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SYNTHESIS OF NiO NANOPARTICLES USING THESPESIA POPULNEA LEAVES BY GREEN SYNTHESIS METHOD

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ABSTRACT
Green synthesis of metal nanoparticles is an interesting and expanding research area due to the potential applications for the ecofriendly development of novel technologies. Pure and NiO nanoparticles were prepared by green synthesis method using Thespesia populena leaf extract and nickel sulfate as raw materials. The synthesized NiO nanoparticles were characterized by powder X-ray diffraction, UV-Vis analysis, FTIR analysis, SEM and EDAX analysis. From UV spectrum the optical properties of the samples were determined. FTIR spectrum reveals the various functional groups present in the sample. SEM image shows the surface morphology of pure and NiO nano particles. From EDX the presence of Ni, and O were confirmed. VSM confirms the super paramagnetism of NiO nanoparticles.
KEYWORDS: Green synthesis, NiO, XRD, UV, FTIR, SEM, VSM.

1. INTRODUCTION

The word Nano meaning dwarf in Greek is already in the market on the wide range of applications. As the definition above, systems in which the dimensions measure in the range of nanometers are covered under the large canopy of the nanoscience and nanotechnology [4]. The nanoscience and nanotechnology has taken an upswing over the frontiers in scientific research. The nanoparticles can be of different types comprising of metal or metal oxide nanoparticles, organic/inorganic nanoparticles, quantum dots, polymeric nanoparticles, liposome, DNA machines, and carbon nanotubes (CNT), graphene, etc [8]. Nanoscience and nanotechnology thus encompass a range of techniques rather than a single discipline and stretch across the whole spectrum of science, touching medicine, physics, engineering and chemistry. There are several methods for creating nanoparticles, including attrition, pyrolysis, hydrothermal synthesis and green synthesis. Most commonly method of solvo thermal, co-precipitation method, bottom-up and top down synthesis, sol-gel synthesis method green synthesis and chemical synthesis [9]. Green synthesis is a cost-effective

system and eco-friendly system [13]. Green synthesis using many fields in various application and it's used to day-to-day life. Green synthesis of nanoparticles is cost-effective and eco-friendly system. Plant- mediated synthesis of nanoparticles is a green chemistry approach that connects nano techonolgy with plants. plants are nature's 'chemical factories'[17]. Green synthesis of Nanoparticles, their small size, so it can penetrate smaller capillaries and are taken up by cells, which allow efficient drug accumulation at the target[11].

The bark of thespesia populnea is sharp in nature. Usually known as the Portia tree, Pacific rosewood, or milo, among other names, is species of flowering plant belonging to the mallow clan, Malvaceae. It is a small tree or arborescent bush that has a pantropical spreading, found on coasts around the world [15]. The Portia tree spreads a height of 6–10 m . Tall and its bole can measure up to 20–30 cm in diameter[1]. It grows at elevations from sea level to 275 m. in areas that receive 500–1,600 mm of annual rainfall [7]. The Portia tree is able to grow in the wide range of soil types that may be present in coastal surroundings, including soils derived from quartz (sand), limestone, and basalt; it favoritisms neutral soils [16].

However, all these green protocols for synthesis of NiO NPs were simple, efficient, and eco-friendly and did not require ample reactants, draggy procedures, and complex apparatus which were required in case of conventional chemical synthetic methods [2]. The synthesis of nickel and nickel oxide nanoparticles by green chemistry is beneficial due to eco-friendliness, economic prospects, feasibility, enhanced biocompatibility, low cytotoxicity, and high antioxidant and high antimicrobial activity of formed nanoparticles [5]. These features help in commercialization of Ni and NiO NPs in the fields of environmental cleaning and nanomedicine.

Synthesis of NiO NPs with by using green synthesis as surfactant. Furthermore, NiO NPs was used as nano catalyst for an efficient and very simple in the solvent free green synthesis [14]. This catalyst is expected to contribute to the development of more environment-benign methods and forms part of nano-metal.[6]. The mildness of the con-version, experimental simplicity, compatibility with various functional groups, excellent harvests, shorter reaction time, and the easy work-up procedure makes this procedure more attractive in synthesizing a variety of these products [10].

