Synthesis of NiO nanoparticles by chemical precipitation method

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Abstract: Over the last few years, a lot of work has been carried out in the field of NiO nanoparticles owing due to their good mechanical and thermal properties. Theyhave a wide a variety of applications in the electronic industry as well in material science. In this present investigation, NiO nanoparticles were synthesized by chemical precipitation method using different surfactants. The size, shape and crystalline nature of the NiO nanoparticles were studied by scanning electron microscopy (SEM) and X-Ray diffraction studies (XRD). It was found that NiO nanoparticles synthesized by using Polyvinylpyrollidine(**PVP)** as a surfactant was found to be efficient in reduction of particle size distribution and had a spherical shape with weak agglomeration as confirmed from the SEM images.

Keywords: NiOnanoparticles, PVP, SEM, XRD

1. Introduction

Over the last decade, a lot of scientists and researchers alike have been focussed on creating small materials which are not only advanced in their properties as compared to their bulk counterparts but which are also cost effective and readily available**[1].**Nanotechnology thus provides the ability to engineer the properties of materials by controlling their size and this has driven researchers to create wide potential uses from nanomaterials. These so called nanomaterials, especially the synthesized crystalline metal oxides demonstrate large surface areas, surface defects while other metal oxides exhibit good mechanical, electronic, magnetic, thermal, catalytic as well as better optical properties **[2-3]** which makes them suitable materials for carrying out further research. From among all the nano-oxides studied, synthesis of nickel oxide is largely studied owing due to its wide array of applications such as in the manufacture of electrochromic, films, magnetic materials, p-type transparent conducting films, gas sensors, catalyst, alkaline batteries cathode, and solid oxide fuel cells anode **[4-7]**. From among all the methods used, chemical precipitation method isfound to be an effective method in the synthesisof crystalline oxide nanopowders. The use of surfactant is in fact

somehow similar to the reverse - micellar methodwhich has been recently demonstrated as a versatile route to produce a variety of nanoparticles **[8, 9].**

The main objective of this paper was to synthesize NiO nanoparticles by chemical precipitation method using different surfactants which are low-cost and inexpensive.

2. Experiment

2.1.Chemicals required: Nickel nitrate hexa hydrate ($Ni(NO_3)_2.6H_2O$), NaOH, Polyethylene glycol **(PEG)**, Polyvinylpyrollidine **(PVP)**, Sodium dodecyl sulphate **(SDS).** All the chemicals and solvents used were of analytical grade.

2.2. Instrument: SEM images for the synthesized samples were recorded on a JEOL JSM 6390 Scanning Electron Microscope, whereas, the AC-Impedance studies were carried out on a CH660 D Electrochemical work station respectively.

2.3. AC-Impedance studies: The nanopowders of the above mentioned process were made as a paste by mixing it with polyvinyl alcohol **(PVA)** and ethanol. The paste was then coated onto the working electrode and was then dipped in a solution containing 0.01 M dil H_2SO_4. The solution consisted of various electrodes such as platinum electrode and saturated calomel electrode. The following parameters were used to obtain the data.

Initial E (volts) : 0.05

Frequency : 10000

Low frequency : 0.01

Amplitude : 0.005

After a time period of 500s, the graphs were obtained in more or less of a straight line.

2.4.Procedure: Two separate solutions were prepared for this experiment.

a) A solution of 8.7g of $(Ni(NO_3)_2.6H_2O$ dissolved in 60 ml of distilled water
b) Solution of 3g NaOH dissolved in 150 ml of distilled water
c) 1g of PEG, PVP & SDS wereadded separately to obtain three different solutions respectively

Here, the former solution was added dropwise *ie,* with the help of the burette into the solution. The solution was then stirred on a magnetic stirrer for approximately 15 mins. The resultant light green solution was then filtered and washed with distilled water and ethanol and then dried at ~ 50º for about 2 hours. The dried samples were calcined at 300º, 450º and 600º for about 30 min. A greyish black product was obtained on calcination. The same procedure was followed for the remaining different surfactants used.

The reactions which occur during this process, is as follows

$$NaOH_{(s)} \rightarrow 2Na^{+}_{(aq)} + 2OH^{-}_{(aq)}$$

$$Ni(NO_3)_2.6H_2O_{(S)} \rightarrow Ni^{2+}_{(aq)} + 2NO_3^{-}{}_{(aq)} + 6H_2O_{(aq)}$$

$$Ni^{2+} + 2\,OH^{-}_{(aq)} + x\,H_2O_{(aq)} \rightarrow Ni(OH)_2.xH_2O$$

$$Ni(OH)_2 \rightarrow NiO + H_2O$$

3. Results and discussion

NiO nanoparticles were synthesized by chemical precipitation method using different surfactants such as **PEG, PVP & SDS.**Y. B. M. Mahaleh*et al***[4]**carried outthermogravimetric studies which were in good agreement with other reports from literature which states that the $Ni(OH)_2$ precipitate is formed at pH 9 followed by thermal decomposition of the molecule to convert $Ni(OH)_2$ into NiO which further gets dehydrated to form NiO nanoparticles. The fabrication process can be divided into four segments:

The first one is related to the evaporation of the absorbed water at 100ºC, at which most of the water molecule gets completely eliminated. In the second region, the residue water is eliminated at > 100ºC. There is a significant weight loss in the third region due to the calcinations of structural water molecule. The intermolecular hydrogen bond exists between water molecules and thus gets eliminated at 300 ºC, which leads to the formation of pure NiO nanoparticles.

