

## Research Article

# Effect of Crystallinity of $\beta$ - and $\beta_{bc}$ -Nickel Hydroxide Samples on Chemical Cycling

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$\beta$ -phases of nickel hydroxide and cobalt hydroxide samples crystallize in cadmium iodide type structure.  $\beta$ -cobalt hydroxide on oxidation generates  $\beta$ -CoOOH which crystallized in  $3R_1$  polytype while the structure of  $\beta$ -phase of NiOOH polytype is less well understood.  $\beta$ - and  $\beta_{bc}$ -phases of nickel hydroxide samples were prepared by using ammonium hydroxide and sodium hydroxide as precipitating agents. Powder X-ray diffraction data shows that  $\beta$ -phase of nickel hydroxide is perfectly crystalline in nature while  $\beta_{bc}$ -phase of nickel hydroxide is poorly ordered.  $\beta$ - and  $\beta_{bc}$ -phases of nickel hydroxide samples were subjected to chemical oxidation using sodium hypochlorite. The oxidized phases of  $\beta$ - and  $\beta_{bc}$ -phases of nickel oxyhydroxide are highly disordered and the broadening of reflections in the powder X-ray diffraction patterns is due to the presence of structural disorder, variations in the crystallite size, and strain. On reduction of  $\beta$ - and  $\beta_{bc}$ -phases of nickel oxyhydroxide, the powder X-ray diffraction patterns visually match the powder X-ray diffraction data of the pristine phases of  $\beta$ - and  $\beta_{bc}$ -phases of nickel hydroxide indicating that the  $\beta$ -phase of nickel hydroxide does not transform to  $\beta_{bc}$ -phase of nickel hydroxide, but the particle sizes are significantly affected.

## 1. Introduction

Nickel hydroxide is used as a cathode material in all nickel based alkaline cells [1]. Nickel hydroxide crystallized in two phases, that is,  $\alpha$ - and  $\beta$ -phases [2]. In addition to  $\alpha$ - and  $\beta$ -phases, poorly ordered  $\beta$ -phase of nickel hydroxide designated as  $\beta_{bc}$ -nickel hydroxide has also been reported [3]. Several articles have been published in area of structure and electrochemistry of nickel hydroxide [4–6]. During the charge-discharge process, redox reaction occurs inducing structural changes in the positive and negative electrode materials. The electrochemical performances of  $\beta$ -nickel hydroxide are lower than that of  $\beta_{bc}$ -nickel hydroxide [7], even though there are several reports on the structural changes which occur during the electrochemical cycling of  $\beta$ -nickel hydroxide [8–11]. It is difficult to separate the oxidized and reduced phases of the active materials used in the battery or electrochemical cell; thus, attracting different methods adopted towards characterization provides information in the local environment [10, 12, 13]. Therefore, the redox process induces structural transformations which was monitored

using powder X-ray powder diffraction patterns. Bode et al. had proposed the two different mechanisms which occur during the cycling of  $\alpha$ - and  $\beta$ -phases of nickel hydroxide [14]. Two redox couples exist, that is,  $\alpha(\text{II})/\gamma(\text{III})$  and  $\beta(\text{II})/\beta(\text{III})$  couple.  $\beta(\text{II})/\beta(\text{III})$  couple induces less structural changes; that is, the interplanar spacing of  $\beta(\text{II})$  phase of nickel hydroxide is 4.6 Å and  $\beta(\text{III})$ -NiOOH is 4.8 Å, respectively, while  $\alpha$ -phase of nickel hydroxide ( $d = 7.6$  Å) to  $\gamma(\text{III})$  phase of NiOOH ( $d = 8$  Å) induces strain during the electrochemical cycling. Barnard et al. reported that the structural changes during cycling of  $\beta(\text{II})/\beta(\text{III})$  redox couple is marginal as it is difficult to differentiate it clearly from powder X-ray diffraction data [15]. Casas-Cabanas et al. and Deabate et al. have also extensively studied the structural changes which occur during the chemical and electrochemical cycling of nickel hydroxide [16, 17]. Also there are reports of using ozone as oxidizing agent to investigate the structural transformations during the electrochemical cycling of nickel hydroxide focused on  $\alpha(\text{II})/\gamma(\text{III})$  and  $\beta(\text{II})/\beta(\text{III})$  couple [18, 19]. To the best of our knowledge, there are no studies which report on the variation in the particle size, lattice strain,

and the nature of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide phases dried materials during the chemical cycling of two polymorphic modifications, that is,  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide. In this work, we have chemically cycled  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples separately using sodium hypochlorite as an oxidizing agent to understand the structural changes induced during the process using powder X-ray diffraction technique.

