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Facile synthesis and optimization of nickel oxide nanoparticles using *Polianthes tuberosa* extract and its anticancer activity

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ABSTRACT

NiO nanoparticle has been synthesized by a greener method using *Polianthes tuberosa* plant extract. The nanoparticle was characterized by UV-Visible, FT-IR, XRD and SEM instrumental techniques. The absorption band appeared at 269 nm in UV-Visible spectrum supported the formation of NiO nanoparticles. The IR spectrum analysis showed a broad band at 554 cm⁻¹ characteristics of NiO nanoparticles. From the XRD results, the crystalline size and shape of NiO nanoparticles was determined to be 3.23 nm with a face centered cubic crystal. The NiO nanoparticles has been distributed well. From SEM results, the synthesized NiO sample has particle size between 5 and 11 nm range. The cytotoxic results showed significant activity of the synthesized NiO nanoparticles against MM2 and HeLa cells.

Keywords: X-Ray diffraction, Infra-Red spectrum, *Polianthes tuberosa*, Anticancer studies, SEM.

INTRODUCTION

Nanotechnology is a technology developed from the knowledge of physics, chemistry, biology and engineering. Based on their structure and shape, they are called as nano-tube, nano-cubes, nanoflowers, clusters etc., (Gray & Luan, 2002; Hosokawa *et al.*, 2007; Laurent *et al.*, 2008). Nowadays, due to the expansion of various industries which are manufacturing commercial products create different types of pollution in the environment. It may be in the form of water, air and soil pollution (Wojcieszak *et al.*, 2010; Rao *et al.*, 2014). These pollutants retard the intrusion of sunlight through water and affects the marine animals. The scarcity of drinking water prohibited by following waste water treatment techniques. Among various techniques involved in waste water treatment, plant-based metal oxide nanoparticles are considered as one of the most favourable tool. Green synthesis of nanoparticle is a harmless method. Biologically active molecules present in the plant parts like leaf, flower, stem, seed etc., are acting as the reducing agent, capping agent and stabilizing agent (Baker, 2006; Azhir *et al.*, 2012; Burt *et al.*, 2013). Nickel oxide nanoparticles are having peculiar characteristics and due to this, they are having multiple applications like heterogeneous catalyst and battery research etc., (Ishihara *et al.*, 1991; Blumenthal, 2004). Especially, NiO

nanoparticles prepared by green method are preferred because of its eco-friendliness (Wolffarth, 1980; Lanje *et al.*, 2010). Even though, metal oxides are available as semiconductors, NiO nanoparticles are considered as an important metal oxide because of its non-hazardous nature, stability at light radiation and easily availability (Mitsuyu *et al.*, 1982). In recent days various research is in place to synthesize new antibiotics without creating any side effects and to find out drug carrier in smaller size is a big battle of the Researchers in medicinal field (Regan & Gratzel, 1991). NiO nanoparticles showed appreciable anticancer activity against human cancer cell lines like cervical cancer and breast cancer cell lines (Naazeeruddin *et al.*, 1993; Bjoerksten *et al.*, 1994).

Lycorine is an alkaloid present in Tuberose bulbs and causes vomiting. The paste prepared by rubbing these bulbs with turmeric and butter was applied over the red pimples of infants. The powdered form of the dried tuberose bulbs is used as a remedy for gonorrhoea. In Java, along with the vegetable juices, the flowers are eaten. Benzyl benzoate, methyl isoeugenol, benzyl salicylate, methyl 2-amino benzoate, methyl isoeugenol, geraniol and 1,8-cineole are the main compounds present in *Polianthes tuberosa*. Javed *et al.* (2020) synthesized nickel oxide nanoparticles using *Rhamnus triquetra* leave.

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Different physicochemical properties were investigated using analytical techniques such as UV-Visible spectroscopy, XRD, FT-IR spectroscopy, SEM, TEM, EDS, DLS and Raman. On subjecting *Rhannus triquetra* NiO nanoparticles for different in vitro biological activities, it exhibited a distinctive biosafe and biocompatibility potential using erythrocytes and macrophages. The cytotoxicity of the synthesized NiO nanoparticles was investigated against breast cancer and cervical cancers using MM2 and HeLa cell lines.

