

## Solidification/Fixation of Nickel Ions in Ordinary Portland Cement

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## Abstract

In this research article we represented the results of the studies of addition of varying concentrations of Nickel ions on the physical, chemical and engineering properties of 43 grade ordinary Portland cement. The studies regarding different physical & chemical parameters of OPC like initial and final setting time of cement, bulk density, and compressive strength, microscopic and spectroscopic properties have been carried out and the result of these studies are presented and discussed in the manuscript. Different building products containing varying concentrations of added nickel ions were prepared and subjected to hydrologic environment to investigate the leaching behavior of nickel ions. The efforts have also been made to establish a quantitative co-relation between the concentration of added Nickel ions and the intensity of any effect in above properties of the cement also the possible fixation mechanism of Nickel ions in cement was suggested. Scanning electron microscopy and X- Ray diffraction study of nickel containing cement samples show that nickel have no greater effect on the cement properties. Ni does not show any effect on the hydration of cement & its main compounds but it slightly retards the setting process. Some amount of Nickel is found to distributed throughout to the C-S-H gel phase Nickel get adsorbed the interstitial phases of the cement and formed the insoluble nickel compound. The C-S-H phase of Nickel containing samples found denser than that of control samples.

**Keywords:** Cement; Heavy metals; Nickel; Setting time; Waste management

## Introduction

Industrialization is vital to a nation's socio- economic development as well as its political stature in the international committee of nations. Industries vary according to process technology, sizes, nature of products, characteristics and complexity of wastes discharged. Ideally citing of industries should strike a balance between socio-economic and environmental considerations. Rapid industrial development and the world global growth have led to the recognition and increasing understanding of interrelationship between pollution, public health and environment. While almost industrial activities cause some pollution and produce waste, relatively few industries (without pollution control and waste treatment facilities) are responsible for the bulk of the pollution. It has been reported that industrial effluent has a hazard effect on water quality, habitat quality, and complex effects on flowing waters. Industrial wastes and emission contain toxic and hazardous substances, most of which are detrimental to human health. This type of waste is generated from metallurgical, mining, chemical, leather industry, distillery, sugar, battery, electroplating and pigment industry These include heavy metals such as nickel lead, cadmium and mercury, and toxic organic chemicals such as pesticides, PCBs, dioxins, polyaromatic hydrocarbons (PAHs), petrochemical and phenolic compound. Generally the metal concentration in the waste is too low for economic recovery but high enough to represent toxicity hazards. Therefore, these industries discharge their waste without treatment or with improper treatment in the landfills. Its disposal in hydrologic environment can cause environmental risk due to the mobility of metal ions. These metal ions get mixed in surface water and ground water system, which may be detrimental to human being as well to the environment [1-10]. Various technologies have been developed which purport to render a waste or to reduce the potential for the release of toxic species into the environment. This technique is used to transform potentially hazardous liquid or solid waste into less hazardous and or non-hazardous solid before disposal in a landfill, thus preventing the waste from contaminating the environment. The United State Environmental Protection Agency also recognizes cementitious Solidification/Stabilization as "the Best

Demonstrated Available Technology for land disposal of most toxic elements. Many formulations have been developed for the S/S process according to the kinds of wastes. Portland cement can be modified for suitable S/S process using flyash, lime slag soluble silicates etc. One of the most difficult problems in this process is that the hydration of cementitious materials is too retarded to set and harden enough due to the inhibition of hydration reaction of heavy metals in a landfill area. It seems that Solidification/Stabilization (S/S) process would be the best practical technology to treat the nickel containing waste [11-31]. There is very less data reported in the literature about the effects of addition of nickel ions on various properties of cement. Therefore, efforts have been made to fill void in this data. The results of these studies on the effects of addition of nickel on various properties of ordinary Portland cement are presented and discussed in this manuscript. A quantitative co relation between the concentration of nickel added and the intensity of any effect has been established.

## Materials, Apparatus and Methods

## Materials

To make the standard solutions of different concentrations of nickel metal ions for preparing cement samples, Nickel Nitrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) was used from Thomas Baker (Chemicals) Ltd. The metal salt was used as obtained without any further purification. Double distilled water was used to prepare the metal ion solutions throughout the study. Commercial ordinary Portland cement 43 grade was used.

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Constituent	Weight percentage
Silica	20.8
Aluminum oxide	4.4
Iron oxide	3.79
Calcium oxide	66.1
Magnesium oxide	3.3
Anhyd. Sulfuric acid	3
Sodium oxide	0.2
Potassium oxide	0.7

Table 1: Chemical composition of cement.