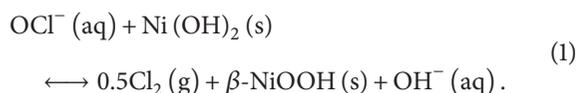
## 2. Experimental

**2.1. Synthesis of  $\beta$ - and  $\beta_{bc}$ -Nickel Hydroxide.**  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples were prepared by following the procedures described elsewhere [20, 21].  $\beta$ -nickel hydroxide was prepared by the addition of 100 mL of nickel nitrate solution ( $1 \text{ mol L}^{-1}$ ) to 100 mL of ammonium hydroxide ( $2 \text{ mol L}^{-1}$ ) solution in aqueous medium at  $65^\circ\text{C}$  under constant stirring.

$\beta_{bc}$ -nickel hydroxide was obtained by adding 50 mL of nickel nitrate ( $1 \text{ mol L}^{-1}$ ) solution to 100 mL of sodium hydroxide solution ( $2 \text{ mol L}^{-1}$ ) in aqueous medium at  $65^\circ\text{C}$  under constant stirring.

The green precipitates of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide obtained above on precipitation were aged in their respective solutions in which precipitation was carried out (aqueous solution) at  $65^\circ\text{C}$ . The slurries were filtered, washed with distilled water, and dried at  $65^\circ\text{C}$ .

**2.2. Chemical Oxidation of  $\beta$ - and  $\beta_{bc}$ -Nickel Hydroxide Samples.**  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples were chemically oxidized by the addition of 100 mg of samples (separately) in 100 mL of sodium hypochlorite ( $2 \text{ mol L}^{-1}$ ) and aged overnight. Chlorine gas bubble evolution was observed during the process and the reaction is



Green coloured solid precursors of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples turned to black in colour due to oxidation and formation of  $\beta$ -form of nickel oxyhydroxide after 2 h but the oxidation was incomplete due to the solid solution interface process. Hence,  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples were aged for 18 h, 36 h, and 48 h separately in NaClO solution. The solids were filtered, washed with distilled water, and dried at room temperature. We have shown the data of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples aged for 18 h in NaClO solution.

**2.3. Chemical Reduction of Oxidized Phases of  $\beta$ - and  $\beta_{bc}$ -Nickel Hydroxides.** The oxidized products were reduced by the addition of 30 v/v% of hydrogen peroxide. The black coloured solid turned to green and the products were filtered, washed with distilled water, and dried at room temperature.

**2.4. Characterization.** The samples were characterized using Bruker D8 advanced powder diffractometer with Cu K $\alpha$  source ( $\lambda = 1.5418 \text{ \AA}$ ) and scan rate of  $2 \text{ degrees min}^{-1}$  at  $0.02^\circ$  steps.

PXRD patterns were simulated using DIFFaX code. The details about the PXRD pattern simulations of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide have been reported elsewhere [21].  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples and chemically oxidized phases of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples (i.e.,  $\beta$ -NiOOH and  $\beta_{bc}$ -NiOOH) of different polytypes, that is, 1H, 2H<sub>1</sub>, 2H<sub>2</sub>, 2H<sub>3</sub>, 3R<sub>1</sub>, and 3R<sub>2</sub>, were generated by using different stacking vectors. The simulated diffraction patterns were examined and compared with the experimental powder X-ray diffraction data.

## 3. Results and Discussion

Figure 1 shows the PXRD patterns of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples. XRD patterns of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide show prominent reflections and the  $2\theta$  values match that of ideal  $\beta$ -nickel hydroxide.  $\beta$ -nickel hydroxide exhibits sharp peaks indicating that the sample is less disordered and has lower quantities of planar defects, when nickel hydroxide is precipitated using ammonium hydroxide as a precipitating agent [22]. When nickel hydroxide is precipitated using sodium hydroxide, poorly crystalline form of nickel hydroxide, namely,  $\beta_{bc}$ -nickel hydroxide, was obtained. The powder diffraction pattern of  $\beta_{bc}$ -nickel hydroxide exhibits nonuniform broadening of non-(hk0) reflections due to the presence of structural disorder (interstratification, stacking faults, and cation vacancies) inside the lattice [23]. The lattice parameters of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples are  $a = 3.13$  and  $c = 4.61 \text{ \AA}$  and  $a = 3.13$  and  $a = 3.124$  and  $c = 4.67 \text{ \AA}$ , respectively. Beside numerous studies on the oxidation of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide using sodium hypochlorite/ozone we have revisited the oxidation process and examined the structural changes that have been investigated.