Even though many methods are available for the synthesis of NiO nanoparticles, in the present work we have reported a green synthesis of nickel oxide nanoparticles using *Polianthes tuberosa*.

MATERIALS AND METHOD

Polianthes tuberosa were purchased from local flower market in Coimbatore, Tamil Nadu and nickel nitrate were used for the synthesis.

Preparation of *Polianthes Tuberosa* Extract

50 g of weighed *Polianthes tuberosa* was crushed thoroughly and mixed with water in the ratio of 1:2. The mixture was refluxed for about 60 minutes. After cooling to the room temperature, the mixture was filtered. The extract was used as the reducing agent.

Synthesis of NiO Nanoparticles

The extract was added to 0.1 N nickel nitrate solution in the ratio of 1:5 and stirred in a magnetic stirrer for about 6 hours. The precipitate formed was dried to get a fine powder. The NiO nanoparticles powder is in brown colour. The prepared nickel oxide nanoparticles were characterized by different analytical techniques. The size, morphology and shape were obtained from IR, UV-Visible, XRD and SEM.

Anticancer Activity

Anticancer activities of NiO nanoparticles synthesized from *Polianthes tuberosa* were evaluated and compared. Anticancer activity of the synthesized nanoparticle has examined by MTT assay with the two most common cancer cells, MM2 (human breast cancer cells) and HeLa (cervical cancer cell line) according to the literature methods.

$$\% \text{ of viability} = \frac{\text{Mean OD of test samples}}{\text{Mean OD of control group}} \times 100$$

RESULTS AND DISCUSSION

The Ultraviolet-Visible spectroscopy was used to fix the production of NiO nanoparticles. A band present in 269 nm is characteristic peak of NiO nanoparticles is represented in Figure 1. The bulk value for NiO nanoparticles is at 230-280 nm. We got the band in appreciable range (Hosokawa *et al.*, 2007).

The FT-IR spectrum of NiO nanoparticles exhibited characteristic stretching vibrations for the different structural forms of metal. FT-IR spectrum of NiO nanoparticles shown in Figure 2. The stretching frequency found at 554 cm^{-1} , confirmed the NiO nanoparticles. The absorption bands exhibited in between 1066 and 2316 cm^{-1} corresponds to benzyl benzoate, methyl 2-amino benzoate, methyl isoeugenol, isoeugenol, benzyl salicylate, methyl salicylate, geraniol and 1,8-cineole present in the *Polianthes tuberosa* extract (Hosokawa *et al.*, 2007).

Figure 3 shows the XRD pattern of NiO nanoparticles. The observed “2 θ ” values come in good agreement with standard “2 θ ” values. This confirms that the prepared compound was NiO. The size of the NiO nanoparticles was observed to be 3.23 nm. From the peak values and corresponding lattice planes, it was confirmed that the NiO nanoparticles are having a face-centered cubic structure. It was further evidenced by the JCPDS card No. 89-5881 (Hosokawa *et al.*, 2007). Table 1 shows the details of XRD of NiO nanoparticles.

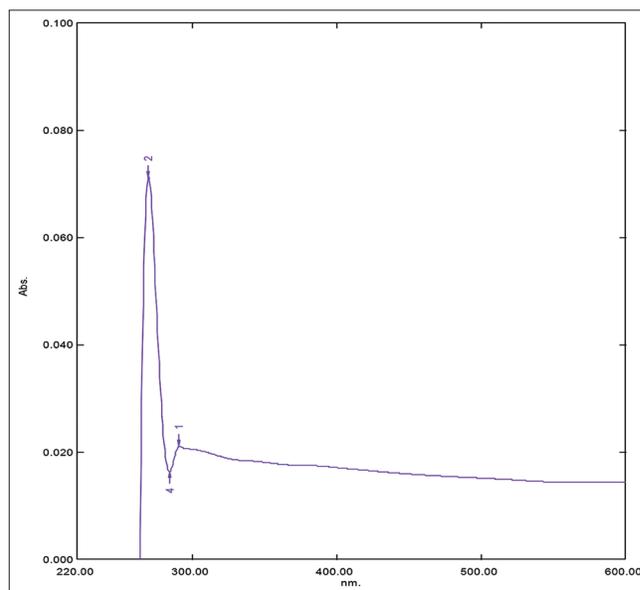


Figure 1: UV-Visible spectrum of NiO nanoparticles (*Polianthes tuberosa*)