2. OBJECTIVES

- To synthesize NiO nano particles using the preferred leaf extracts.
- To characterize the structural properties of NiO nano particles using powder xrd, SEM and EDAX studies.
- To study the magnetic behaviour using VSM analysis.
- To analyses the optical characteristics using UV and Photoluminescence studies.

3. METHODOLOGY

3.1 Synthesis of Leaf Extract

The Thespesia populnea leaves were collected from Thenkasi surrounding area which is near the western ghats region. The leaves were dried for one day under the shadow and are thoroughly washed with distilled water to remove dust particles and sun dried to remove moisture content and these leaves are cut into small pieces. Then the leaves are boiled in distilled water in a flask for 6 hours and placed in a water bath at constant temperature for three hours at 400 ^{0}C. The solution was filtered and finally got 100 ml leaf extract is obtained and the extract was filtered using whatmann filter paper and as a result we obtain leaf extract.

3.2 Synthesis Of Nickel Oxide Nanoparticles

Nickel sulphate was used as precursor for synthesizing nickel nanoparticles solution of concentration 1M was taken and was mixed with 100ml of double distilled water . In order to synthesize the nanoparticles, leaf extract was mixed with metal salt of 1M and stirred. The immediate color change will be occurring and the color change into dark green within the mixture indicates the presence of NiO nanoparticles. This mixture was filtered using Whatmann filter paper and the precipitate was obtained was cleaned twice by distilled water and then kept in oven under 500˚C. Finally we obtained the fine powder of NiO nanoparticles and it was calcinated at 600^{0}C.

4. RESULTS AND DISCUSSION

The synthesized NiO nanoparticles are subjected to various characterization studies to study its structural, optical and magnetic properties such as powder XRD, UV –visible spectrometer, FTIR spectrometer, SEM, energy dispersive X-ray, and VSM analysis. The results obtained are discussed below.

4.1 Powder XRD Studies

Powder XRD is used to determine the identification of purity and quantitative analysis of nanoparticles are explained by the XRD pattern. Powder Xrd spectra of NiO nanoparticles are given in Fig.1. The spectra shows diffraction peaks appeared at 2θ values of 37.40, 43.44 ,63.01 reflections can be indexed to the known rhombohedral structure of NiO with lattice constants of a = 2.954 Å, c = 7.226 Å, the

peaks of the diffraction pattern are compared with standard available data for the confirmation of the JCPDS card no. 897390. It shows the presence of (111), (200), and (220) planes. The XRD pattern confirms the formation of NiO and the broad peaks correspond to the presence of oxygen vacancies and local lattice disorder in the sample. Calculated crystalline size of the NiO nanoparticles is around 48.9 nm.

4.2 UV – visible spectrum Studies

The UV spectral studies are taken for the NiO nanoparticles in the regions ranging between 100 to 900 nm. The absorption spectra of NiO nanoparticles interms of wavelength are shown in fig. 2. The UV spectra show that the lower cut off wave length for NiO nanoparticles are around 362 nm. From the absorption spectra, it is confirmed that no peaks are due to the phenolic compounds in the extract. The plots of variation of $(\alpha h\nu)^{1/2}$ versus $h\nu$ for the NiO nanoparticles are presented. These plots are known as the Tauc's plots and they are used to find out the accurate optical band gap value by the extrapolation of the linear part. This suggests that the optical transition in the sample is an in direct transition. Fig. 3 shows the Tauc's plot of NiO nano particles from which the calculated band gap energy is 3.3 eV.

4.3. FTIR Studies

Fourier transform infrared spectrometer involve examination of the twisting, binding, rotation and vibrational modes of the atoms in a molecule upon interaction with infrared radiation portion of incident radiation are observed at specific wavelength and functional groups of sample can be identified from the spectrum. The FTIR spectrums are recorded in the range of 500 to 4000 cm-1. The recorded spectrum is shown in the fig.4. The broad band at 3400 – 3700 cm^{-1} is because of the stretching vibration mode of the chemically bonded hydroxyl group, and it is due to the water molecules adsorbed on the surface of NiO. The adsorption of water molecules by the nanoparticles indicate that the synthesized nanomaterial possess high surface area. The bands at 1020.23cm^{-1} refer to C-O stretching bond. The band observed at 3448.92 cm^{-1} belongs to O-H stretching band. The peaks observed in 853.30 and 467.59 cm^{-1} are due to the Ni – O stretching band, vibration band. The peak is 669.45 cm^{-1} is due to Ni band. The peak observed at 1385.56 belongs to Aromatic stretching vibration band. The obtained FTIR peaks with assigned functional groups.Thus the results of FT-IR spectrum coincide with the XRD data and in turn confirm the formation of highly pure single NiO nanoparticles.