Figure 2 shows the PXRD patterns of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples on oxidation using sodium hypochlorite as an oxidizing agent. The oxidized phases of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide, that is,  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide samples, exhibit broad peaks in their powder X-ray diffraction patterns.

The diffraction patterns of the charged phases of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide and  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide cannot be indexed due to the disappearance of peaks. It should be emphasized that small shifts in peak positions can be detected easily when comparing the diffraction patterns of  $\beta$ -NiOOH and  $\beta_{bc}$ -NiOOH instead of comparing unit cell parameters derived from several peaks. The broadening of the diffraction lines on oxidation indicates that there will be a decrease in the crystallite size and also the disorder along the  $c$ -crystallographic direction in the redox process of  $\beta(\text{II})/\beta(\text{III})$  couple. Therefore, the actual structure of the oxidized form of  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide is well understood and is also not determined by powder X-ray diffraction studies. Earlier we had reported that the perfectly crystalline form of  $\beta$ -nickel hydroxide possesses 1–3% of planar defects while poorly ordered phase of  $\beta_{bc}$ -nickel hydroxide comprises >20% of  $\alpha$ -motifs, 15–20% stacking faults, and 10–17% cation vacancies [23]. This promotes us to

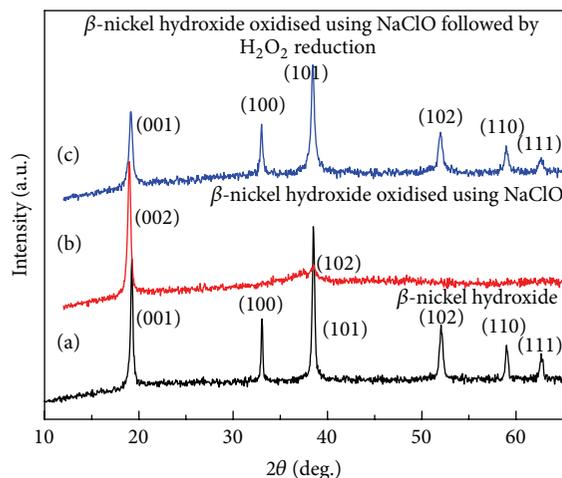


FIGURE 1: Powder XRD patterns of (a)  $\beta$ -nickel hydroxide, (b) oxidation of (a) using sodium hypochlorite at 25–30°C, and (c) reduction of product of (b) using hydrogen peroxide at 25–30°C.

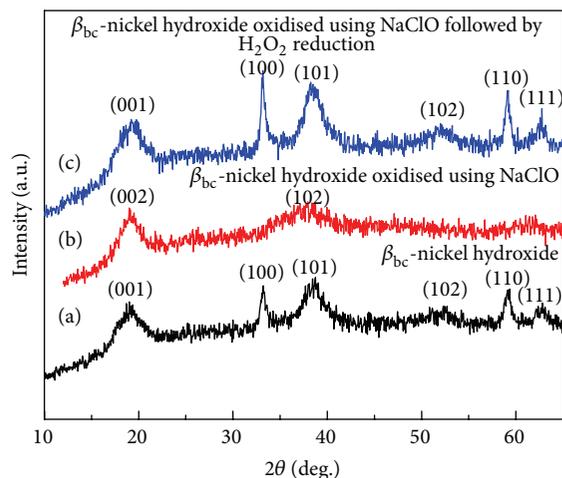


FIGURE 2: Powder XRD patterns of (a)  $\beta_{bc}$ -nickel hydroxide, (b) oxidation of (a) using sodium hypochlorite at 25–30°C, and (c) reduction of product of (b) using hydrogen peroxide at 25–30°C.

examine the oxidation-reduction process in  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide and validate the variations in the structural disorders during the chemical cycling process. One of the advantages of using sodium hypochlorite is that the rate of oxidation of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide phases can be finely controlled. On oxidation of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples using NaClO, (001) peak positions of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples at 19.24° and 18.99° ( $2\theta$ ) were shifted to 18.98 and 19.06° ( $2\theta$ ), respectively. Small shift in the (001) reflections corresponding to the  $c$ -cell parameter was observed. This indicates that there is close structural similarity/relation between the pristine material ( $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide) and chemically oxidized species ( $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide) and also topotactic relation between the phases of (100) $\beta$ (III)/(100) $\beta$ (II) and (110) $\beta$ (III)/(110) $\beta$ (II) also exists. The pristine material ( $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide) and chemically oxidized ( $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide) structures differ in an increased disorder in the  $ab$  plane.

