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# A carbothermal reduction method for the preparation of nickel foam from nickel oxide and sucrose



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## ABSTRACT

A new method involving thermo-foaming of NiO powder dispersions in molten sucrose at **140 °C** followed by carbonization and carbothermal reduction at **1050 °C** has been used for the preparation of nickel foam. The molten sucrose acts as a medium for the dispersion of NiO powder for thermo-foaming as well as the source of carbon for the carbothermal reduction. The nickel foam produced from a composition consisting of 100 g sucrose and 150.5107 g NiO had purity as high as 99.5 wt%. The nickel foam had interconnected cellular structure with a porosity of nearly 93.8% and showed an electrical conductivity of  $3.97 \times 10^5$  S/m suitable for electrode applications.

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## 1. Introduction

Nickel foams are increasingly used as electrodes in advanced electrochemical systems such as Ni-H<sub>2</sub> and Ni-Cd batteries, fuel cells, supercapacitors and dye sensitized photovoltaic cells [1–3]. They are also used as catalyst and catalyst support [4]. They are generally prepared by electrodeposition or chemical vapour deposition (CVD) of nickel on polyurethane foam template and solid-gas eutectic transition (gasars) technique. In electrodeposition method, non-conducting polyurethane foam template is first made conducting by coating either with nickel metal by electroless deposition, sputtering and CVD techniques or with a conducting paste [5]. Subsequent electroplating of nickel followed by burnout of the polyurethane template and sintering under reducing atmosphere results in reticulated nickel foam. On the other hand, in the gasars technique, hydrogen gas dissolved in molten nickel at high pressure undergoes eutectic transformation to a heterogeneous two phase system consisting of solid metal and hydrogen gas on cooling. The precipitated gas molecules are trapped as pores within the metal body [6]. In the CVD technique, deposition of nickel metal on a polyurethane foam template is accomplished by decomposing nickel tetracarbonyl [Ni(CO)<sub>4</sub>] at temperatures in the range of 150–200 °C [7]. However, the nickel carbonyl used in the CVD technique is extremely toxic and explosive in air. In this

study, an easy method for the synthesis of nickel foam from nickel oxide (NiO) and sucrose has been reported. In this case, the sucrose is used as a medium for the preparation of NiO powder dispersion for thermo-foaming as well as source of carbon for the carbothermal reduction of NiO.

## 2. Experimental

Analytical reagent grade NiO powder procured from Merck India Ltd. Mumbai was calcined at **1000 °C** for 1 h in a muffle furnace. Intimate mixture of 200 g sucrose (Analytical reagent grade, Merck India Ltd, Mumbai) and 301.02 g NiO powder was prepared by ball milling them in an acetone medium using a planetary ball mill (Fritsch, Germany) followed by drying in an air oven at **70 °C**. The NiO powder dispersion in molten sucrose was prepared by heating the sucrose-NiO mixture in 2.5 L borosilicate glass tray at **185 °C** and stirring with a glass rod. The NiO powder dispersion in molten sucrose was heated in an air oven at **140 °C** for foaming and setting to form NiO-sucrose polymer composite foam. The NiO-sucrose polymer composite foam was cut into pieces of 8 cm × 6 cm × 4 cm size and heated in an ultrapure argon atmosphere in a tubular furnace at **1050 °C** for 6 h. The heating rate used was **2 °C/min**. The samples were unloaded from the furnace after cooling to room temperature. The process flow chart for the preparation of nickel foam is given in Fig. S1 (Supplementary information).

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Porosity of the nickel foam was calculated from the bulk density estimated from its weight and dimensions. The X-ray diffraction analysis was carried out using an XRD analyzer (X'pert Pro, Philips, USA) using copper K- $\alpha$  radiation. **Microstructural** analysis was carried out using a scanning electron microscope (SEM, FEI Quanta FEG200). Average cell and average grain size were calculated from the SEM images using ImageJ software. The purity of the nickel foam was gravimetrically determined by estimating the nickel as nickel-dimethylglyoxime complex using the procedure reported elsewhere [8]. The electrical conductivity of the nickel foam was measured using a four probe method.