4.4 SEM Analysis

The morphology of the samples was investigated by scanning electron microscopy and it is used for morphological characterization at the nanometer to micrometer scale. Fig.5 shows the SEM analysis of the NiO nanomaterial prepared using thespesia populnea leaf extract and this figure clearly shows the surface features of the prepared NiO nanoparticles sample. From the SEM image, it is observed that the NiO nanoparticles are arranged as regular beads in shape and it is clear that the particle size are in nanometer scale and this indicates that green synthesis method is a successful method to prepare nickel oxide nanoparticles.

4.5. EDX Analysis

Energy – dispersive X-ray spectroscopy is an analytical technique used for the elemental analysis or chemical characterization of a sample. It's used to know the presence of Nickel oxide. It relies on an interaction of some source of X-ray excitation and a sample. The purity and composition of the prepared particles were analyzed by EDX. Fig shows the EDX spectrum of NiO nanoparticles from plant extract method. EDX spectrum not only identifies the elements corresponds to each of its peak, but the type of X-Ray to which corresponds well. The higher a peak in a spectrum, the more concentrated the element is in the spectrum. The peaks also reveals the percentage of elements present in the synthesized sample which clearly indicates the prepared nanoparticles contains Ni and O ions. The EDX spectra is shown in Fig.6 which confirms 20.39% of Ni element and 16.93% of O present in the prepared sample.

4.6 Vibrating sample magnetometer studies

The magnetization behavior of the synthesized NiO was investigated by sweeping the external magnetic field between +20 kOe to−20 kOe. Fig shows the plot between magnetization Vs magnetic field (H). The NiO exhibited slight curves in the low magnetic field, and a linear portion at higher magnetic field regions. It was also observed that the sample never attained saturation even at higher magnetic field of about (20 kOe), which confirms the presence of super paramagnetism. If there is a reduction in the particle size, the magnetic property will be changed from diamagnetism to super paramagnetism.

The NiO exhibited slight curves in the low magnetic field, and a linear portion at higher magnetic field regions. It was also observed that the sample never attained saturation even at higher magnetic field of about (5 kOe), which confirms the presence of super paramagnetism [18]. Hence the super paramagnetic behavior exhibited is due to the reduced particle size , which are also proven by the obtained XRD results. As the magnetic behavior is sensitive towards the magnetic impurities, the existence of super paramagnetic behavior even at higher magnetic field proves the purity of the sample. The methodology employed in synthesizing NiO nanoparticles using

green synthesis in the present work has resulted in reduced particle size with super paramagnetic behavior, and in turn proves to be a promising material for various magnetic applications. The hysteresis loops shows a maximum symmetric magnetization, M_{max} at 5.2901 E – 3 emu. The coercivity H_c of the synthesised nano particles is 145.50 Oe . The retentivity value is 131.31 E - 6 emu.

5. SUMMARY AND CONCLUSION

In conclusion, this paper provides an overview of green synthesis of nickel and nickel oxide nanoparticles by using plant extract. Although all these green protocols for NiO nanoparticles synthesis have their own advantages and limitations use of plants extract as reductant is more beneficial as compared to microbial extract because of rapid rate of production of nanoparticles with former green reductant.

Synthesis of NiO nanoparticles was carried out by the green synthesis method using Thespesia Populnea leaf extract. XRD and FT-IR confirms the formation of pure and crystalline NiO nano particles to synthesize with high purity and crystallinity, reduced particle size and more surface defects, which are the advantageous factors to be used for various environmental and applications. The UV- visible spectra shows that the lower cut off wavelength is around 362 nm and the band gap energy is calculated as 3.3eV. FTIR studies are carried out for the crystalline sample and the vibrational bands are assigned. VSM studies confirm the presence of super paramagnetism in the prepared sample. From SEM images, it is observed that the NiO nanoparticles are arranged as regular beads in shape. EDX spectrum shows the Ni & O nanoparticles are present in the prepared nanoparticles.