3.1.SEM studies:Figs1-3 shows the SEM images of the product synthesized by chemical precipitation method using **PEG, PVP & SDS** respectively. From the SEM images, it can observed that NiO nanoparticles were successfully synthesized in the particle size range of 30-60 nm. In case of NiO nanoparticles synthesized by using PEG, the minimum particle size was 50 nm, while the maximum particle size seen was of 60 nm. It can be clearly seen that the particle size distribution is not uniform. Whereas most of the particles formed using **PVP** surfactant sample were of more uniform size of ~45 nm, while the minimum size distribution was found to be 39 nm. NiO nanoparticles synthesized by using SDS surfactant were of uniform size having a particle range of~ 50 nm with uniform distribution. From the SEM image of NiO nanoparticles synthesized by using **PVP** surfactant, most of the particle size was observed in the range of 39-45 nm. This showed that the surfactant **PVP** has a higher reduction capacity in comparison with **PEG & SDS** surfactants. The control in size reduction by the surfactant is due to intermicellar forces operating between NiO nanoparticles and surfactant**[8]**.

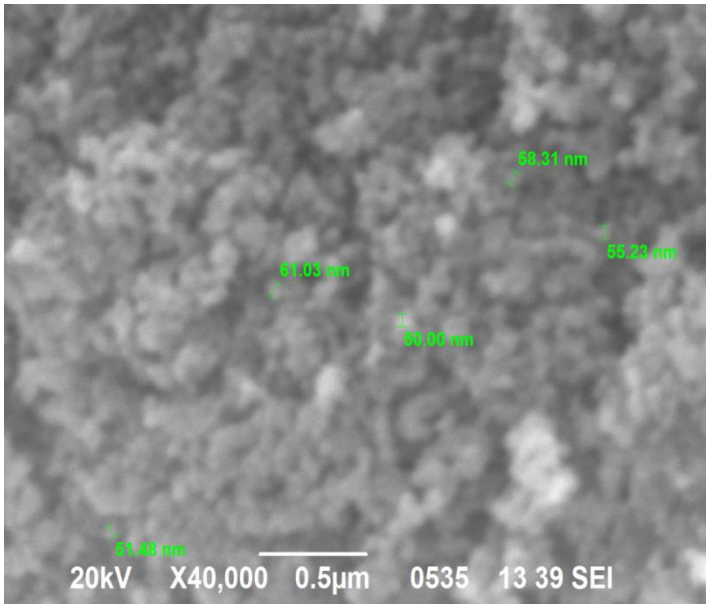

Fig 1: SEM image of NiO nanoparticles synthesized using **PEG**

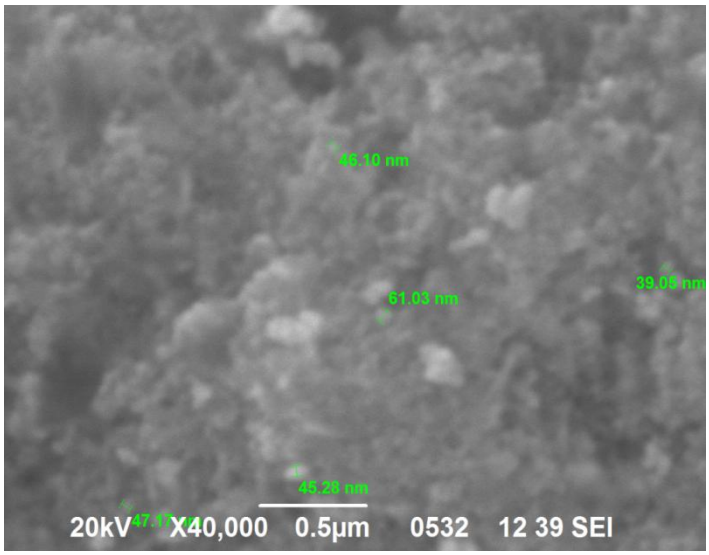

Fig 2: SEM image of NiO nanoparticles synthesized using **PVP**

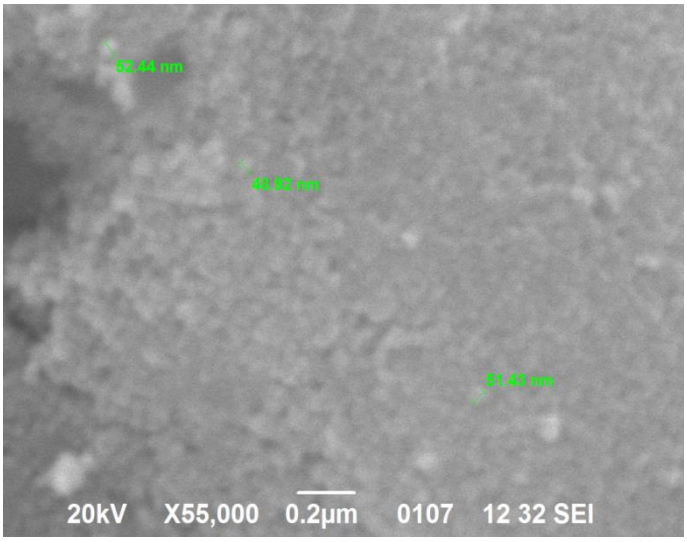

Fig 3: SEM image of NiO nanoparticles synthesized using **SDS**

3.2.X-Ray diffraction studies: The XRD patterns of the NiO nanoparticles synthesized by chemical precipitation methodusing different surfactants (**Figs 5-7)**showed a standard peak of $Ni(OH)_2.H_2O$ which is in agreement with the diffraction peaks of $Ni(OH)_2.H_2O$ and which is consistent with predicted reactions as mentioned earlier.The peaks seen are in good agreement with all the surfactants used in the process. The 2θ values observed in all the figures were 37.5, 43.5 and 63.5 respectively which confirmed the presence of NiO nanoparticles.On comparing the XRD standard table with respect to the samples, the diffraction peaks obtained by using **PVP** surfactant was found to be in a very good agreement with the diffraction peak of $Ni(OH)_2.H_2O$. The XRD-calcined product obtained by using PVP, showed better crystallization of NiO particles and elimination of the former phase (NiOH). From this study, it was observed that by increasing the calcinations temperature resulted in sharp diffraction peaks, which indicates that bigger particle size may be generated from higher calcination temperatures.