The decrease in the line intensities of chemically oxidized species of  $\beta$ -nickel oxyhydroxide/ $\beta_{bc}$ -nickel oxyhydroxide samples could be due to (i) the formation of structural disorder (loss of ordering along  $c$ -crystallographic direction), (ii) the changes in the crystallite size, (iii) induction of strain due to the repulsion between the sheets due to the formation of higher valent nickel ion in the nickel oxyhydroxide sheets, and (iv) random or systematic rotations/translations of layers. The small shifts in the peak positions of the first reflections of  $\beta$ -nickel hydroxide/ $\beta_{bc}$ -nickel hydroxide samples with their chemically oxidized products indicate that interconversion of the discharged and charged phases occurs in solid state mode and the process is a simple proton deintercalation/diffusion process.

Figure 3 shows the PXRD patterns of chemically reduced phases of  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide using hydrogen peroxide.

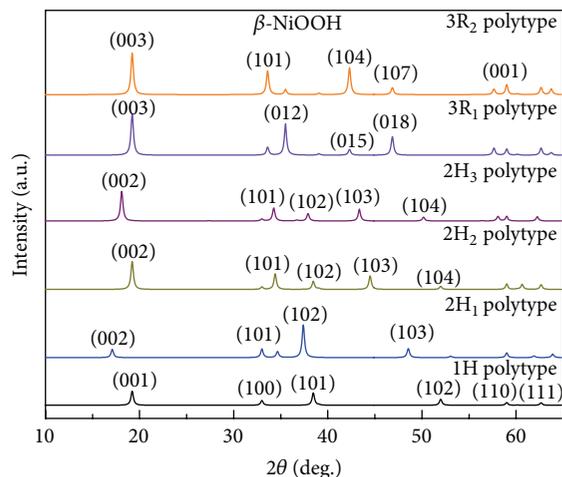


FIGURE 3: DIFFaX simulated powder XRD patterns of (a) pure 1H, (b) 2H<sub>1</sub>, (c) 2H<sub>2</sub>, (d) 2H<sub>3</sub>, (e) 3R<sub>1</sub>, and (f) 3R<sub>2</sub> polytypes of  $\beta$ -phase of nickel oxyhydroxide separately.

On reduction of  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel oxyhydroxide, we get the diffractions patterns which are similar to that of parent phases of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide indicating that the samples have no practical differences in their powder X-ray diffraction patterns. On careful examination, we observe variations in the peak widths of chemically reduced phases (see Table 1). The phase changes during the charge-discharge process could be due to a charge diffusion or topotactic mechanism.

Crystallite sizes of all the peaks of pristine, chemically oxidized, and reduced phases of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide were calculated using the Scherrer formula. Corrections for instrumental effects on peak broadening were made by using the full width at half maxima (fwhm) of the silicon peaks. Results after calculations of the crystallite sizes and lattice strain in  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel

TABLE 1: Full width at half maxima (fwhm) of as-prepared, oxidized, and reduced phases of  $\beta$ -nickel hydroxide.

hkl	$\beta$ -nickel hydroxide			$\beta_{bc}$ -nickel hydroxide		
	As prepared	oxidised phase using NaClO	oxidised phase using NaClO followed by reduction using $H_2O_2$	As prepared	oxidised phase using NaClO	oxidised phase using NaClO followed by reduction using $H_2O_2$
001	0.275	0.364	0.423	3.521	2.54	4.382
100	0.183	—	0.275	0.799	—	0.542
101	0.279	0.932	0.407	2.534	6.495	2.385
102	0.391	—	0.502	3.784	—	3.083
110	0.296	—	0.440	0.902	4.40	0.5606
111	0.437	—	0.601	1.744	—	1.347

TABLE 2: Crystallite sizes of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide (as prepared),  $\beta$ -nickel oxyhydroxide, and  $\beta_{bc}$ -nickel oxyhydroxide (oxidized phases) and chemically reduced phases of and reduced phases of  $\beta$ -nickel oxyhydroxide and  $\beta_{bc}$ -nickel hydroxide samples.