Physical parameter	Results
Loss on ignition	0.70%
Consistency	31.58%
Soundness	1.0 mm
Bulk density	1.421g/cm <sup>3</sup>
Initial setting time	175 min
Final setting time	300 min

Table 2: Physical properties of cement.

## Apparatus

Atomic Absorption Spectrophotometer (AAS) from Hitachi (Z-7000) was used to determine the metal ion concentrations. Hazardous waste filtration system from Millipore (YT-30142 HW) was used to carry out Toxicity Characteristic Leaching Procedure as recommended by United State Environment Protection Agency (USEPA); UV-Visible spectrophotometer (Aquamate) from Thermo Corporation was used for spectrophotometric studies. Vicat apparatus was used to determine initial and final setting time of control cement as well as metal ion doped cement samples. Compressive strength testing machine from Central Scientific Instruments Company was used to determine the compressive strength of mortar samples. Scanning Electron Microscope (SEM) from LEO 438VP, UK was used for the microstructure visualization of fractured cement surfaces. X-Ray powder diffraction analysis was carried out by using the instrument from Rikagu, Japan to identify the crystalline phases present in the control as well as in metal containing cement samples.

## Methods

The chemical analysis of the cement was carried out according to Indian standard specifications IS: 4032:1985 guidelines. The physical properties were tested according to the Indian standard specification IS: 4031:1996 guidelines. The chemical composition and physical properties of Ordinary Portland Cement used are summarized in Tables 1 and 2 respectively.

**Preparation of cement pastes and mortars:** The Initial and final setting times of blank as well as the cement samples, containing varying concentrations of nickel ions, were determined according to IS: 4031:1996. The results obtained for controls were compared with those obtained for nickel containing samples to know the effect of addition of nickel on the setting time of cement paste. To investigate the effect of addition of nickel on engineering properties like compressive strength, bulk density of mortar samples containing cement and flyash, were cast in 2.78” cubic iron molds. The samples were demolded after 24 hours and were dipped into water for curing the compressive strength of the cubes were determined on 3, 7, 28, 60, 90, 180 and 360 day of curing. Six samples were taken each time and the average value of these results was reported (shown in peak Table 3).

**X-ray diffraction analysis:** It is possible to find and quantify crystalline components which have over 1% abundance in a sample

if the correct experimental technique is used. XRD can give a semi quantitative or quantitative analysis of the components of the crystalline fraction. Thus XRD gives most information from the crystalline components of the material under investigation. X-ray diffraction analysis was performed with a Rikagu, Japan (Dmax 2200 VK/PC) automated X-ray diffractometer. The samples were crushed to a fine powder with porcelain and sieved through a 45 µm sieve. The XRD scan was made with copper ka radiation from 3°-70° 2θ with 0.02° step width and 1 to 3 s counting time.

**Scanning Electron Microscopy:** Scanning Electron Microscopy (SEM) is a technique used to know the fractured surface of cementitious material and three dimensional particle level morphology. It is generally used to magnify an image. It can magnify image up to three lack times. There are Three types of rays emitted from the instruments that is back scattered electron rays, secondary rays. Both are used for imaging. And X-rays are used for elemental analysis of the sample. It is used for