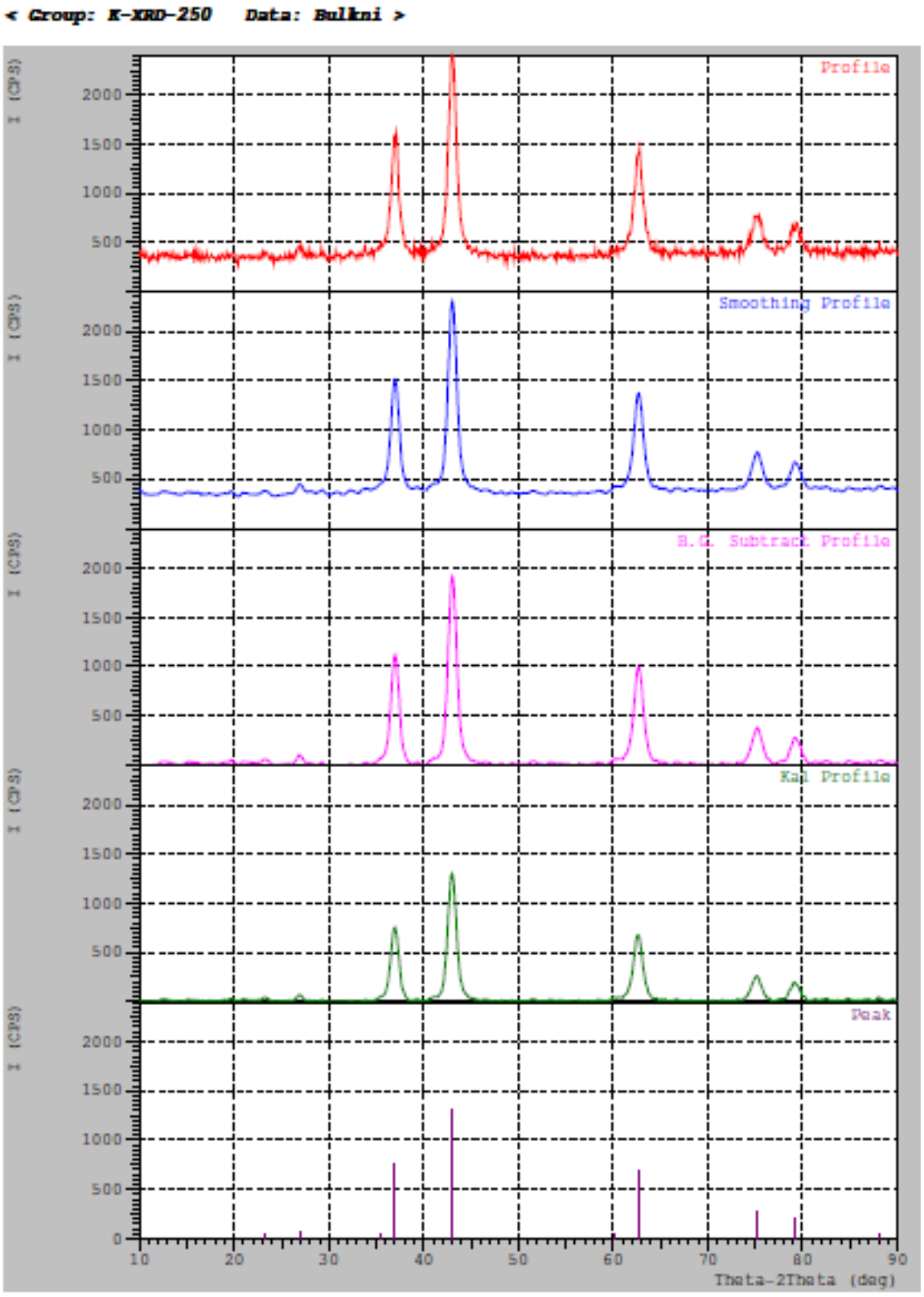

Fig 4: XRD image of Bulk NiO particles

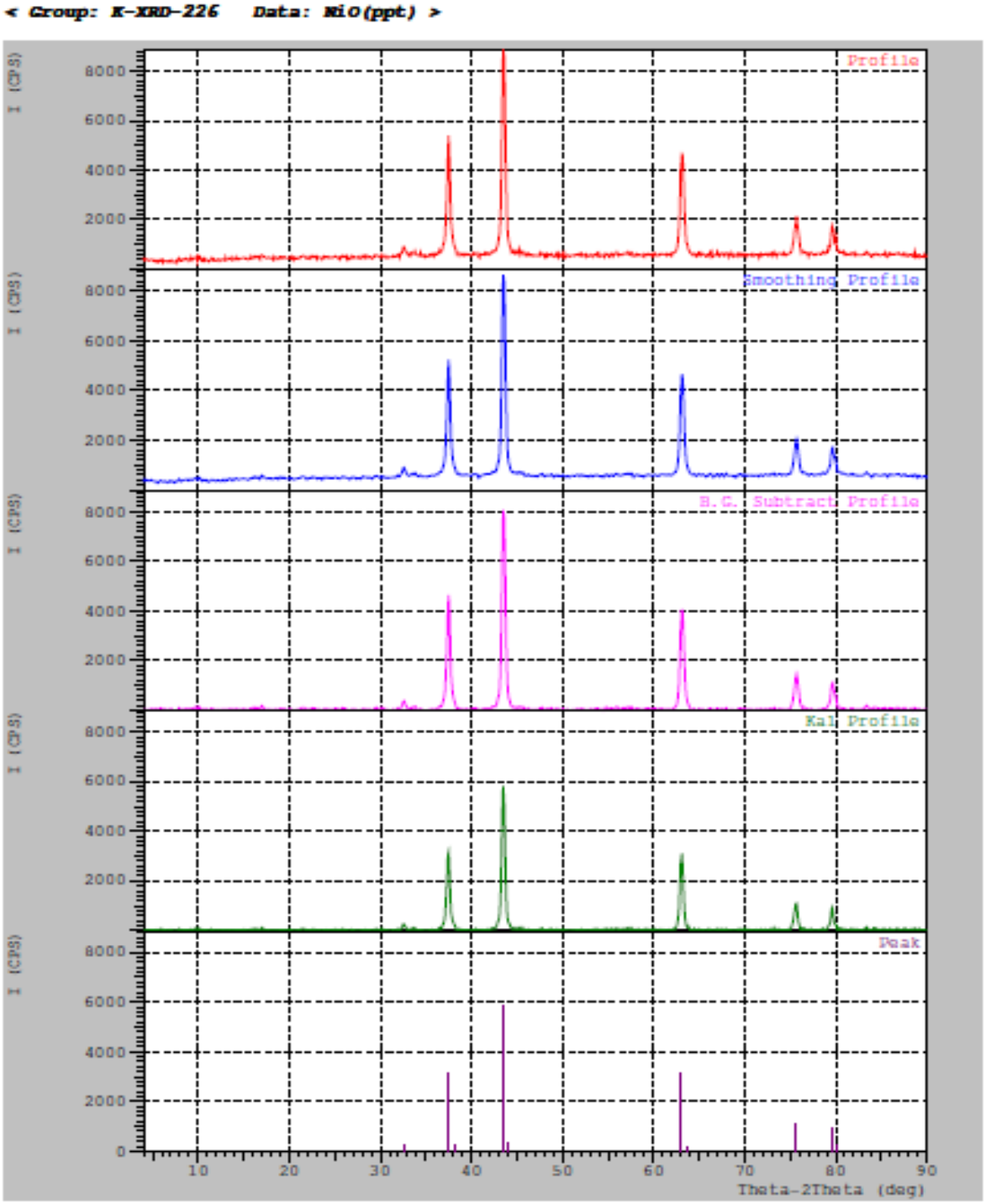

Fig 5: XRD image of NiO nanoparticles synthesized using **PEG**

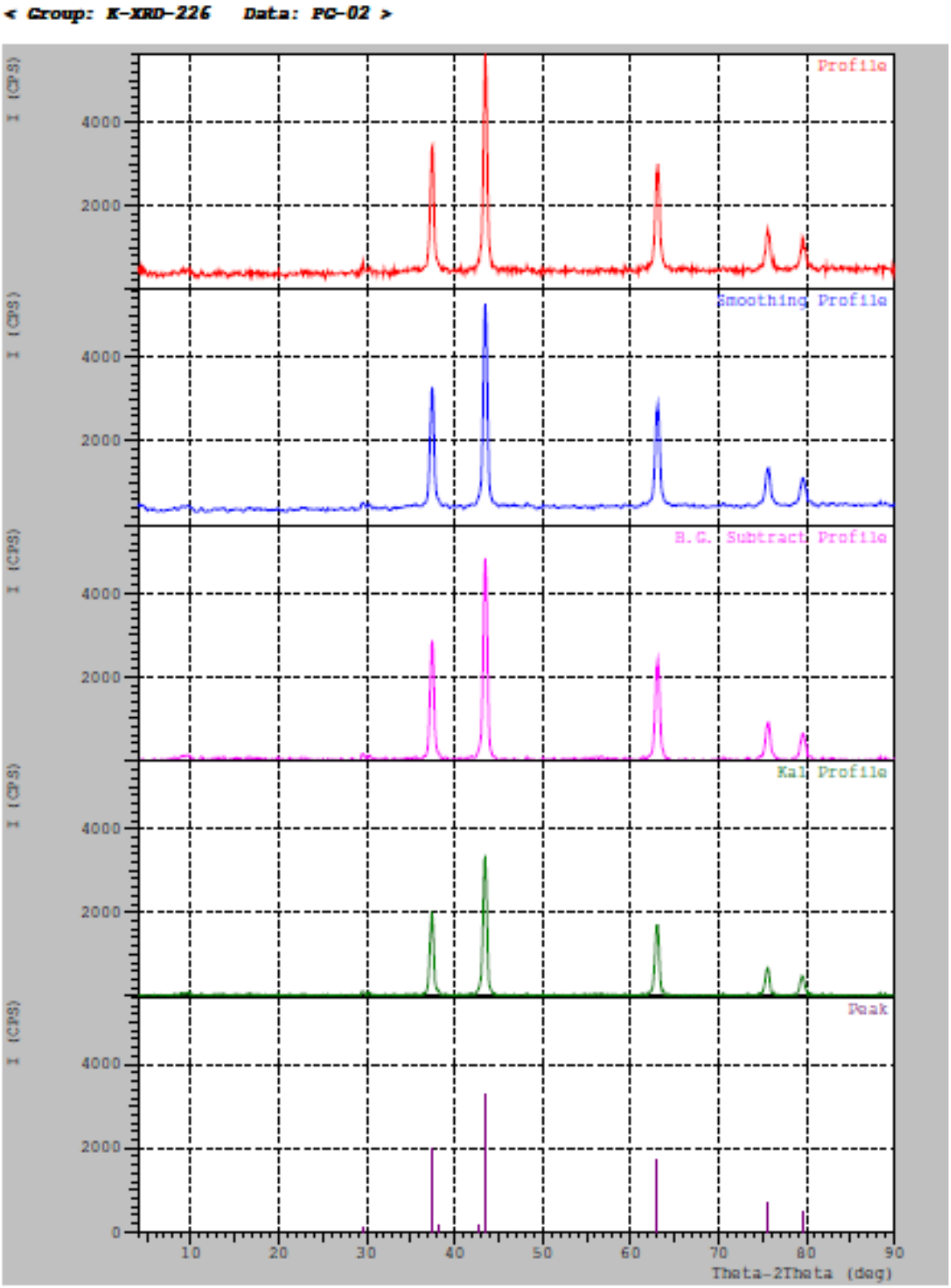