6. FIGURES

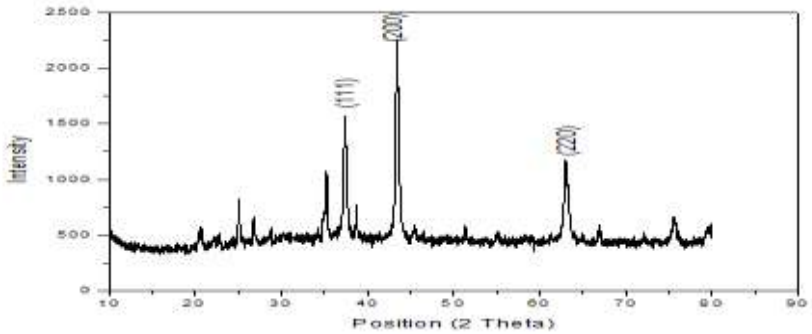

Fig. 1 XRD – pattern of NiO nanoparticles

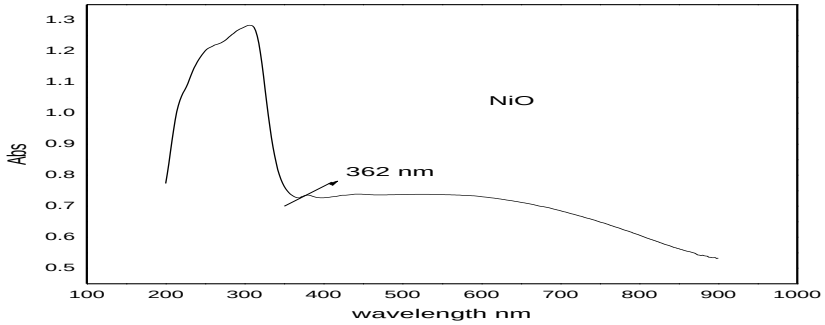

Fig. 2 UV spectrum of NiO nanoparticles

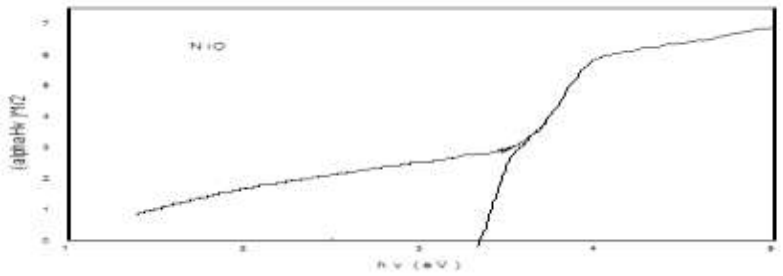

Fig -3 Tauc's plot

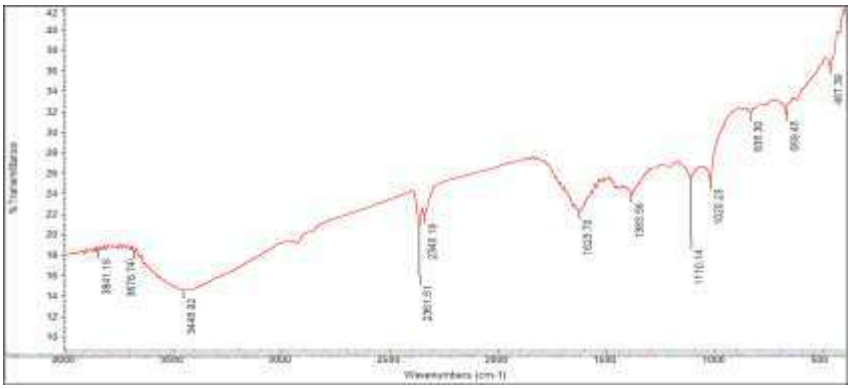

Fig- 4 FTIR spectrum of Nio nanoparticle

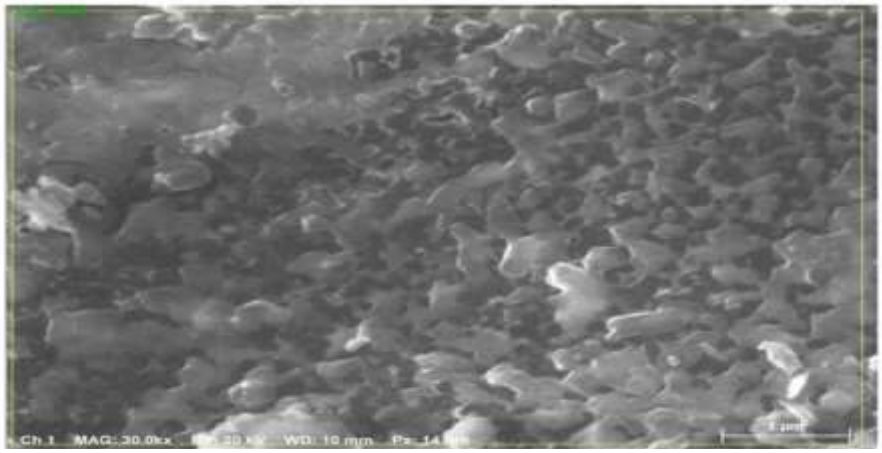

Fig. 5 SEM analysis of NiO nanoparticle

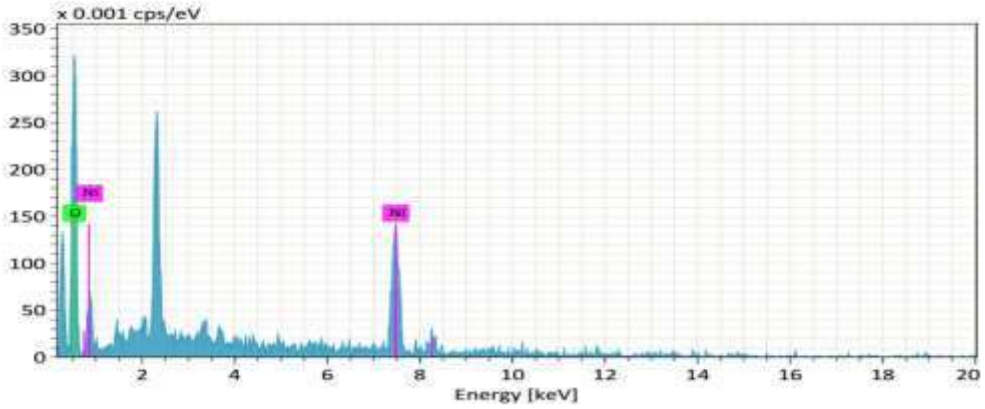

Fig 6 EDX spectrum for NiO nanoparticles

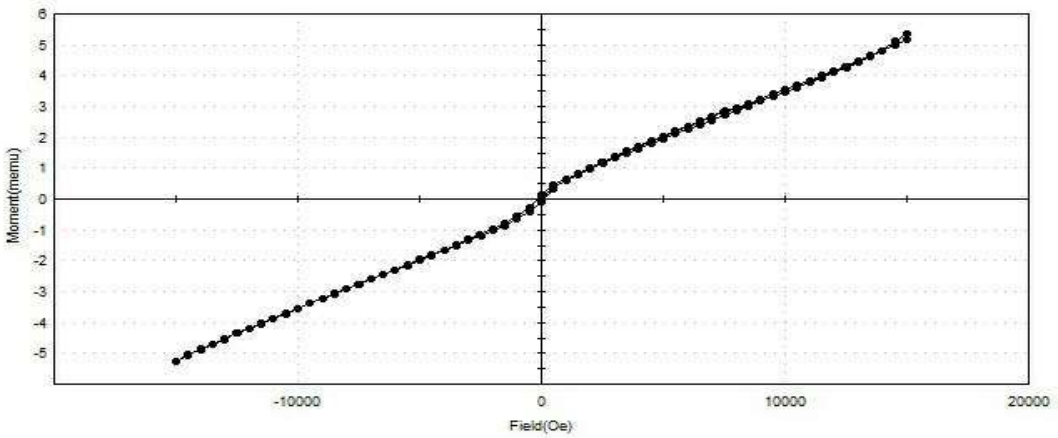

Fig 7 Magnetic hysteresis curve for NiO nanoparticles

7. REFERENCES

1. *H. Rui., R. Xing., Z. Xu., Y. Hou., S. Goo., S. Sun., (2010), "Synthesis, functionalization, and biomedical applications of multifunctional magnetic nanoparticles ", Adv. Mater. 22 p.p : 2729-2742.*
2. *K. Kaviyarasu., E. Manikandan, J. Kennedy, M. Jayachandran, R. Ladchumanananda sivam,U. Umbelino De Gomes, M. Maaza. (2016), "Synthesis and characterization studies of NiO nanorods for enhancing solar cell efficiency using photon upconversion materials", Cer. Int. 42 p.p : 8385- 8394.*
3. *E. Manikandan, J. Kennedy, G. Kavitha, K. Kaviyarasu, M. Maaza, B. K. Panigrahi, U. Kamachi Mudali. (2015), " Hybrid nanostructured thin-films by PLD for enhanced field emission performance for radiation micro-nano dosimetry applications", J. Alloys & Comp. 647 p.p:141-145. (2002)*
