Effect of nickel containing mixing water on the properties blended cement mortar

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Abstract. Influence of Nickel(Ni) present in the water which is used for mixing of blended cement was experimentally evaluated. The properties investigated were setting times, soundness, compressive strength, and durability aspects like acid attack, alkaline attack and sulphate attack. Specimens were prepared by using the de-ionized as well as Nickel(Ni) spiked de-ionized water for reference and the test samples respectively. The concentrations of Nickel(Ni) tested for different samples are 10, 50, 100, 500, 1000, 2000, 3000, 4000 and 5000mg/L. It is observed that on comparison with reference specimens, the setting process of cement of test specimens has increased with increase of Nickel proportion in de-ionized water. Increase of initial setting time was insignificant up to 100 mg/L concentration whereas the same for final setting time was significant for all concentrations. Change in expansion of cement mortar is insignificant up to 3000 mg/L concentrations of the Nickel(Ni). The change in strength was noticed to be insignificant at all the concentrations of Nickel(Ni). However, there is an increase in strength with higher Nickel proportion up to 2000 mg/L. This proportion of 2000 mg/L was considered as the reference mix for conducting durability tests. The strength as well as weight loss for the specimen mortar cubes placed in acidic medium are severe compared to that of alkaline and sulphate solutions with increase in age.

Keywords: Nickel(Ni), De-ionized Water, Blended cement mortar, Micro Silica, setting times, compressive strength, soundness, durability.

1. Introduction
Continuous increases in population and Industrialization are generating lot of liquid and solid waste causing many environmental problems over the Globe. Waste water generated from industries is being diverted directly into rivers and streams without proper treatment in most of the cases. Construction industry is one of the largest consumers of water. Generally, potable water is suitable as mixing water for cement concrete. Due to pollution of water bodies, availability of potable water is becoming scarce for the construction industry there by forcing it to use alternate sources of water. In this regard, industrial waste water plays an important role. Reuse and recycling of industrial waste water may be practiced to attain sustainable development of construction industry.

Industrial treated waste water containing heavy metals like Copper, Zinc, Manganese, Cadmium, and Iron was successfully used in cement mortar (Reddy Babu G., Sudarsana Rao H. and Reddy Ramana I.V.,2009) ¹. Heavy metals like Pb, Hg, Ni, Fe and Cr were found useful for cement mortar up to 600mg/L(Reddy Babu G., Sudarsana Rao H, and Reddy Ramana I.V.,2007) ². Using of wastewater from industries for casting of concrete did not show any detrimental effect over the behavior of concrete. Concrete with improved initial compressive strength could be made with
reclaimed wastewater used partially or totally for the mixing water (Tay J.H. and Yip W.K., 1987) 4. Quantity as well as quality of mixing water in cement concrete and mortar have significant impact over the properties of cement mortar as well as cement concrete (Neville, Adam 2000) 5. Heavy metals such as Copper, Zinc, Lead etc., present in mixing water cause a retardation of the early hydration and strength development of cement mortar (Tashiro C.,1980) 6. It was suggested the term HPC for concrete mixtures that possess the following three properties: high strength, high workability, and high durability (Aitcin,2000) 7. It was found that the strengths of both cement mortar and cement paste increases when 15% of the cement is replaced by silica fume (Darwin, et al) 8. Silica Fume added to concrete increases water demands, often requiring one additional pound of water for every pound of added Silica Fume. This is due to high surface area of silica fume. This problem can be easily overcome by using super plasticizer or HRWR (Per Fidjestol et al 2012) 9. Organically polluted water has shown indistinct results on cement mortar (Reddy Ramana I.V, Reddy Babu G, and Chiranjeevi P,2006) 10. It was investigated the effect of acid attack on concrete with fly ash and micro silica. 150 mm size concrete cubes which were cured for 28 days and immersed in water with 1 % of sulphuric acid for 45 day were tested and it was reported that the loss in weight of control concrete was 2.5% where as the concrete with 15% fly ash and 7.5% micro silica showed only 1.09% loss in weight (Natesan,2003) 11. At low concentrations, hydrochloric acid and nitric acid caused higher deterioration in terms of both strength and weight loss compared to sulfuric acid during the testing period(Turkel S et al , 2007) 12. Though reclaimed industrial wastewater are reported to be used in cement mortar, still very small information is available on the permissible limit of heavy metals they may be present in mixing water made with metal spiked de-ionized water exposed to acid attack, alkaline attack and sulphate attack. Therefore, an attempt was made to estimate the influence of Nickel (Ni) exist in mix water over setting times, soundness, cube strength, acid attack, alkaline attack, sulphate attack and chloride ion penetration on high performance cement mortar.