2-Theta	d(A)	BG	Height	I%	Area	I%	FWHM
9.134	9.674	227	103	14.8	1419	12.6	0.234
15.759	5.619	174	113	16.2	992	8.8	0.149
18.04	4.913	169	359	51.4	5376	47.9	0.255
18.938	4.682	163	89	12.8	1030	9.2	0.197
22.902	3.88	157	149	21.3	2448	21.8	0.279
26.271	3.39	180	62	8.9	1248	11.1	0.342
26.679	3.339	189	152	21.8	1493	13.3	0.167
29.4	3.036	208	563	80.7	10613	94.6	0.32
30.024	2.974	228	69	9.9	643	5.7	0.158
31.015	2.881	220	88	12.6	1025	9.1	0.198
32.14	2.783	235	474	67.9	11220	100	0.402
32.539	2.749	244	698	100	11108	99	0.271
34.082	2.628	217	517	74.1	10563	94.1	0.347
35.057	2.558	186	64	9.2	1050	9.4	0.279
36.676	2.448	177	83	11.9	648	5.8	0.133
37.256	2.412	168	77	11	1080	9.6	0.238
38.665	2.327	166	56	8	512	4.6	0.155
39.417	2.284	156	105	15	2603	23.2	0.421
40.88	2.206	173	101	14.5	1538	13.7	0.259
41.222	2.188	158	243	34.8	6064	54	0.424
41.578	2.17	161	85	12.2	2479	22.1	0.496
43.183	2.093	166	55	7.9	741	6.6	0.229
44.098	2.052	162	64	9.2	1562	13.9	0.415
44.622	2.029	158	62	8.9	1926	17.2	0.528
45.758	1.981	154	100	14.3	1711	15.2	0.291
47.159	1.926	158	225	32.2	5996	53.4	0.453
49.885	1.827	172	49	7	770	6.9	0.267
50.318	1.812	161	53	7.6	2172	19.4	0.697
50.801	1.796	169	97	13.9	1248	11.1	0.219
51.732	1.766	148	142	20.3	1517	13.5	0.182
54.358	1.686	148	75	10.7	519	4.6	0.118
55.265	1.661	140	57	8.2	631	5.6	0.188
56.401	1.63	142	98	14	1504	13.4	0.261
57.264	1.608	149	44	6.3	271	2.4	0.105
59.984	1.541	146	49	7	903	8	0.313
60.589	1.527	140	50	7.2	1072	9.6	0.364
62.239	1.49	132	134	19.2	2921	26	0.371

Table 3: Peak Search Report (37 Peaks, Max P/N = 11.4) [Sam.7\_1.raw] Sam. Nickel1000ppm  
PEAK: 21-pts/Parabolic Filter, Threshold=3.0, Cutoff=0.1%, BG=3/1.0, Peak-Top=Summit

S. No.	Concentration of metal ion (ppm)	IST	FST
1	10	175	300
2	50	175	300
3	100	175	300
4	500	180	300
5	1000	190	310
6	2000	190	310
7	5000	205	330
8	7000	205	335
9	8000	205	335
10	10000	205	335

IST- Initial setting time of cement in min  
FST- Final setting time of cement in min

Table 4: Effect of fixation of nickel ions on initial and final setting time of cement.

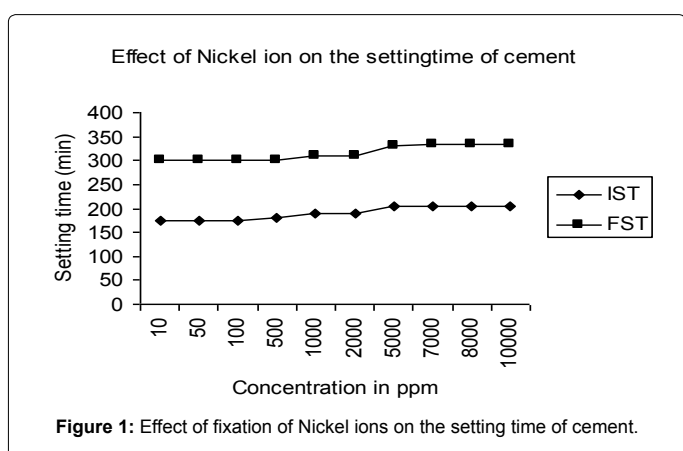


Figure 1: Effect of fixation of Nickel ions on the setting time of cement.

Binder system	No. of days	Average bulk density ( $\pm 0.001 \text{ g/cm}^3$ ) nickel		
		Control	500 ppm	1000 ppm
Cement + fly ash	1	2.303	2.303	2.303
	28	2.381	2.359	2.388

Table 5: Average bulk density values of solidified products on 1 and 28 days of curing.

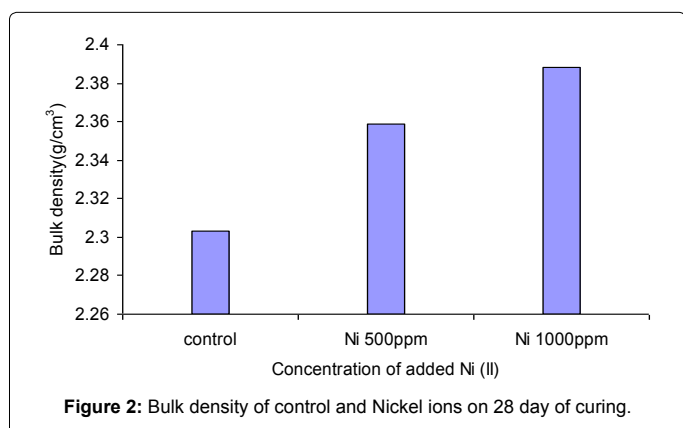


Figure 2: Bulk density of control and Nickel ions on 28 day of curing.

conducting material for non-conducting and biological material like cement coat a conducting material on it. It may be carbon or gold.