4. *I. Brigger, C. Dubernet, P. Couvreur. (2002) , " Nanoparticles in cancer therapy and diagnosis", Adv. Drug Deliv. Rev. 54 p.p:141-145. .*
5. *K. Kaviyarasu, A. Raja, Prem Anand Devarajan. (2013), "Structural elucidation and spectral characterizations of Co3O4 nanoflakes Spectrochimica Acta", Part A: Mol. & Biomol. Spec. 114 p.p:141-145.*
6. *F. Fang, J. Kennedy, D.A. Carder, J. Futter, P. Murmu, A. Markwitz. (2010), "Modulation of Field Emission Properties of ZnO Nanorods " During Arc Discharge, J. Nanosci. & Nanotech. 10 p.p:141-145.*
7. *F. Fang, J. Futter, A. Markwitz, J. Kennedy (2009), "UV and humidity sensing properties of ZnO nanorods prepared by the arc discharge method ", Nanotechnology 20 p.p:141-145.*
8. *A.K. Gupta, M. Gupta (2005), "Cytotoxicity suppression and cellular uptake enhancement of surface modified magnetic nanoparticles, BioMater. 26, p.p:.1565-1573.*
9. *R. Joerger, T. Klaus, C.G. Granqvist (2000), "Biologically produced Ag-C composite for optically functional thin film coating "s, Adv. Mater. 12, p.p:407-409.*
10. *K. Kavi "yarasu, D. Sajan, Prem Anand Devarajan. (2013), "A rapid and versatile method for solvothermal synthesis of Sb2O3 nanocrystals under mild conditions ", Appl. Nanosci. 3 p.p:529-533.*
11. *P. Laokul, V. Amornkitbamrung, S. Seraphin, S. Maensiri. (2011), "Growth of p-type ZnO*