2. Materials and Methods
The materials utilized in this experimental work are:
1. Ordinary Portland Cement(53 Grade)
2. Fine aggregate (Ennore sand – grade I, grade II and grade III)
3. Micro Silica
4. Water (De-ionized)
5. Super plasticizer
6. Heavy Metal (Ni)
7. Chemicals

2.1 Cement
Ordinary Portland Cement (53 grade) was used for this experimental study. Preliminary tests such as fineness, specific gravity, setting times, soundness and compressive strength were conducted. All properties of the cement tested are under the limits of IS 12269:1987 and are given in table 1 and table 2.

| Parameter                  | Value    |
|----------------------------|----------|
| Specific gravity           | 3.15     |
| Finess                     | 325 m²/kg|
| Initial setting time       | 150 minutes|
| Final setting time         | 360 minutes|
| Compressive strength       | N/mm²    |
| 3 day                      | 39       |
| 7 day                      | 48       |
| 28 day                     | 55       |
| Expansion                  | 1.20mm   |
Table 2. Chemical Parameters of OPC

| Oxide                | Proportion |
|----------------------|------------|
| Calcium oxide        | 65.19      |
| Silicon dioxide      | 21.53      |
| Alumina              | 5.27       |
| Iron oxide           | 4.36       |
| Magnesium Oxide      | 1.10       |
| Alkalies(Na₂O,K₂O)   | 0.002      |
| Silicate             | 1.5        |

2.2 Fine Aggregate
The fine aggregate used throughout this investigation was obtained from Ennore, Tamil Nadu minerals limited, Chennai. It is approved by Bureau of Indian Standards (BIS) to manufacture and supply of Indian Standard sand conforming to IS 650:1991. The physical and chemical properties of the sand are presented in table 3 & table 4:

Table 3. Properties of Ennore sand

| Parameter                        | Result          |
|----------------------------------|-----------------|
| Specific gravity                 | 2.64            |
| Bulk density (kg/m³)             | 15.54           |
| Fineness modulus                 | 2.72            |
| Particle size variation (mm)     | 0.09 to 2       |
| Color                            | Greyish white   |
| Water absorption in 24 hours     | 0.8%            |
| Shape of grains                  | Sub angular     |

Table 4. Chemical properties of Ennore sand

| Parameter | Result  |
|-----------|---------|
| SiO2      | 99.30%  |
| Fe₂O₃     | 0.10%   |
| Loss on ignition | 0.11% |

2.3 Micro Silica
Micro silica used throughout this investigation was obtained from Corniche India (P) Ltd, Navi Mumbai, India. The properties of the Micro silica are presented in the table 5 and table 6 respectively:

Table 5. Physical Properties

| Parameter         | Result  |
|-------------------|---------|
| Specific gravity  | 2.30    |
| Mean particle size| 0.15 microns |
| Bulk density      | 227 kg/m³ |
Table 6. Chemical Properties

| Chemical compound     | Composition |
|-----------------------|-------------|
| Silicon dioxide       | 96.65       |
| Alumina               | 0.41        |
| Iron oxide            | 0.27        |
| Calcium oxide         | 0.18        |
| Magnesium oxide       | 0.09        |
| Sulfur trioxide       | 0.46        |
| Sodium Oxide          | 0.51        |
| Ignition loss         | 1.43        |

2.4 Water

De-ionized water was utilized in reference test samples and the same with Nickel spiked in various concentrations was utilized in the experimental test samples.

2.5 Superplasticizer

Commercially available ‘conplast SP-430’ water reducing agent was used. The properties are given in the table 7.

Table 7. Properties of conplast SP-430

| Property               | Value                                      |
|------------------------|--------------------------------------------|
| Specific gravity       | 1.20 – 1.22 at 30°C                        |
| Chloride content       | Nil as per IS: 9103-1999 and BS : 5075     |
| Air entrainment        | Approx. 1% additional air over control     |

2.6 Heavy Metal

Nickel(Ni) is a heavy metal with atomic number 28. It is readily soluble in water. Nickel heavy metal spiked into de-ionized water in fixed proportions of 10 to 5000mg/L.

Physical properties of Nickel(Ni):

| Physical properties | Specific Density | Melting point | Boiling point |
|---------------------|------------------|---------------|--------------|
|                     | 8.5 gm/cc        | 1455 °C       | 2730 °C      |
2.7 **Chemicals**

2.7.1 **Acids.** Sulphuric acid of 2.5 % concentration mixed in de-ionised water and Hydrochloric acid of 2.5 % concentration mixed in de-ionised water.