**Leaching studies:** A suitable way to know the effectiveness of the immobilization of contaminants after solidification process is to perform the extraction test. Usually this test is performed under the chosen condition of leaching medium. Standard method No.1311

recommended by United State Environment Protection Agency is followed. Crushed solid material has been taken in hazardous waste filtration system with zero head space extractor. A measured volume of water was added and this assembly has been shaken for 18 hours continuously in agitation assembly. Filtered extract was collected in a closed vessel and analyzed for metal concentration by using Atomic Absorption Spectrophotometer. The results were compared with that of blank samples to know the actual enhancement of the particular metal in the leachate. It was observed that the leachate of the samples doped with 500 ppm and 1000 ppm nickel containing 3.0 ppb and 5.0 ppb nickel in the TCLP leachate in water medium while in acid medium the leaching is somehow high as 0.25 ppm and 0.65 ppm of nickel is leached out in acid medium. It was found that Nickel mainly incorporated in solid solutions combined with Calcium oxide (CaO) only a little amount of nickel was found in di calcium silicate ( $\text{C}_2\text{S}$ ) and tri calcium silicate ( $\text{C}_3\text{S}$ ). Nickel has only marginal effects on the formation and hydration of its main compounds. Thus the complete nickel added to the cement mortar is get adsorbed through the matrix and very low leaching is due to the precipitation of nickel.

## Results and Discussion

Although hydration of cement begins immediately as water is added into it but there is a period of time-several hours in which cement remains in fluid like state before setting as a rigid, load bearing material. Setting refers the change of state from fluid paste to rigid form. According to IS: 8112:1996. The initial setting time of a 43 grade ordinary Portland cement should be at least 30 min. and final setting time should be within 600 min. The initial and final setting time of control as well as nickel containing samples were determined and presented in Table 4. Nickel was added in the concentrations 10, 50, 100, 500, 1000, 2000, 5000, 7000, 8000 and 10000 ppm respectively the addition of Ni (II) ions upto a concentration of 500 ppm caused no significant effect either on initial setting time or final setting time of cement. Both these parameters increased significantly when the concentration range of Ni (II) ions was increased from 500-7000 ppm as the initial setting time of cement paste bearing 7000 ppm Ni (II) ions was increased to 205 min. and final setting time was increased to 335 min. in comparison with blank sample which shows initial setting time and final setting time as 175 and 300 min. respectively. The retarding effect of Ni (II) ions gets constant when the concentration of Ni (II) is increased. Based on these results it may be stated that Ni (II) ions work as retarder in the concentration range 500-7000 ppm. All the experiments were carried out in triplicate to assure accuracy and reproducibility. All the samples were observed for any visual change before the compressive strength testing each time. There was no significant change found in colour and shape except the surface of nickel containing samples were smoother than that of control samples (Figure 1).

Bulk density of control samples as well as nickel ions containing samples was determined on 28 day of curing. Six samples were taken each time and the average value of these observations was reported. There was no significant change in this parameter for nickel ions containing sample in comparison with control (Table 5). Bulk density of control samples was  $2.381 \pm 0.001 \text{ g/cm}^3$ , while it changed to 2.359, and  $2.388 \pm 0.001 \text{ g/cm}^3$  for 500 and 1000 ppm, nickel containing samples respectively at 28<sup>th</sup> day of curing (Figure 2). The figure shows that bulk density values of nickel containing samples increased with the increase of concentration of nickel although the change is in significant. Compressive strength of control mortar samples as well as samples containing nickel ions was determined on 3, 7, 28, 60, 90, 180 and 360 days of curing. The results of this study are presented in Table 6. It is

Added Nickel ions (ppm)	No. of days							% change in compressive strength in comparison of control						
	3	7	28	60	90	180	360	3	7	28	60	90	180	360
Control	323.4	436	486	570	570	620	620							
500	305	426	445	580	580	600.6	600	94.3	97.7	91.5	101.7	101.7	97.8	96.7
1000	320	425	426	590	600	632	660	98.9	97.5	87.6	103.5	105.2	101.9	106.4

Table 6: Compressive strength (kg/cm<sup>2</sup>) of mortar samples.