thin film on n-type silicon substrate and its application ", Curr. Appl. Phys, p.p:101- 111.

12. *A. Kar and A. K. Ray. (2014), " Synthesis of nano-spherical nickel by templating hibiscus flower petals ", J. Nanosci. Nanotechnol. 2, p.p:17-20.*
13. *J.F. Morton. (1991), "The horseradish tree, Moringa pterygosperma (Moringaceae) a boon to arid lands, "Econ. Bot. 45, p.p: 318-333.*
14. *P.H. Chuang, C.W. Lee, J.Y. Chou, M. Murugan, B.J. Shieh, H.M. Chen (2007), "Antifungal activity of crude extracts and essential oil of Moringa oleifera Lam, Biores ". Technol. 98, p.p: 232-236.*
15. *K. Kaviyarasu, Prem Anand Devarajan, S. Stanly John Xavier, S. Augustine Thomas, S. Selvakumar (2012), "One pot synthesis and characterization of cesium doped SnO2 Nanocrystals via a hydrothermal process, J. Mat. Sci. & Tech. 28, p.p: 15-20.*
16. *S.G. Mahajan, A.A. Mehta, (2008), " Effect of Moringa oleifera Lam., seed extract on oval bumin- induced airway inflammation in guinea pigs ", Inhal. Toxicol. 20 p.p: 897-909.*
17. *A.A. Mariam, M. Kashif, S. Arokiyaraj, (2014), "Bio-synthesis of NiO and Ni nanoparticles and their characterization, Dig. J. Nanomater. Bios. 9 , p.p:1007-1019.*
18. *M. Chen, Y. Zhang, B. Huang, (2013) , "Evaluation of the antitumor activity by Ni nanoparticles with verbascoside ", J. Nanomater, p.p:1-6.*