2.7.2 **Alkalies.** Sodium hydroxide of 2.5 % concentration mixed in de-ionised water.

2.7.3 **Sulphates.** Magnesium sulphate of 2.5% concentration mixed in de-ionised water.

2.8 **Experimental Methods**

The investigational methods used were in line with the specified methods laid down in Bureau of Indian Standards. Nickel spiked into the de-ionized water at pre determined proportions of 10 to 5000mg/L. Based on the literature, the compositions of admixtures used are arrived at 9% Micro Silica replacement in cement and addition of Super Plaster of 0.8% was fixed for reference high performance cement mortar specimens. Physical parameters obtained for reference test samples are shown in table 8.

| Parameter        | Value  |
|------------------|--------|
| initial setting time | 72 min. |
| final setting time | 135 min. |
| cube strength     | N/mm²  |
| 3 day             | 48     |
| 7 day             | 62     |
| 28 day            | 67     |
| soundness         | 1mm    |

 Nine series of samples were prepared with “cement plus 9% SF plus 0.8% SP plus Nickel”. Nickel metal combinations ranging from 10 to 5000mg/L were spiked in de-ionized water used for experimental test samples. The mass of cement, fine aggregate and water required per cube were 200g, 600g and water of “(P/4+3)”, where ‘P’ stands for percentage of water necessary to obtain a cement paste of standard consistency. Setting times were arrived by using Vicat’s apparatus. As per IS456-2000, to test the quality of water under question for its suitability to use for construction purpose, the compressive strength of the specimens made with water in question should not differ by 10 percent with that of the cubes made from de-ionized water. Also, if the difference in initial setting time of the sample under question is more than 30 minutes it is significant otherwise it is insignificant. Lechatelier’s apparatus utilized to get the expansion of tested samples. If the expansion of the sample is more than 10mm, it is significant otherwise it is insignificant. For determining each parameter average of three specimens were considered.

The reference and experimental test samples made with cube moulds of 70.6x70.6x70.6mm for finding the compressive strength of cement. The blend of cement and fine aggregate ratio is 1:3 by mass as per the standard ratio given by code to determine the strength of cement binder. Cube strength of reference and experimental tests samples was studied at 3, 7, 28, 90 and 180 days. The fresh samples with moulds under wet gunny bags were kept at a controlled temperature of 27°C ± 2°C and at 90 percent relative humidity for 24 hours. After removing the samples from moulds, they were immersed in de-ionized water for another 27 days for curing. The compressive strength of three experimental test samples was compared with that of three reference samples. The maximum strength was attained for test samples made with Nickel concentration of 2000 mg/L. Hence, the effect of durability was studied at Nickel concentration of 2000 mg/L.

In order to study the durability aspect, effects of acidic alkaline and sulfate were investigated. Solutions of Sulphuric acid, Hydrochloric acid, Sodium hydroxide and Magnesium sulphate were
prepared with de-ionized water at 2.5% concentration in four non absorbent plastic tanks. Mortar cubes of 500 mm$^2$ cross sectional area were cast and cured in the prepared solutions then found out the strength and weight loss at 30 days, 58 days, 84 days and 180 days and the same were compared with reference samples which were exposed to de-ionized water for same age.

3 Results and Discussions

Influence of Nickel metal present in water used for mixing on the properties of blended cement are analyzed by laboratory testing for respective parameters. They include setting times, soundness, compressive strength, acidic and alkaline environment tests.

3.1 Influence on setting times

The effect of Nickel metal present in different concentrations in the mixing water on setting durations were represented graphically in the figure 1. It is noticed that the setting process of cement has increased with the increase of Nickel proportion in de-ionized water. Increase in the initial setting time is insignificant up to 4000 mg/L concentration and is significant at 5000 mg/L of Nickel, but the increase in the final setting times are more continuously but well within the permissible limit. The increase in initial setting time is 32 minutes at 5000 mg/L concentration of Nickel when compared with the test specimens done by de-ionized water, for all other concentrations it is less than 30 minutes. The final setting time is 278 minutes at 5000 mg/L, which is less than 600 minutes, the maximum value permissible. Hence, the effect of Nickel on an average over setting times is insignificant for all the specimens tested.