hkl	$\beta$ -nickel hydroxide			$\beta_{bc}$ -nickel hydroxide		
	As prepared	oxidised phase using NaClO	oxidised phase using NaClO followed by reduction using $H_2O_2$	As prepared	oxidised phase using NaClO	oxidised phase using NaClO followed by reduction using $H_2O_2$
001	602.2	364.7	286.1	19.03	37.3	23.8
100	1975	—	619.2	207.04	—	126.1
101	610.7	106.5	316.7	37.63	13.4	35.2
102	365.8	—	250.7	30.2	—	24.33
110	774.8	—	348.8	239.5	—	239.4
111	359.4	—	220.1	80.1	—	79.68

hydroxide samples are shown in Tables 2 and 3. From Tables 2 and 3, it was found that the  $\beta$ -nickel hydroxide has larger crystallite size with an average diameter and thickness of 602 and 774 Å, respectively, while  $\beta_{bc}$ -nickel hydroxide sample has 19 Å (thickness) and 239 Å (disc diameter), respectively. Table 3 shows the strain and particle size of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples during chemical cycling process.

Compared to crystallite values obtained for  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples, the crystallite sizes of the chemically oxidized phases followed by reduced phases are smaller in size (see Table 2). The (110) peaks in  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples are broadened on oxidation and are comparable to  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples (precursors).

In our previous work, we have classified that nickel hydroxide can have different polytypic modifications, that is, 1H, 2H<sub>1</sub>, 2H<sub>2</sub>, 2H<sub>3</sub>, 3R<sub>1</sub>, and 3R<sub>2</sub> modifications. In the above, there will be variation in the stacking sequence of each nickel hydroxide sheet. Casas Cabanas has demonstrated using high resolution transmission electron microscopic data that 1H polytype of  $\beta$ -nickel hydroxide (with stacking sequence of AC AC AC - - -) transforms to 2H<sub>3</sub> polytype of  $\beta$ -nickel oxyhydroxide (AC BA AC - - - stacking sequence) [16]. If the above stacking sequence is correct then we should expect slight increase in the interplanar distance of  $\beta$ -nickel hydroxide on

chemical oxidation. Several articles have reported increase in the  $d$ -values on chemical oxidation of  $\beta$ -nickel hydroxide. To verify the scheme and identify the theoretical powder X-ray diffraction patterns of oxidized phases of different polytypes, we have simulated the powder X-ray diffraction patterns of 1H, 2H<sub>1</sub>, 2H<sub>2</sub>, 2H<sub>3</sub>, 3R<sub>1</sub>, and 3R<sub>2</sub> polytypic modifications of nickel oxyhydroxide. Figure 3 shows the simulated PXRD patterns of 1H, 2H<sub>1</sub>, 2H<sub>2</sub>, 2H<sub>3</sub>, 3R<sub>1</sub>, and 3R<sub>2</sub> polytypic modifications of nickel oxyhydroxide obtained on oxidation of  $\beta$ -nickel hydroxide. 2H<sub>3</sub> polytype will have AC BA AC - - - stacking sequence in which we observe octahedral and prismatic stacking in the interlayer region thus leading to slight increase in the  $d$ -spacing. There are also other reports which do not show significant changes in the  $d$ -spacings of  $\beta$ -nickel hydroxide and  $\beta$ -nickel oxyhydroxide. If we observe 2H<sub>2</sub> type of polytypic modification (AC AB AC AB - - -) for  $\beta$ -nickel oxyhydroxide, then the interlamellar stacking will have octahedral interactions only resulting in negligible change in the  $d$ -spacing of nickel oxyhydroxide. To examine it, in Figure 4 the simulated PXRD patterns of 1H polytype and 2H<sub>2</sub> polytype are shown and compared with the observed PXRD patterns. We could not easily identify the polytypes due to the poorly ordered peaks in the powder diffraction patterns of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples. Also it is difficult to conclude which

TABLE 3: Estimation of lattice strain of as-prepared, oxidized, and reduced phases of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples.