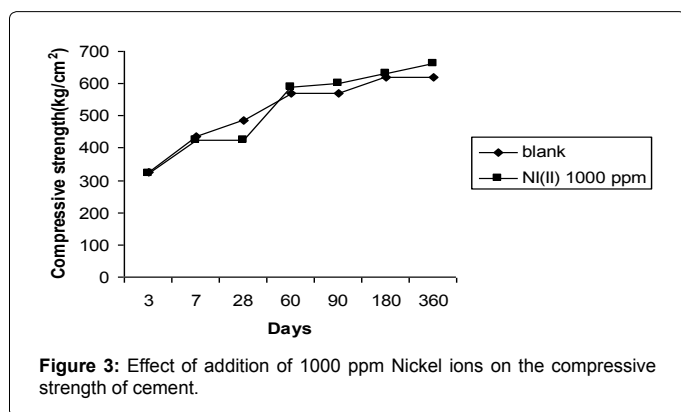


Figure 3: Effect of addition of 1000 ppm Nickel ions on the compressive strength of cement.

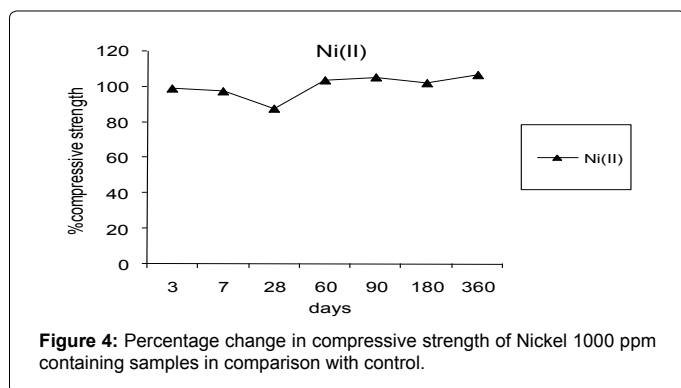


Figure 4: Percentage change in compressive strength of Nickel 1000 ppm containing samples in comparison with control.

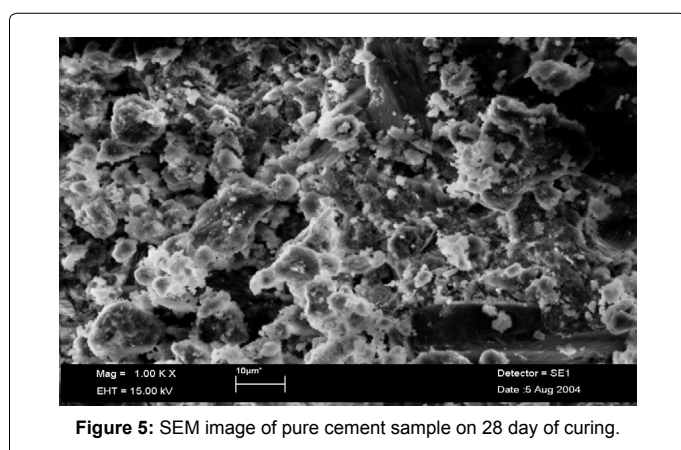


Figure 5: SEM image of pure cement sample on 28 day of curing.

important to mention that according to IS:1882:1989 the compressive strength of 43 grade ordinary Portland cement should be at least 230 kg/cm<sup>2</sup>, 330 kg/cm<sup>2</sup> and 430 kg/cm<sup>2</sup> for 3, 7 and 28 days of curing. The addition of nickel ions containing 500 ppm and 1000 ppm of metal ion also caused a decrease in compressive strength in early hydration period as a compressive strength of 3 day is only 305kg/cm<sup>2</sup> and 320 kg/cm<sup>2</sup> for 500 and 1000 ppm and it is only 94.3 and 98.9 percent of the control

sample this decrease is intensified while CS is 445.0 and 426.0 kg/cm<sup>2</sup> on 28 day of curing and the percentage change is 91.4 and 87.5. Utmost care was taken to reduce variability associated with batch preparation step and reagent addition to avoid any substantial variability within a batch. A perusal of data presented in Table 6, exhibits that compressive strength of nickel containing samples decreases significantly in comparison with control sample (Figures 3 and 4).

