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STRUCTURAL AND MAGNETIC PROPERTIES OF NI NANOMATERIALS SYNTHESIZED BY HYDRAZINE REDUCTION METHOD IN PRESENCE OF SDS SURFACTANT

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ABSTRACT
Nickel nanomaterials have been synthesized from nickel chloride hexahydrate by a reduction method using hydrazine as the reducing agent in the presence of an anionic surfactant - sodium-dodecyl sulphate (SDS). The nanoparticles were characterized by X-ray diffraction and the angle 2θ distinctly reveal absorption of nickel crystal. The particles have shown magnetic properties as the magnetization is 0.17845 emu.

INTRODUCTION
Nanotechnology becomes a broad, multidisciplinary field covering biological, chemical, physical, technological, etc., subjects and the nanomaterials have been getting very large applications in almost all branches of science and in most of the industries. The nanomaterials have significant properties which are different from the same materials with large dimensions. Innumerable methods for synthesis of different nanomaterials have known in literature. According to Hussain and Haque [1], two approaches – top down and bottom up have been used for synthesis of these materials. In the top down approach, bulk material is broken down followed by stabilization while the bottom up approach is related to the wet chemical method for synthesis of the nanoparticles. The synthesized material has a number of morphologies and these morphological materials exhibit different properties which may not be similar to each other and thus find applications in diverse fields.

Nickel nanomaterials have large applications; a very good catalyst in chemical reactions. Different morphologies of nickel nano such as nano spheres, nano rod, nano belt, nano wire, hollow sphere, etc., have been known [2-5]. Cordente et al. [6] reported that the electric and magnetic properties of Ni nanoparticles depend on the morphology and shape of the particles. Various methods for the synthesis of Ni nano have been known in which reduction method is relatively common and simple. Hydrazine is a common reducing agent, but other reducing agents are reported in literature [7]. In the synthesis of Ni nanomaterials, generally sodium dodecyl sulphate (SDS) is used as a surfactant; however, other surfactant is also used [8]. In the present study, an attempt has been made to synthesize nickel nanomaterials and to study structural and magnetic property of the materials.

MATERIALS AND METHODS
Preparation of nickel nanoparticles
100 ml aqueous solution of 0.42M $NiCl_2.6H_2O$ was kept at 50°C. 2.0g sodium dodecyl sulphate (SDS), as surfactant, was added to the nickel salt solution. The solution was stirred continuously by electric stirrer at a speed of 1000 rpm. Then slowly NaOH solution was added, at 70^OC under stirring condition, till pH attained 11.0. Temperature was then increased to 80°C. 20.0 ml hydrazine hydrate was added to the stirred solution slowly. The mixture was kept for one hour under the same condition. As the reaction continued, heavy froth appeared and gray/black particles were formed. These particles were centrifuged out, washed with distilled water and finally with ethanol. The material;s were kept in a dessicator. Some Ni particles were also present in the forth and these were washed with acetone and distilled water before drying.

X-ray diffraction (XRD) study
The crystalline nature of the synthesized Nickel nanomaterials were verified by X-Ray Diffraction pattern. The XRD measurements of the particles was carried out using a Bruker AXS and the X-ray diffraction was determined with CuKα radiation with wavelength, λ= 1.54178A° at the Bragg angle (2 theta) ranging from 10 – 100° at a scan rate of 5^O min^{-1}.

Study of magnetic property
Magnetic properties of the nickel nanomaterials were studied in a Lakeshore 7410 vibrating sample magnetometer (VSM). The measurement was done by taking 0.02 g of solid sample on the tips of the vibrating rod and analyzed at room temperature.

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RESULTS AND DISCUSSION

In the synthesis of nickel nanoparticles, $NiCl_2$ was used as a precursor. On adding NaOH solution, the solution became basic and the pH was kept at 11.0. Hydrazine is a normal reducing agent and in basic medium, under study, it reduces Ni^{2+} to Ni and itself oxidized to N_2. The reaction is shown below.

$$2Ni^{2+} + N_2H_4 + 4OH^- = 2Ni + N_2 \uparrow + 4H_2O$$

As soon as nickel particles were formed at a temperature 70^OC, two important things happened. Firstly, thick froth appeared on the surface of the reaction solution taken in a container and secondly, a huge volume expansion of the reacting solution was seen. The volume expansion was due to the presence of sodium dodecyl sulphate as surfactant and the gradual formation of nitrogen gas. The importance of using SDS is that it reduces the interfacial tension between the newborn particles by adsorbing the surfactant at the liquid-metal interface so formed [8]. Special care was taken for selecting the bigger size reaction vessel, otherwise the reacting solution could be lost due to volume expansion. However, grey/black coloured nickel particles were deposited at the bottom of the container. The crystalline nature of the Ni nanoparticles were examined by XRD.

The X-ray diffraction pattern for Ni nanomaterials is presented in Figure1. Distinct peak in the XRD pattern has revealed fine crystallinity of the nanomaterials. Our approach clearly demonstrates that the reduction method with hydrazine in presence of SDS can synthesize fine nickel nanoparticles. The 2θ and d-values and their intensities are given in Table 1. It appears that highest intensity is found in the angle 2θ at 43.34^O followed by 37.30^O and 51.86^O; these are mostly characteristic absorption of nickel crystal.

