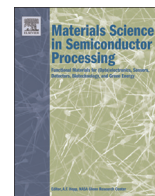




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Combustion synthesis and characterization of NiO nanoparticles

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ABSTRACT

Nickel oxide nanoparticles (NiO NPs) have been prepared by gel-combustion technique using 1:0.5 (N 105) and 1:1 (N 11) weight ratios of oxidizer, nickel nitrate hexahydrate and fuel, cassava starch, respectively. The X-ray diffraction pattern of the samples revealed cubic phase of nickel oxide and the average crystallite size of 28 and 38 nm for N 105 and N 11, respectively, were noted. In comparison to particle size measured using TEM (35 and 60 nm), the average particle size obtained from DLS (250 and 350 nm) was larger for N 105 and N 11, respectively. NiO NPs were found to have indirect band gap (2.98 and 2.3 eV) as revealed by optical spectroscopy. They were tested for energy storage, antimicrobial activity and photocatalytic applications. Electrochemical studies showed NiO NPs had a reversible capacity of 940 and 785 mA h/g for N 105 and N 11, respectively and they retained a capacity of 59% and 47% upto 50 cycles. The antimicrobial activity was tested against two bacterial strains and a fungal strain. The photocatalytic activity was measured for degradation of methylene blue in ultra-violet as well as sunlight. NiO, obtained by using 1:1 oxidizer to fuel ratio, (N 11) exhibited 94% degradation efficiency in sunlight because of a visible light-active band gap (2.3 eV).

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1. Introduction

In the past few decades, metal oxide nanoparticles have been gaining momentum amongst researchers. One of the more extensively used transition metal oxides, nickel oxide (NiO), has a wide band gap [1] and varied applications [2]. They include catalysis [3], lithium ion batteries [4,5], smart windows [6], anti-ferromagnetic film [7], dye-sensitized photocathodes [8,9], photocatalysis [10], anti-microbial activity [11], thermal conductivity [12] and field emission studies. [13]. Recent studies of nickel oxide nanoparticles (NiO NPs) prepared by a cost-effective and eco-friendly method have been used to evaluate its antimicrobial activity [14] and Manigandan et al. used NiO NPs for photocatalytic degradation of Rhodamine B [15]. Various research groups have synthesized NiO NPs by the hydrothermal [16] solvothermal process [1], chemical precipitation [17], emulsion [18], solid state chemical decomposition [19] thermal decomposition [20,21] sol-gel [22] and reverse micro-emulsion method [23]. NiO synthesized by microwave thermal treatment by Cho et al. has been used as an anode material in fuel cells [24]. However, most of these methods usually require special equipment, elaborate procedure or high processing temperatures. On the other hand, the gel-combustion synthesis is a unique and versatile method where metal salts

together with suitable organic complexing agent, here, cassava starch, are mixed, evaporated to get a homogeneous gel called oxidizer-precursor gel [25]. The organic matter is removed by ignition of the gel. The uniqueness and versatility of combustion synthesis are (1) it is an easy and fast process requiring less energy and time (2) the product is homogenous and crystalline with a high degree of purity [26–28]. In addition to the advantages of the method, the fuel (cassava starch) used in the present work, is natural, economical, abundant, easily available, the method of preparation does not require complicated procedures and most importantly, the oxidizer and fuel do not form a homogeneous solution that is expected to form in all solution combustion reaction. When cassava starch is heated above its gelatinization temperature (65 °C) [29] the hydrogen bonds present in the carbohydrates break followed by attachment of water molecules to the liberated hydroxyl group. This results in the swelling of the granules and these the starch granules act as nuclei and template for the growth of the NiO NPs [30]. In this work, cassava starch not only functions as fuel but also as a bio-template for nanoparticle growth. For combustion reaction to occur effectively, carbohydrate content of the fuel plays a major role. Cassava starch has been chosen as it has higher carbohydrate content compared to the other starchy counterparts like corn and potato [31]. Cassava (*Manihot esculenta*, botanically) is a major crop in the tropical region [32]. It is the primary source of carbohydrates in sub-Saharan Africa [33]. The tubers (storage root) are rich in starch [34] and are consumed by more than 500 million people of the world [35].

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Though it ranks sixth among the crops in the global production [36], cassava has received less attention by researchers giving it an 'orphan crop' status [37].

The present study reports the use of cassava starch as a fuel to synthesize NiO NPs by combustion synthesis and to the best of our knowledge, this is a novel fuel used for such synthesis to obtain high quality NiO NPs. The obtained NiO NPs is evaluated for its potency as energy storage in lithium batteries, in antimicrobial activity and as photocatalyst in degradation of organics.

2. Experimental section

2.1. Preparation of cassava starch

Cassava pearls (purchased from local market) were hand-picked to remove the solid impurities, washed in tap water to remove suspended impurities and then rinsed well in distilled water. To 500 g cassava pearls, a known volume of water was added, heated on a hot plate at 150 °C for almost an hour. When the pearls became glassy and paste-like, the contents were transferred, in small portions, on to a clean plastic sheet. The sheet, covered with a thin cloth to prevent dust contamination, was exposed to bright sunlight for a week till it dried to the point of being brittle. The crystals were powdered and stored in an airtight vessel to prevent infestation and was termed 'Cassava Starch'. This procedure was developed by our research group and hence our own procedure [38].