### Microscopic and spectroscopic analysis of the cement paste

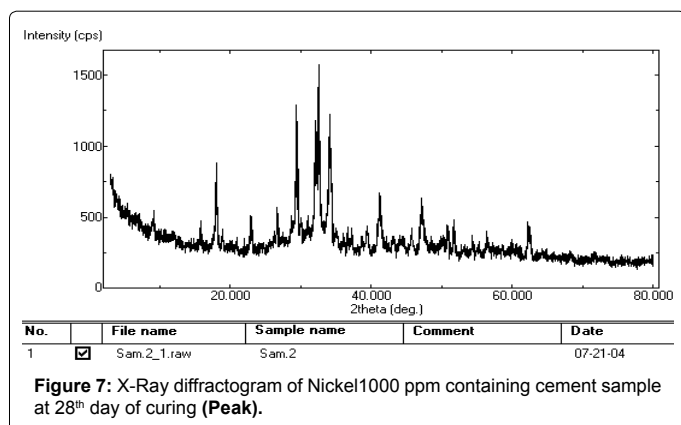
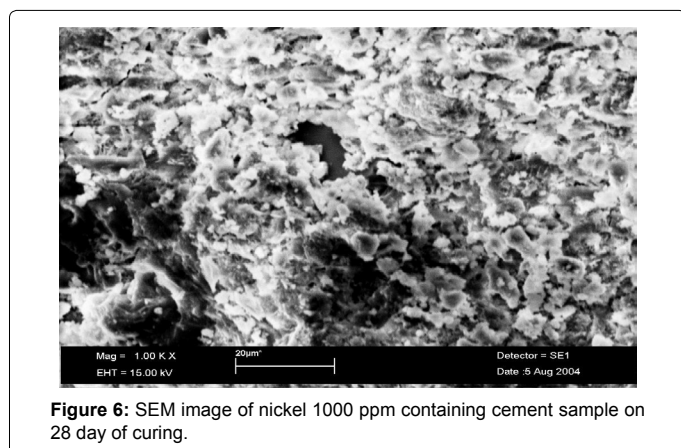
The microstructures of hardened control cement paste as well as nickel ions containing cement paste were investigated by Scanning Electron Microscopy (Figures 5 and 6). Scanning electron microscopic analysis was carried out to know the change in the morphology of hardened cement paste on 28 and 180 days of curing.

The graphs obtained by the XRD analysis of the blank as well as the samples containing the varying concentration of heavy metal ions are presented. The qualitative XRD studies correlate well with the findings from the SEM analysis. It is often claimed that stabilization of metal involves the formation of insoluble metal silicate but the SEM and XRD examinations did not reveal any identifiable crystalline zinc silicate, though amorphous gel of calcium silicate was observed in both pure chemfix and metal-bearing chemfixed solids. Scanning electron microscopy and X- Ray diffraction study of nickel containing cement samples (Figures 6 and 7) show that nickel has no greater effect on the cement properties. Nickel does not show any effect on the hydration of cement & its main compounds but it slightly retards the setting process. Some amount of Nickel is found to distributed throughout to the C-S-H gel phase Nickel get adsorbed the interstitial phases of the cement and formed the insoluble nickel compound. The C-S-H phase of Nickel containing samples found denser than that of control samples.

### Conclusions

The effect of fixation of nickel on physical, chemical and engineering properties of 43-grade Ordinary Portland Cement was studied. A quantitative correlation between the concentrations of nickel used and the intensity of any change in the properties of 43 grade Ordinary Portland Cement like initial and final setting time, compressive strength, bulk density, leaching studies, microscopic and spectroscopic studies etc. has been established. Nickel was used in the concentration of 500 and 1000 ppm. It may be stated that Ni (II) ions work as retarder in the concentration range 500-7000 ppm. The retarding effect of Ni (II) ions gets constant when the concentration of Ni (II) is increased further.

The addition of nickel ions containing 500 ppm and 1000 ppm of metal ion also caused a decrease in compressive strength in early hydration period. This decrease is intensified while CS is 445.0 and 426.0 kg/cm<sup>2</sup> on 28 day of curing and the percentage change is 91.4 and 87.5. Scanning electron microscopy and X- Ray diffraction study of nickel containing cement samples show that nickel have no greater effect on the cement properties but Some amount of nickel is found to distributed throughout to the C-S-H gel phase, nickel get adsorbed



the interstitial phases of the cement and formed the insoluble nickel compound. The C-S-H phase of nickel containing samples found denser than that of control samples.

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