Fig 6: XRD image of NiO nanoparticles synthesized using **PVP**

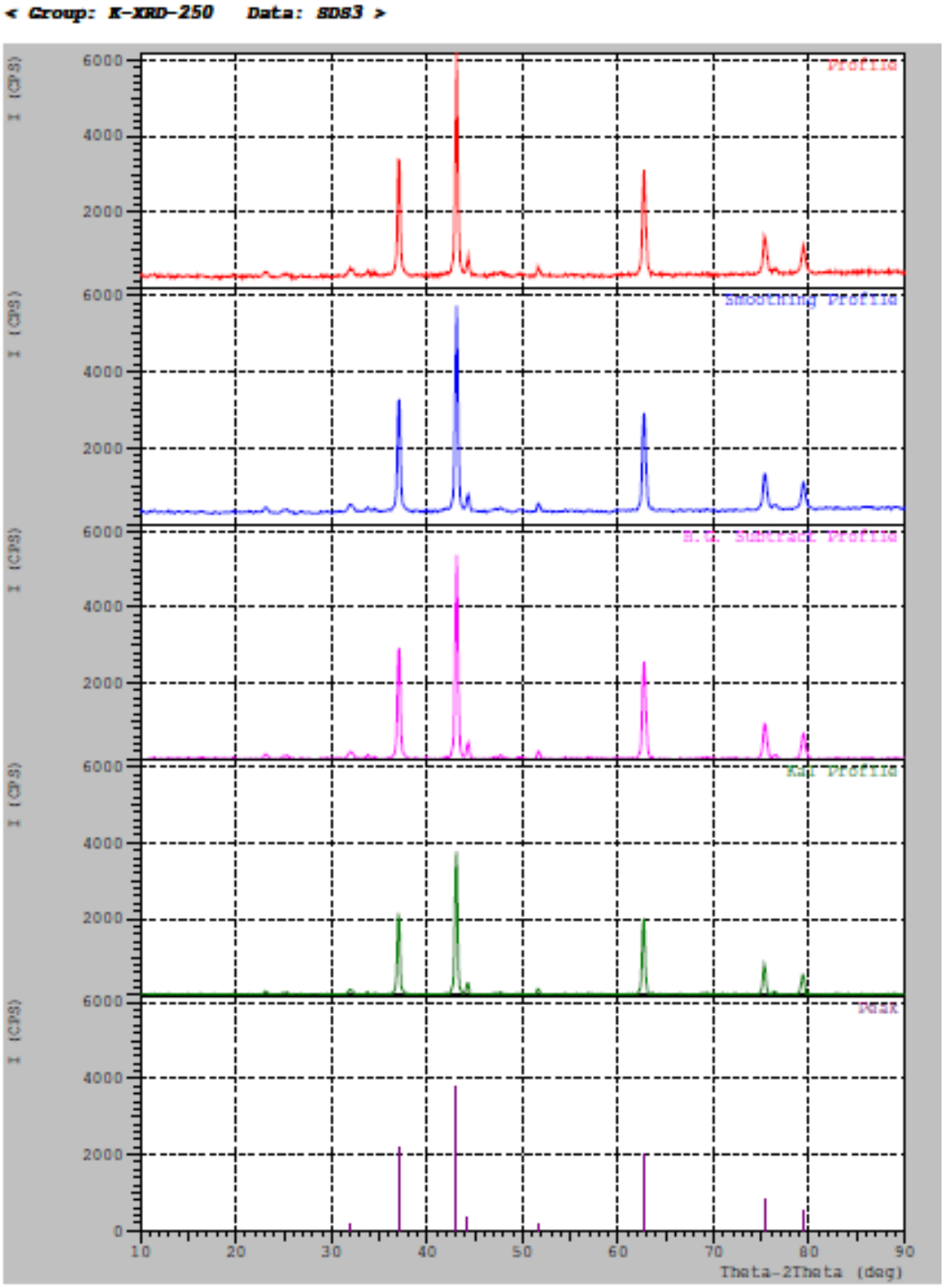

Fig 7: XRD image of NiO nanoparticles synthesized using **SDS**

3.3.Particle size distribution: On plotting a graph of the number of particles versus the size of the particles**(Figs9-11)**, it was found that the NiO nanoparticles obtained by using **PEG**, **PVP**&**SDS** were approximately in the range of 55 nm, 30-60 nm & 50 nm respectively. All the particles showed narrow size distribution and almost uniform shape.

