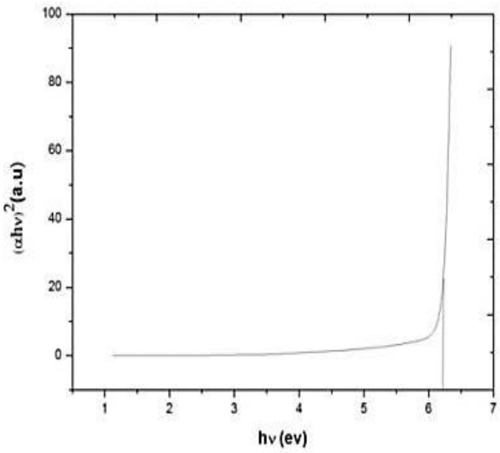


(c)

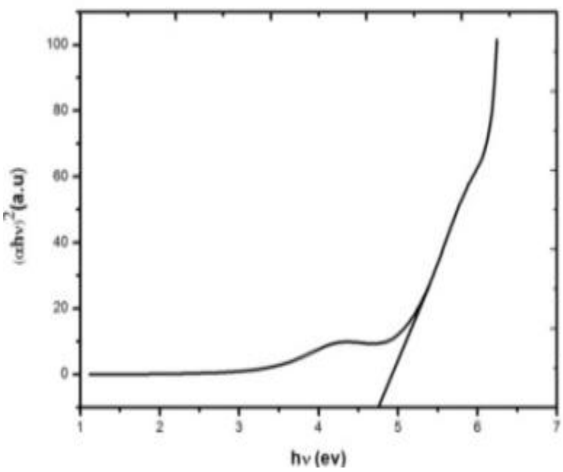
Fig. 4.2 Transmittance Spectra of (a) Fe-ZnS, (b) Ni-ZnS, (c) Co-ZnS respectively



(a)



(b)



(c)

Fig.4.3 Energy band gap determination of (a) Ni-ZnS,(b) Co-ZnS, (c) Fe-ZnS respectively

4.2 Photoluminescence Spectrum

Photoluminescence spectrum of Fe, Ni, Co, doped ZnSare shown in Fig.(4.6) Some emission peaks are observed in photoluminescence spectrum.[10,11]A strong peak of $FeCl_3$ doped ZnS (FZ-1) at 426nm, near the band edge due to free excition emission is observed in photoluminescence spectrum.[16-19] The broad band in the region of 400-800 nm, in photoluminescence spectrum of $FeCl_3$ doped ZnS is related to the amount of non-stochio-chiometric intrinsic defects and the same may be due to pure Fe doped ZnS as reported.[12] From photoluminescence analysis, it is observed that all are have fine luminescence property. Similarly all Fe doped ZnS are exhibiting the luminescence spectrum at 512 nm Niand 532 nm Co doped ZnS (FZ-3)respectively. Generally Ni doped ZnS(FZ-2)shows the ultraviolet emission with the red shift emission with compared to UV absorptions values in the photoluminescence analysis[13].

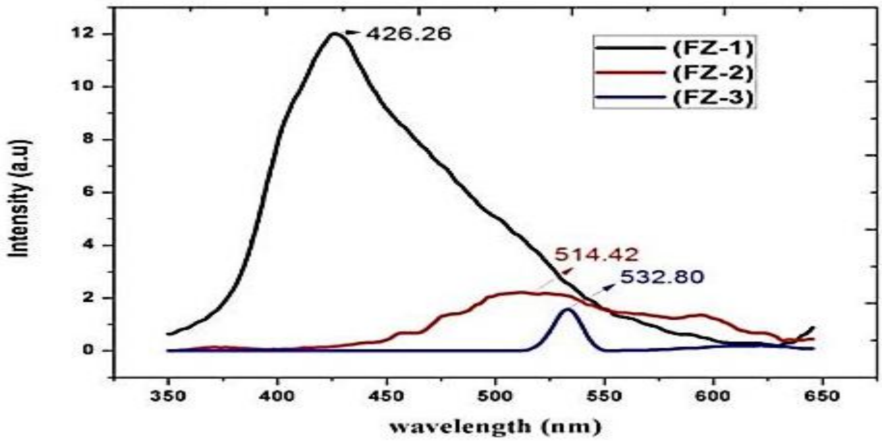


Fig., 4.6 Photoluminescence

V. Anti Bacterial Activity

Procedure

1.3g of Nutrient broth (Himedia) was mixed into 100ml of distilled water is kept for autoclave at 121ºc.[14] After autoclaving this was allowed to cool for bearable temperature then it was poured into sterile petriplates. The petripltes were allowed to solidification. After solidification, this plates were used for culture suaping for testing sensitive or resistant for given sample.[15,16]