![Figure 1. IST and FST of various concentrations of Nickel](image)

3.2 Influence on Soundness

The effect of Nickel on soundness of blended cement mortar is represented graphically in figure 2. The effect of Nickel at all concentrations on soundness of the cement was studied. The expansion after soundness test for the reference specimen obtained with de-ionized water is 1mm. The same obtained for maximum concentrated Nickel at 5000mg/L is 1.18mm. Expansion of cement mortar with all concentrations of Nickel is well within the prescribed limits. Therefore, the effect of Nickel metal present in mixing water for blended cement mortar is insignificant.
3.3 Influence on Compressive Strength

The effect of Nickel present in the water used for mixing at different concentrations over cube strength of high performance cement is graphically represented in figure 3 below. Slight progressive increase of strength was visualized with respect to the variation of Nickel ranges up to 2000 mg/L and beyond this, the compressive strength decreases but insignificantly. Figure 4 represents the percent change in compressive strengths at various concentrations of Nickel at different ages. Hence, the effect of Nickel heavy metal on compressive strength up to 5000mg/L is insignificant.

![Graph showing soundness at various concentrations of Nickel](image1)

**Figure 2.** Soundness of specimens at various concentrations of Nickel

![Graph showing compressive strength at various concentrations of Nickel](image2)

**Figure 3.** Compressive strengths of mortar cubes at various concentrations of Nickel at different ages.
Figure 4. Percent change in compressive strengths with different proportions of Nickel at various Ages

3.4 Influence on Durability

For conducting durability tests among the reference mix cubes made with de-ionized water, cement mortar cubes made with 2000mg/L Nickel concentrated mixing water were taken for testing, at which the maximum compressive strength was attained.

3.4.1 Resistance against Acid Attack. The compressive strength results and the loss in weight of the acid attacked blended cement mortar cubes cast with different mixing compounds in deionised water are graphically represented in figure 5 and figure 6.

3.4.2 Resistance against Alkaline Attack. The compressive strength results and the loss in weight of the alkaline attacked blended cement mortar cubes cast with different mixing compounds in de-ionised water and Nickel spiked de-ionized water are graphically represented in figure 5 and figure 6.

3.4.3 Resistance against Sulphate Attack. The compressive strength results and the loss in weight of the sulphate attacked blended cement mortar cubes cast with different mixing compounds in de-ionized water and Nickel spiked de-ionized water are graphically represented in figure 5 and figure 6.
The strength of the reference and specimen mortar cubes decreases with age for all types of exposures. The decrease in strength differs from curing solutions. The lowest strength is observed for acid attack and less reduction in strength is observed for sulphate attack. The lowest strength is observed for the Sulphuric acid and then for hydrochloric acid and less reduction in strength is observed for Magnesium sulphate. Next to Magnesium sulphate, less reduction in strength is observed for alkaline concentrated water. Change in the cube strength of specimens cast with Nickel when compared with control mortar decreases as the age increases. The rate of decrease is more during initial periods of exposure. This is due to the reason that the surface of the cube erodes initially at higher rates and as the age increases the rate of erosion decreases slowly. The weight loss of the blended cement mortar cubes cast with de-ionized water when immersed in acidic media is more and considerable and the same is negligible when exposed to Alkaline and Sulphate solutions. The maximum loss in weight was found to be at 180 days in acidic media. The minimum loss in weight was found to be at 30 days in sulphate solution. This is due to the variation in pH values. The figure 6 shows the loss of weight of blended cement mortar when immersed in different media.

**Summary**

- The setting times of blended cement have augmented with rise of Nickel proportion in de-ionized water. Increase in initial setting time is insignificant up to 4000 mg/L concentration and is significant at 5000 mg/L of Nickel, but the increase in the final setting times are more continuously but well within the permissible limit.
• Expansion of blended cement spiked with Nickel deionised water is well within limits at all concentrations.
• Increase of cube strength was noticed with the proportion of Nickel rises up to 2000 mg/L. Beyond 2000 mg/L the compressive strength decreases rapidly. Percent change of cube strength at all concentrations of Nickel is insignificant.
• The blended cement mortar cubes cast with Nickel de-ionized water immersed in H2SO4, HCl solutions showed more drop in cube strength than that of controlled blended cement mortar. The same showed very less reduction of cube strength than that of controlled blended cement cubes exposed to NaOH and MgSO4 solutions. The reduction in compressive strength is more during early exposure periods when compared to longer duration exposures.
• Weight loss of the specimens is significant for blended cement cubes cast with Nickel spiked water and immersed in H2SO4 solution. The same is insignificant when the specimens were exposed to HCl, NaOH and MgSO4 solutions.

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