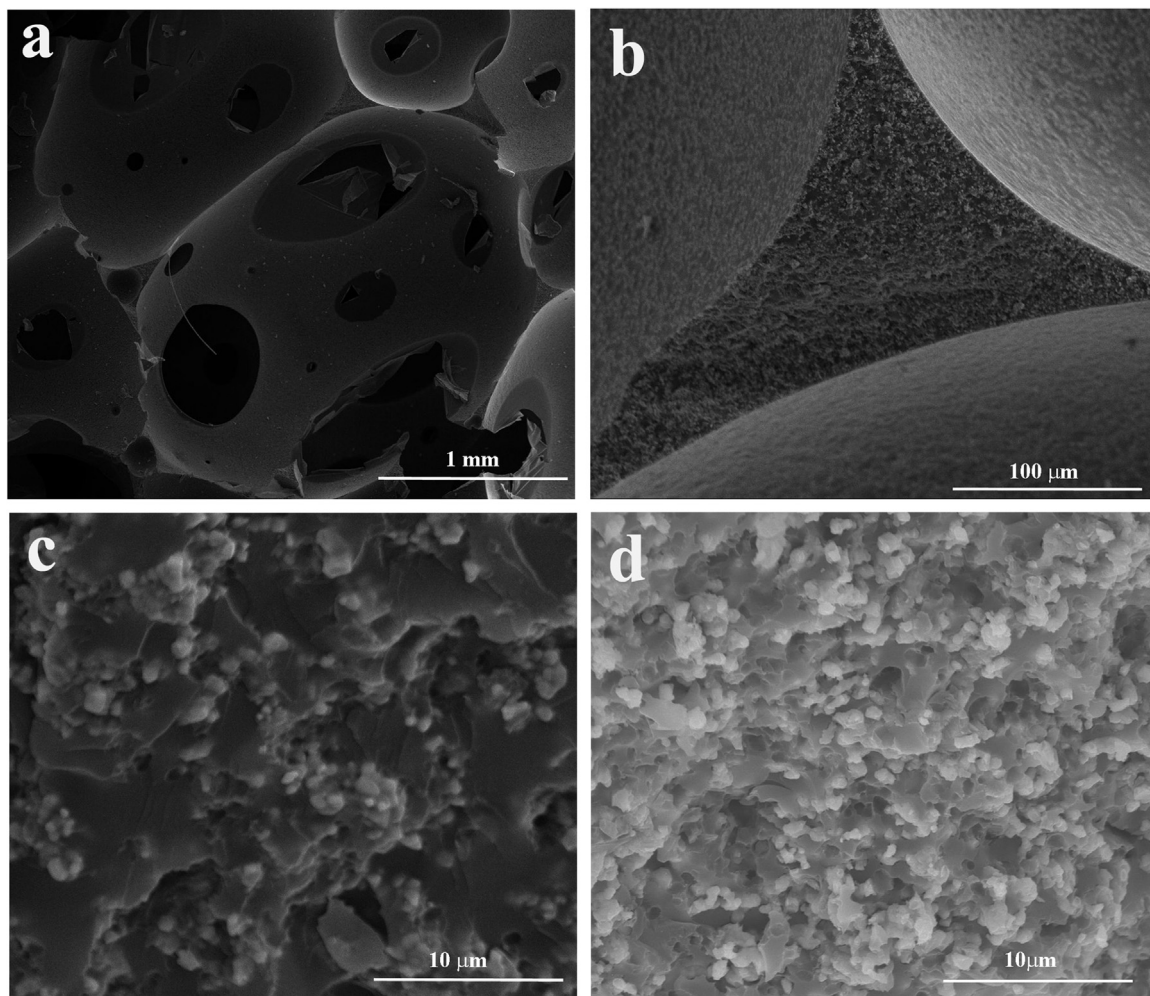
### 3. Results and discussion

The NiO disperses well in molten sucrose due to the interaction of sucrose hydroxyl groups with the hydrophilic NiO surface [9]. The NiO powder dispersion in molten sucrose undergoes slow foaming at **140 °C** due to the bubbles generated by water vapour produced because of condensation between sucrose hydroxyls [9,10]. The foam produced **had** volume nearly 6.1 times the volume of the NiO powder dispersion in molten sucrose. The continued polymerization and cross-linking of sucrose in the foamed dispersion **resulted** in its setting. The time taken for foaming and total time taken for foaming **and** setting **were** approximately 8 and 27 h, respectively. The SEM photomicrograph of **the** NiO-

sucrose polymer composite foam **showed** interconnected cellular structure. The NiO particles **were** uniformly distributed in the sucrose polymer matrix. The sucrose in the NiO-sucrose composite foam **underwent** carbonization when heated in an inert atmosphere at temperatures in the range of 150–600 °C to form NiO-carbon composite foam. Fig. 1 shows SEM photomicrograph of NiO-sucrose polymer and NiO-carbon composite foams.

The carbon present in the NiO-carbon composite foam reduces NiO to Ni and the nickel produced undergoes proper sintering during the heat treatment at **1050 °C** [11]. The reduction of NiO to Ni by the carbon is evidenced from the XRD analysis. Fig. 2 shows the XRD spectrum of NiO-sucrose polymer composite foam and nickel foam produced by the carbothermal reduction at **1050 °C**. All the peaks observed in the XRD spectrum of the NiO-sucrose polymer composite foam were corresponding to the NiO. No reflections corresponding to the sucrose polymer was observed in the XRD spectrum indicating its amorphous nature. On the other hand, the foam sample heat treated at **1050 °C** in an inert atmosphere **showed** all peaks corresponding to face centered cubic nickel metal and no peak corresponding to NiO, indicating complete reduction of NiO to nickel.

The NiO-sucrose polymer composite foam undergoes a total volume shrinkage of 78.3% during carbonization and subsequent carbothermal reduction. However, there is no crack or deformation observed in the foam body during the carbothermal reduction. Fig. 3 shows the photograph of NiO-sucrose polymer composite



**Fig. 1.** SEM photomicrographs showing (a) cellular structure of NiO-sucrose foam, (b) strut region of NiO-sucrose foam, (c) high magnification image of fractured strut of NiO-sucrose foam and (d) fractured strut region of NiO-carbon composite foam.

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