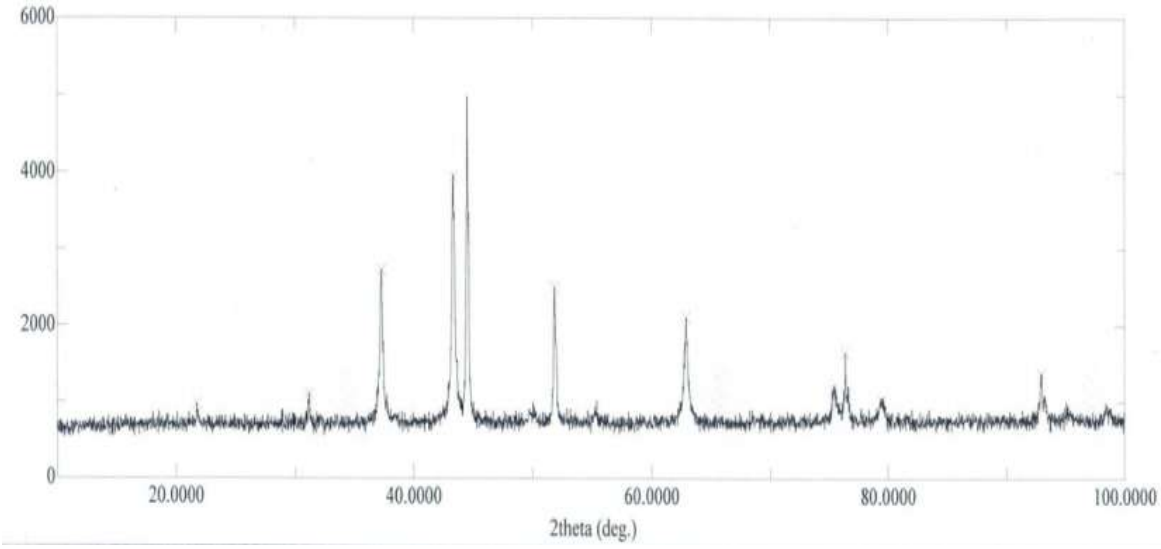

Figure 1. XRD pattern of nickel nanomaterials.

Table 1. Magnitude of 2θ, d- values and intensities of Ni nanomaterials obtained from XRD pattern.

Sl. No.	2θ	d-value	Intensity	I/Io
1	21.720	4.0883	975	25
2	31.180	2.8661	1113	29
3	37.300	2.4087	2733	70
4	43.340	2.0860	3942	100
5	51.860	1.7616	2504	64
6	62.920	1.4759	2117	54
7	75.360	1.2602	1167	30
8	76.380	1.2459	1650	42
9	79.480	1.2049	1017	26
10	92.960	1.0623	1369	35

The magnetic behaviour of the nickel nanoparticles was studied by VSM. The materials show magnetic behaviour by showing low magnitization, i.e., 0.17845 emu. The magnetic field versus magnetization curve at room temperature is recorded with the field strength of -15 kOe to 15 kOe; the hysteresis loop is shown in Figure 2. From these measurements, the saturation magnetization (Ms), remanence magnetization (Mr) and coercivity (Hc) are derived. The figure reveals the formation of single-phase material.with low magnetic property. The saturation magnetization for the Ni nanoparticles was reported to be 60 emu/g [9] and 57.39 emu/g [10]. These values for Ni exhibited a saturation magnetization greater than that of the bulk nickel (55 emu/g) [11]. Although **bulk** Ni shows ferromagnetic behaviour, but various morphologies of Ni nanoparticles, exhibit superparamagnetism [12, 13].

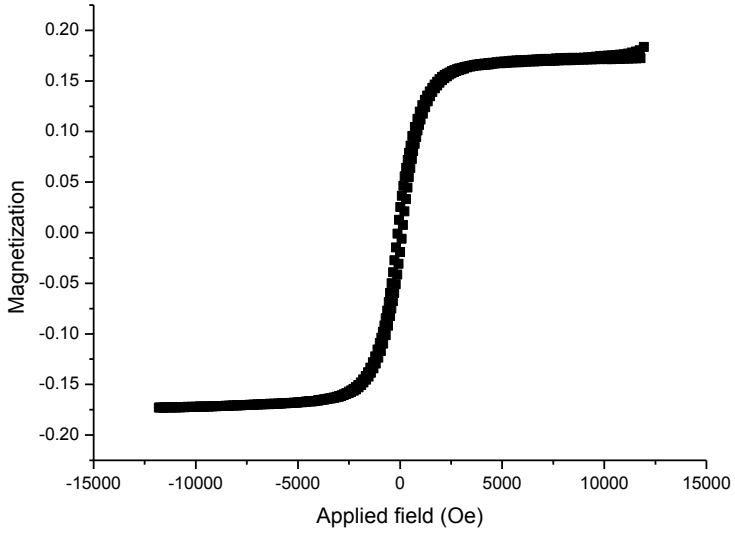

Figure 2. Magnetic hysteresis curve measured at a room temperature for Ni nanoparticles.

CONCLUSIONS

In conclusion, we have synthesised Ni nanoparticle with fine crystallinity by using hydrazine as a reducing agent and SDS as an anionic surfactant. The synthesized nanoparticles were characterized by X-ray diffraction. Moreover, the magnetic behaviour of the nickel nanoparticles was studied by VSM and found to display low magnetization.

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