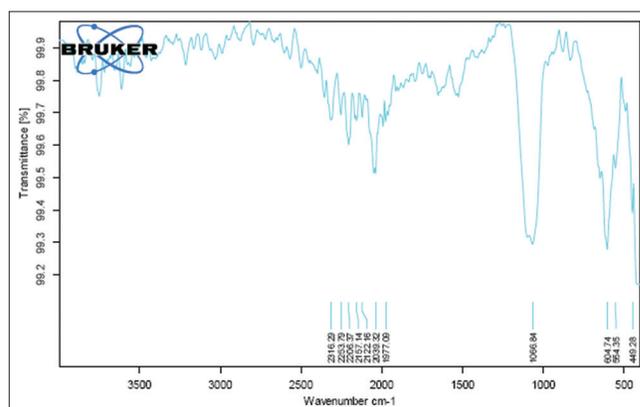


Figure 2: FT-IR spectrum of NiO nanoparticles (*Polianthes tuberosa*)

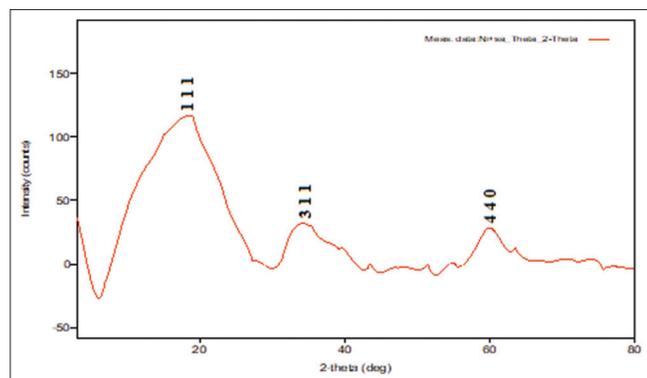


Figure 3: XRD Spectrum of NiO nanoparticles (*Polianthes tuberosa*)

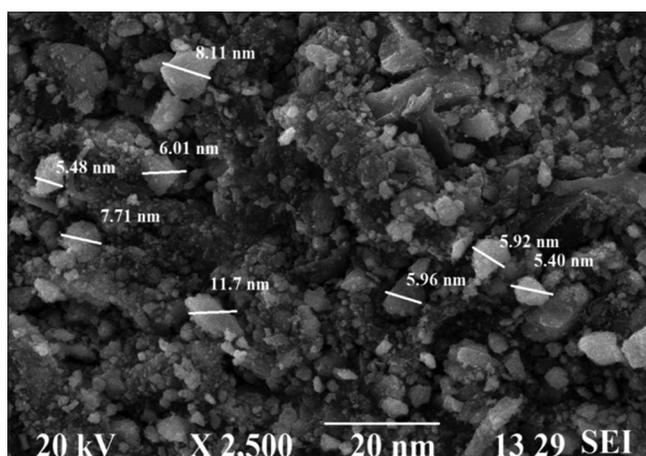


Figure 4: SEM image of NiO nanoparticles (*Polianthes tuberosa*)

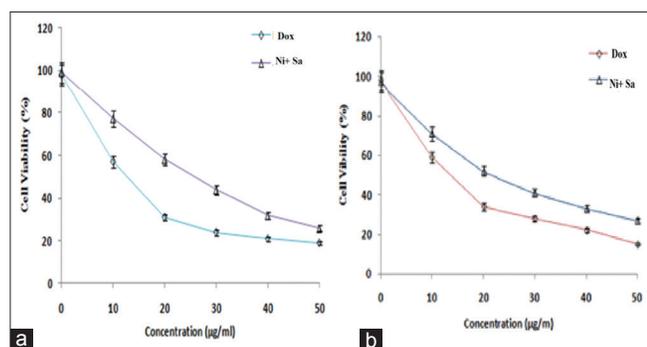


Figure 5: Anticancer activity of NiO nanoparticles a) MM2, b) HeLa

SEM of NiO nanoparticles prepared using *Polianthes tuberosa* displayed sphere and flake like aggregates as in Figure 4. The NiO nanoparticles have a well-defined distribution also. From this, the synthesized NiO nanoparticles has a particle size in between 5 and 11 nm range. The SEM data were obtained from imagej software and shown in Table 2.