Table 5.1

Zone of inhibition in mm				
Name of the microbes	Different concentration			
	0.5 mg	1 mg	2 mg	4 mg
Staphylococcus aureus(Gram +ve)	17±0.02	21±0.00	28±0.20	31±0.40
salmonella typhi(Gram -ve)	18±0.40	22±0.20	26±0.35	32±0.00
Bacillus subtilis	13±0.28	20±0.16	23±0.16	26±0.33
Escherichia coli	18±0.00	21±0.28	24±0.33	28±0.00

Fig, 5.1 Bacillus Subtilies

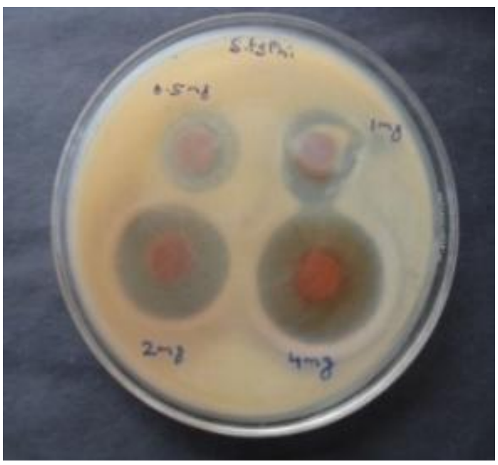
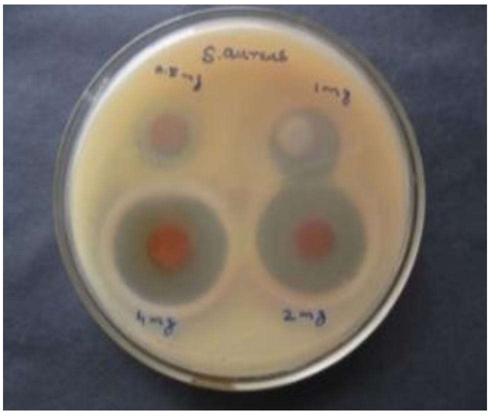
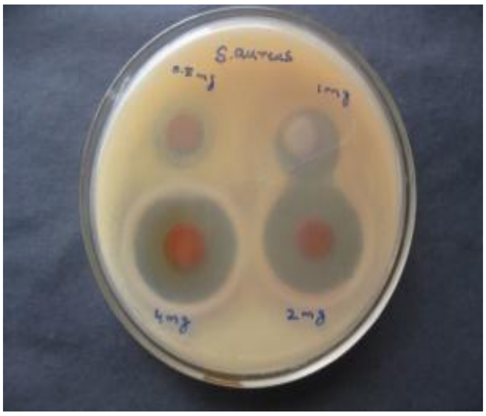


Fig. 5.2 Salmonella typhi

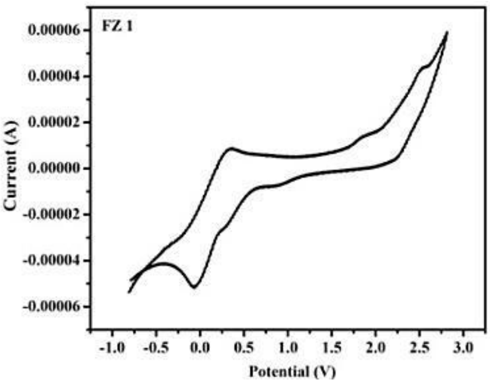




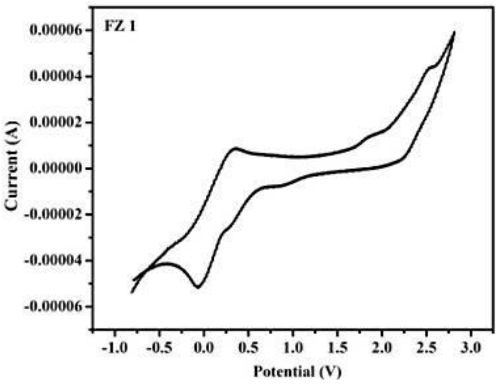
Fig, 5.3 Staphylococcus oureusFig. 5.4 Escherichia coli