hkl	$\beta$ -nickel hydroxide Lattice strain			$\beta_{bc}$ -nickel hydroxide Lattice strain		
	As prepared $\beta (\text{Cos}\theta/\lambda) \times 10^{-3}$	$\beta$ -nickel hydroxide oxidised phase $(\text{Sin}\theta/\lambda)$	$\beta$ -nickel hydroxide oxidised phase reduction using $\text{H}_2\text{O}_2$ $\beta (\text{Cos}\theta/\lambda) \times 10^{-3}$	As prepared $\beta (\text{Cos}\theta/\lambda)$	$\beta_{bc}$ -nickel hydroxide oxidised phase using NaClO $\beta (\text{Cos}\theta/\lambda) \times 10^{-3}$	$\beta_{bc}$ -nickel hydroxide oxidised phase using NaClO followed by reduction using $\text{H}_2\text{O}_2$ $\beta (\text{Cos}\theta/\lambda)$
001	1.49	0.1083	3.145	0.03769	0.0261	0.04727
100	4.55	0.1844	1.45	$7.13 \times 10^{-3}$	0.0678	$4.346 \times 10^{-3}$
101	1.47	0.2139	2.841	0.0255	—	0.0239
102	2.46	0.2841	3.589	0.0369	—	0.0298
110	1.161	0.3194	2.57	$7.122 \times 10^{-3}$	0.04109	$3.762 \times 10^{-3}$
111	2.51	0.3374	4.08	0.01512	—	0.32
				0.337	0.3299	0.3369

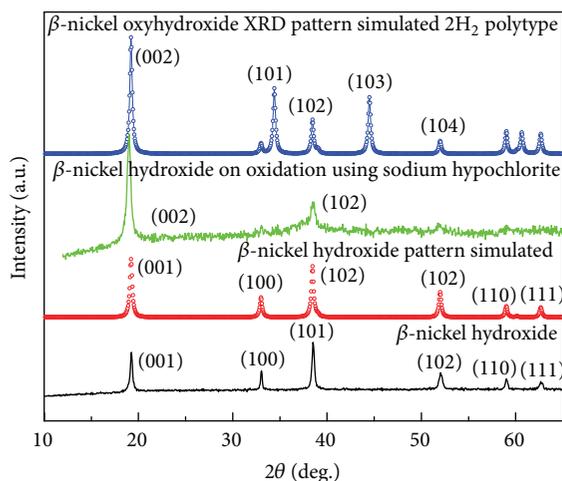


FIGURE 4: Powder X-ray diffraction patterns of (a) observed  $\beta$ -nickel hydroxide, (b)  $\beta$ -nickel oxyhydroxide obtained on oxidation of  $\beta$ -nickel hydroxide using sodium hypochlorite, (c) DIFFaX simulated PXRD patterns of 1H polytype of  $\beta$ -phase of nickel oxyhydroxide, and (d) DIFFaX simulated PXRD patterns of  $2H_2$  polytype of  $\beta$ -phase of nickel oxyhydroxide.

polytype is dominant but from peak positions we can predict that, during oxidation,  $2H_2$  polytype might have formed by comparing the peak positions at higher angles in the powder X-ray diffraction patterns.

Even though nickel hydroxide and cobalt hydroxide are isostructural, their oxidation-reduction mechanism is altogether different. On the basis of peak positions, we had reported that  $\beta$ -cobalt hydroxide on oxidation generates  $\beta$ -CoOOH which crystallized in  $3R_1$  polytype while  $\beta$ -phase of NiOOH crystallizes in the  $2H_2$  polytype [24]. The change in the unit cell volume during the chemical oxidation-reduction process of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples has been calculated. There are no significant changes in the cell volume during the oxidation and reduction process, thus favoring the charge-discharge process in nickel based alkaline cells.

One of the major highlights of this study is that the crystallographic changes induced during chemical cycling process have been evaluated in detail and the information on strain and the particle size has been reported during the chemical cycling of  $\beta$ -nickel hydroxide and  $\beta_{bc}$ -nickel hydroxide samples.

$\beta$ -nickel hydroxide does not undergo transformation to  $\beta_{bc}$ -nickel hydroxide and vice versa during chemical oxidation-reduction process, but significant changes are observed in the crystallite sizes of the samples.

#### 4. Conclusion

The relative intensities and the peak positions in the powder X-ray diffraction data can provide an insight into the nature of stacking of layers in  $\beta$ -nickel hydroxide samples. Based on the simulated powder X-ray diffraction patterns, we have attempted to determine the nature of polytype which could have formed in case of nickel oxyhydroxide. The oxidation in

$\beta$ -phases of nickel hydroxide occurs from 1H to  $2H_2$  polytypic transformation and on reduction  $2H_2$  polytype changes to 1H. The structural disorders do not lead to interconversion among  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide but particle sizes are significantly affected during chemical cycling of  $\beta$ - and  $\beta_{bc}$ -nickel hydroxide samples.

#### Conflict of Interests

The author declares that there is no conflict of interests regarding the publication of this paper.

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