2.2. Synthesis of NiO NPs using cassava starch

Nickel(II) nitrate hexahydrate (Merck) and cassava starch were taken in two weight ratios viz. 1:0.5 (known as N 105) and 1:1 (known as N 11), respectively. Based on our earlier report, only two ratios were selected as they were the ones that provided better results [38]. They were mixed well with 30 mL water and heated on a hot plate at 200 °C till they formed a viscous gel. The gel was introduced into a pre-heated muffle furnace at 475 °C where the combustion reaction occurred. Combustion was initiated by smoldering and later the self-propagating wave spread throughout the reaction vessel bringing the reaction to completion in 10 min. To remove traces of the un-burnt fuel, the sample was calcined at 500 °C for 5 h in air.

2.3. Fabrication of batteries and coin cells for lithium battery studies

The electrochemical cell consisted of the prepared samples, NiO NPs as the positive electrode, Li metal as negative electrode and 1 M lithium hexafluorophosphate (LiPF₆) (in 1:1 (volume ratio) mixture of ethylene carbonate (Merck) (EC) and diethyl carbonate (Merck) (DEC)) as electrolyte. Celgard2502 membrane (methyl-hydrosiloxane–dimethylsiloxane copolymers supported on a polypropylene microporous flat sheet membrane) was used as a separator. The coin cells (CR2016) were assembled in an argon-filled dry glove-box. To fabricate the positive electrode, NiO NPs were thoroughly mixed and ground well in a mortar along with, commercially available conductive agent, C-super P Black. Slurry of the sample and C-super Black along with poly vinylidene fluoride (PVDF), binder (Kynar 2801) dissolved in N-methyl-2-pyrrolidone (NMP) was prepared in the weight ratio of 70: 15: 15, respectively and applied on Cu foil, current collector. Charge–discharge cycling and cyclic voltammetric studies were carried out at 25 °C using Bitrode multiple battery tester (model SCN, Bitrode, USA) and a computer controlled MacPile II (Biologic, France) unit, respectively.

2.4. Antimicrobial activity of NiO NPs

Antimicrobial activity of the NiO NPs was done by agar well diffusion method against two pathogenic bacterial strains *Bacillus cereus* (gram positive, MTCC 2155), *Klebsiella aerogenes* (gram negative, MTCC 109) and a fungal strain (*Candida albicans*, MTCC 227). Muller-Hinton agar (for bacteria) and Potato Dextrose agar, (for fungus) were poured into sterile petri plates in laminar air-flow and was left aside for solidification. Then the agar plates were swabbed with 100 µL each of overnight cultures of *B. cereus*, *K. aerogenes* and *C. albicans* using a sterile L-shaped glass rod. Using a sterile cork-borer, wells (6 mm) were created in each petri plate. Varied concentrations of NiO NPs (500 µg/150 µL and 750 µg/150 µL for bacteria and 100, 200, 300 and 400 µg/ 80 µL, respectively for fungus) were loaded onto the petri plates followed by incubation for 17 h at 37 °C, for bacteria and 3–5 days at room temperature for fungus. After the incubation period, the diameter of the zone of inhibition (DZI) was recorded. The experiments were carried out in triplicates for each concentration and average values reported.

2.5. Photocatalytic activity

Photo catalytic activity of NiO NPs were evaluated on the degradation of methylene blue (Merck, India) (MB) in an aqueous solution under UV light (125 W/m², Osram) and solar light (940 W/m², average value) illumination. Known weight of the catalyst, 0.01 g, was added to known volume of the dye, 0.2 L which resulted in a suspension. The suspension was stirred for uniform exposure of the catalyst to light. The distance between the lamp and base of the beaker under UV illumination was 13 cm. Each experiment (both under UV and solar light illumination) was conducted for 120 min with 10 mL sample aliquots withdrawn every 15 min. The degradation of the dye was monitored after the removal of photocatalyst (centrifugation at 4000 rpm for 30 min). Its decreased absorbance was measured at regular intervals of time (0 to 120 min) using Shimadzu UV 1650 PC spectrometer.

2.6. Characterization

The crystal characteristics of the NiO NPs were studied by Philips X'Pert PRO X-Ray diffractometer with Cu-K (α) (1.5418 Å) radiation. Photoluminescence (PL) measurement was done using Shimadzu spectrofluorimeter (model RF 5301 PC). Scanning electron microscope (SEM) (JEOL-JSM-6490 LV) (carried out by coating a thin layer of gold to avoid charging of the samples) and Transmission electron microscope (JEOL JEM 1200) operating at 120 kV (samples were dispersed in alcohol, a drop of the sample was coated on carbon coated Cu grid) were used to study the morphology. Particle size and zeta potential were measured by diffusion light scattering technique using DLS Malvern ZetaSizer, (ZS, UK). The NiO NPs were sonicated in double-distilled water before the measurement of hydrodynamic diameter. Raman spectra of the sample was recorded by JASCO 4100LE spectrometer and LabRAM HR, Horiba Jobin Yvon (France) using a 514.5 nm air-cooled Ar⁺ laser with 50× objective, respectively. The absorption spectrum of the sample was recorded by Shimadzu UV 750 UV–vis spectrophotometer.

3. Results and discussion

3.1. Structure, morphology and other physical properties

Rietveld refined X-ray diffraction patterns of NiO are shown in Fig. 1. The broad lines in the pattern signify smaller crystallite size.

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