Anticancer Studies

The effect of the nanoparticles to arrest the proliferation of cancer cells was investigated using MM2 and HeLa cancer cell lines. From the results, we observed that the nanoparticles showed good cytotoxicity activity with the tested cell lines.

Table 1: XRD Data of NiO nanoparticles prepared using *Polianthes tuberosa*

S.No.	2 θ (deg)	FWHM (deg) B	Θ in Radians	Cos Θ	D= $K\lambda/\beta\text{Cos}\Theta$ in nm	h	k	l
1	17	9.7	0.148352	0.989015	1.5	1	1	1
2	34.61	5.4	0.302029	0.954734	2.8	3	1	1
3	60.4	3.1	0.527089	0.864274	5.4	4	4	0
Average particle size in nm					3.23			

Table 2: SEM Data of NiO nanoparticles

S. No.	Label	Area	Mean	Min	Max	Angle	Length
1		0.849	146.348	18	189.889	-6.34	5.488
2		0.941	110.932	65	135.125	-18.886	6.085
3		1.263	138.701	95	186.87	-32.8	8.111
4		1.194	103.819	52.471	158.706	-19.502	7.715
5		1.194	103.819	52.471	158.706	-19.502	7.715
6		1.791	113.396	41.954	174.942	-17.266	11.741
7		0.918	71.891	45.832	106	-23.962	5.969
8		0.918	145.812	67.071	181.669	-32.471	5.926
9		0.849	153.415	105	179.272	-26.565	5.421
10	Mean	1.102	120.904	60.311	163.464	-21.922	7.13
11	SD	0.304	26.887	26.758	27.635	8.221	2.013
12	Min	0.849	71.891	18	106	-32.8	5.421
13	Max	1.791	153.415	105	189.889	-6.34	11.741

Cells were treated at the concentrations of 10, 20, 30, 40, and 50 $\mu\text{g/mL}$ for 24h and cytotoxicity was measured using MTT assays. MTT data have shown cytotoxicity effect in a dose-dependent fashion. In MM2 cells, cell viability was reduced to 79%, 58%, 39%, 33%, and 25% for the dosage of 10, 20, 30, 40, and 50 $\mu\text{g/mL}$, respectively. The results revealed that about 50% of the cell viability (IC_{50}) was found to be at $25 \pm 1.4 \mu\text{g/mL}$ concentration of NiO nanoparticles.

Similarly, in HeLa cells, cell viability was decreased to 70%, 51%, 40%, 38%, and 34% for the dosage of 10, 20, 30, 40, and 50 $\mu\text{g/mL}$, respectively. The results revealed that about 50% of the cell viability (IC_{50}) was reduced at $20 \pm 0.3 \mu\text{g/mL}$ concentration of NiO nanoparticles (Figure 5).

CONCLUSION

Using *Polianthes tuberosa* extract, NiO nanoparticles were successfully prepared. The UV-Visible spectrum of NiO nanoparticles using *tuberosa* extract showed a functional band at 269 nm. FT-IR spectroscopy exhibited a characteristic peak at 554 cm^{-1} responsible for NiO nanoparticles. SEM and XRD analysis gave the morphology and size of the nanoparticles. From XRD results, it was concluded that NiO nanoparticles are having face centered cubic and 3.23 nm sized crystals. SEM analysis gave the size of nickel oxide nanoparticles between 5 and 11 nm. NiO nanoparticles synthesized using *Polianthes tuberosa* showed significant activity against MM2 and HeLa cancer cell lines. IC_{50} values against MM2 and HeLa cell lines are $25 \pm 1.4 \mu\text{g/mL}$, $20 \pm 0.3 \mu\text{g/mL}$ respectively.

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