5.1 Electrochemecial Property-Cyclic Voltammetry

The electrochemical properties of the Fe dopedZnS, Ni doped ZnS, Co doped ZnS nanomaterial as an electrode material for super capacitors were investigated using cyclic voltmeter (CV) and shown in the fig (5.1). It can be clearly understood that both the curves are rectangular in shape without any radix peak currents exhibiting the ideal capacitive behavior of the prepared electrodes. The integrated area under the CV curves is proportional to the specific capacitance. Fig (5.1) shows the CV curve of the above three electrodes under different scan rates in 1M Na2SO4 and all the curves are rectangular in shape and exhibit mirror image characteristics which indicate that the Faraday radix reactions re electrochemically reversible as well as an ideal electrochemical capacitive behavior. [17] It can be seen that the current under curve increases with as increase in the scan rate which indicates that the voltammetry current is always directly proportional to the scan rete and the CVs of the synthesized nanomaterial's retain a similar shape even at high scan rate, indicating an excellent capacitance behavior.



(a)



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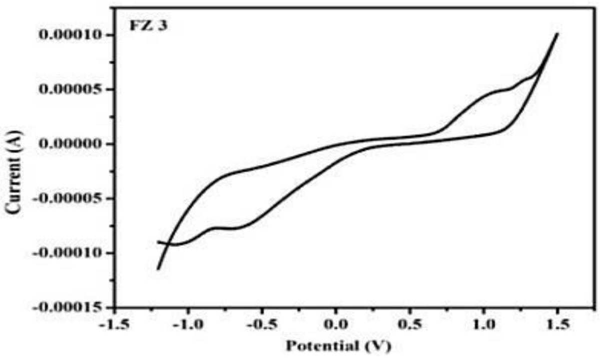

(c)

Fig 5.1 CV of(a).Fe doped ZnS (FZ-1) (b).Ni doped ZnS (FZ-2)(c).Co doped ZnS(FZ-3)respectively

VI. Conclusion

In the present investigation, Ferric(Fe) doped Zinc sulphide (ZnS)nanomaterial was synthesized by chemical precipitation metal at room temperature.Nanoparticles of Fe, Ni, Co doped have been synthesized successfully by simple chemical precipitation method. The powder X-ray diffraction pattern of the samples confirm, the formation of Nanoparticles of $FeCl_3$doped ZnS and their cubic structure. Particle size has been calculated from XRD analyses which confirm thenano structure of the samples. UV-Visible spectra of Fe, Ni and Co doped ZnS were recorded and their optical transmittance are properly were determined Results show that Ni doped ZnS nanomaterial have a good optical transmittance property compare to Fe and Co dopedZnS. Therefore the optical transmittance of the nanomaterial improved the addition of translation metal iron doping. Consequently shifts the material transparency limits towards a blue wave length. Hence this material is also suitable for short wavelength generation. The optical transmittancelies in visible region is one of the advantages of display devices and visible light LED'S.

The molecular structure of the title compound was determined by the FT-IR analysis and the different vibrational bands confirmed the functional groups present in the sample.

Current voltage graph of Fe doped ZnS were also drawn the specific capacity of the sample were also calculated. It shows enhanced electrical property of the nanomaterial. The Photo Luminescent spectra were also recorded for the given materials. This nanomaterial has good photoluminescence property suitable for preparing optoelectronic devices.

Fe doped ZnS nanomaterial has better antibacterial activity. in the Staphylococcus oureus and Salmonellaetyphi microbes, Hence the synthesized Fe doped ZnS nanomaterial is suitable for preparing nanomedicine in order to improve the hemoglobin content of an anemic patient.

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