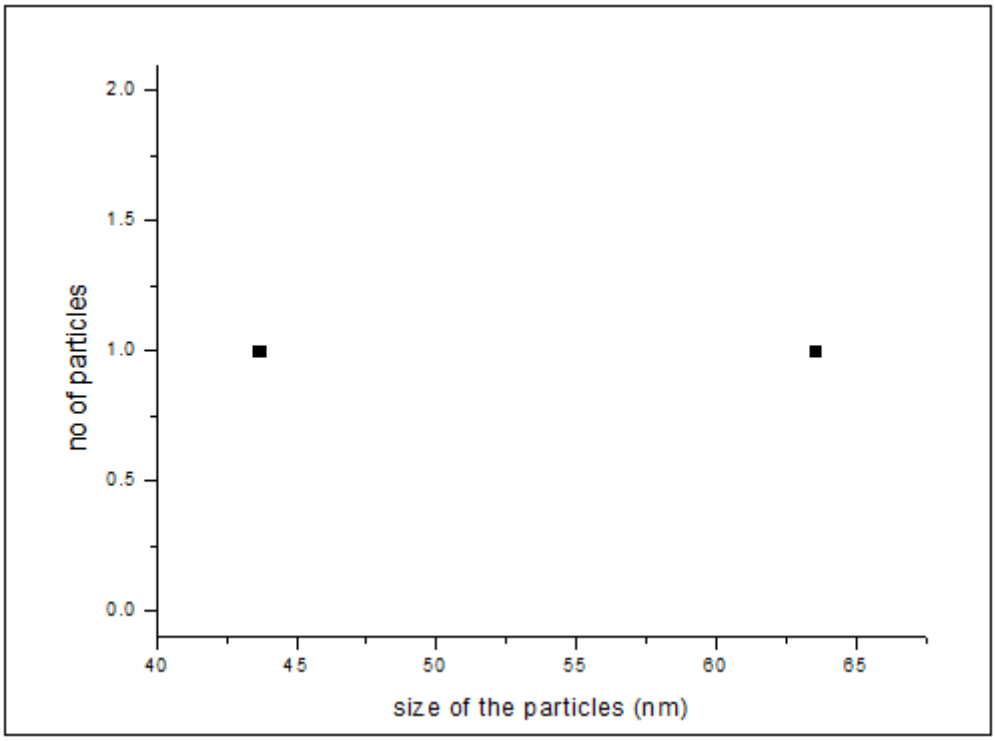

Fig 8: Graph of number of particles against size of particles of Bulk NiO

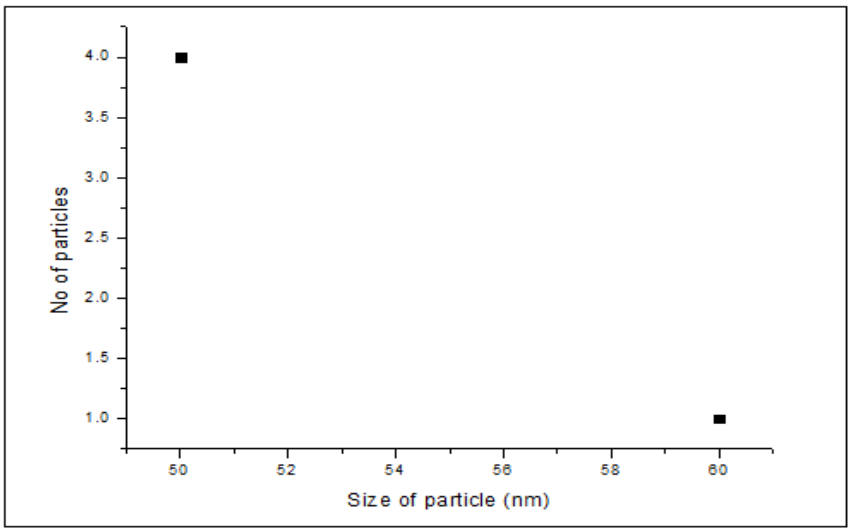

Fig 9: Graph of number of particles against size of particles using **PEG**

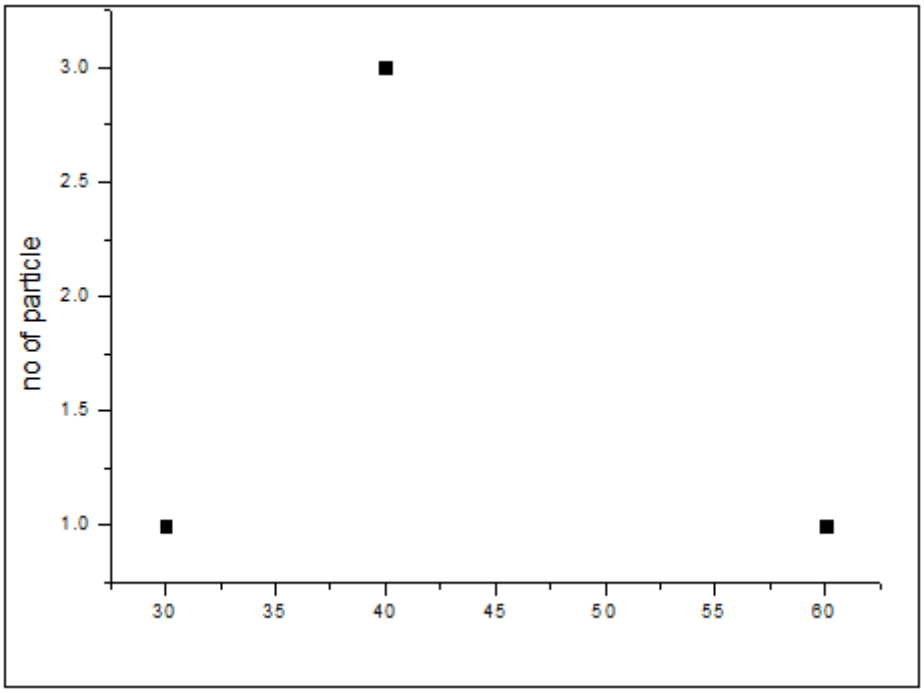

Fig 10: Graph of number of particles against size of particles using **PVP**

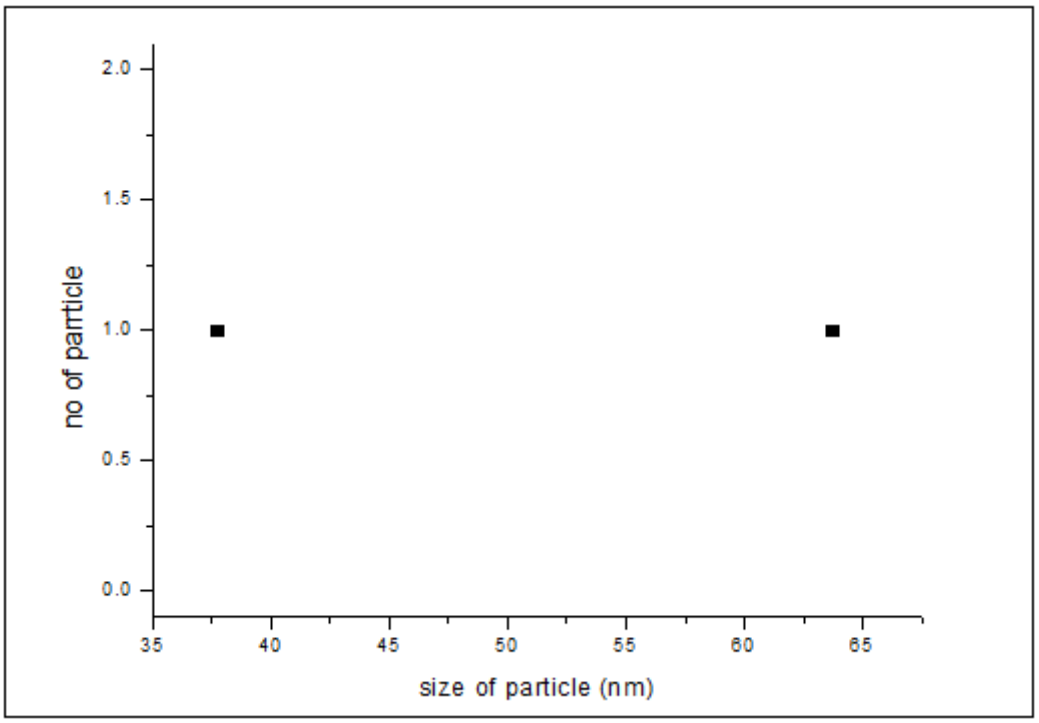

Fig 11: Graph of number of particles against size of particles using **SDS**

3.4.AC-Impedance studies:

Surfactant	Resistance (Ω)	Conductance (mF)
PEG	92.73	3.781
PVP	222.4	8.547X10-3
SDS	197.2	9.621X10-3

Table1: Calculation of resistance and conductance of NiO nanoparticles using **PEG**, **PVP&SDS**

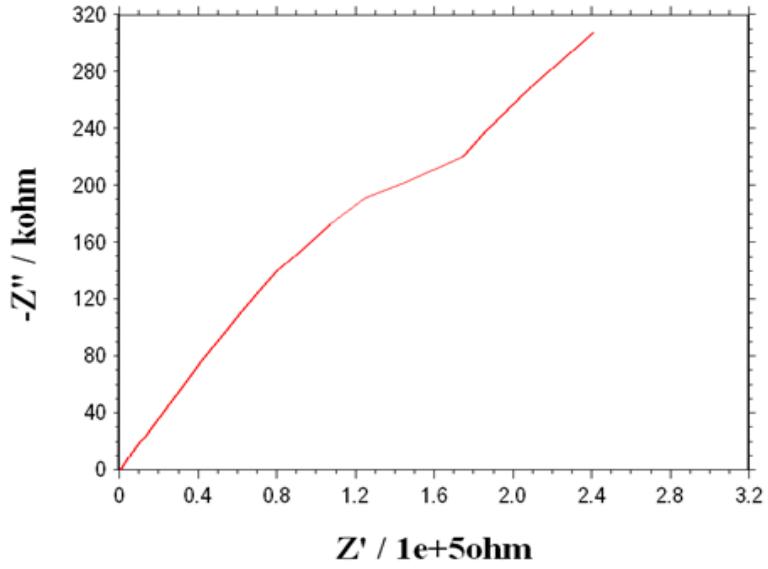

Fig 12: Graph of conductance for NiO nanoparticles synthesized using **PVP**

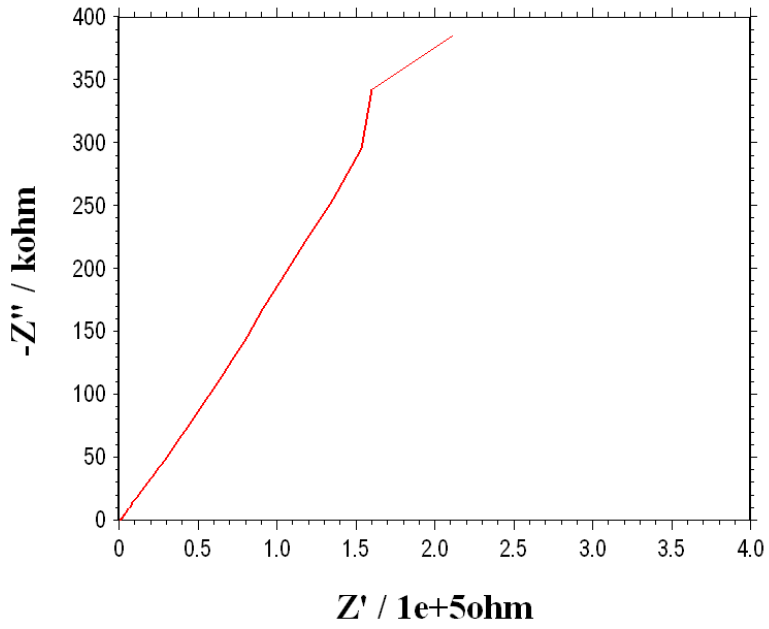

Fig 13: Graph of conductance for NiO nanoparticles synthesized using **SDS**

Conclusion: Three different surfactants namely **PEG, PVP** and **SDS** were used to synthesize NiO nanoparticles by chemical precipitation process. It is noted that NiO nanoparticles synthesized by using **PVP** as a surfactant was found to be efficient in reduction of particle size distribution and had a spherical shape with weak agglomeration as confirmed from the SEM images. Resistance and conductance properties of the synthesized NiO nanoparticles were also studied and are compared. From the studies, it was observed that **PVP** acted as a better surfactant. Based on its resistance and capacitance values, it can be concluded that it does not only serves as a good surfactant in the synthesis of NiO nanoparticles, but is also can be used widely for a large number of products especially in the applications of capacitive and supercapacitive materials owing due to its better resistance power.

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