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Submission date: 24-May-2025 11:13AM (UTC+0700)

Submission ID: 2548488995

File name: Prosiding_International_ICONLIG_Lisa_Oksri_Nelfia.pdf (620.55K)

Word count: 3549

Character count: 17220

Analysis of High Magnesium Nickel Slag Powder as a Supplementary Cementitious Materials for High Strength Concrete

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Abstract. Concrete demand is increasing along with the development of infrastructure that is emerging in Indonesia. The development of science in the sector of concrete technology allows the use of waste as a concrete material by utilizing industrial waste, such as nickel slag due to the smelting process of nickel ore after going through the combustion and screening process. In this study, nickel slag will be used as a supplementary cementitious material to evaluate results on high-strength concrete. Samples were made with water cement ratio of 0.31 and will be compared with 100% OPC Type 1 concrete as a reference (Ref). Nickel slag powder (NSP) substitution of cement was 5%, 10%, 15%, 20%, 25% and 30% with the curing age of 3 days, 7 days, 14 days dan 28 days. Specific gravity tests for NSP and cement were done by the Le Chatelier method. Mineral characterization using Scanning Electron Microscopy (SEM) was carried out on nickel slag powder used and concrete substitution 30% NSP aged 3 days. The mechanical characteristics include ease of work in the field (workability) and compressive strength. Concrete was targeted to reach an average compressive strength of ≥ 72 MPa at age 28 days using superplasticizer type Sika Viscocrete 8015. The compressive strength result of NSP concrete substitution at age 28 days was 82.94 MPa for 5% NSP, 82.28 MPa for 10% NSP, 79.98 MPa for 15% NSP, 77.89 MPa for 20% NSP, 77.26 MPa for 25% NSP and 73.80 MPa for 30% NSP.

Keywords: Compressive strength, Nickel slag powder, Supplementary cementitious materials, Scanning electron microscopy.

1 Introduction

The development of concrete technology has made it possible to use waste as a concrete material, so that waste can be utilized optimally. Based on these thoughts, many studies

tried to improve the quality of concrete from the materials side by substituting it with natural materials, both in coarse aggregates or fine aggregates, and some are used as supplementary cementitious material (SCMs) to increase the adhesion of concrete binders by utilizing industrial waste for concrete mixtures, for example; nickel slag powder. In the city of Southeast Sulawesi, Kabaena Island has a fairly high nickel content, it was first discovered in 1970, when PT. INCO Tbk, one of the largest nickel companies in Indonesia, sent some experts of its geologists to conduct a survey and ensure that 80% of the island has a fairly large nickel content and a fairly high level of nickel [1]. In every year, PT. INCO produces a relatively large amount of solid waste, namely nickel slag, which is around 70,000 – 72,000 tons. Much of the concrete produced (either normal or high-strength concrete) made with waste has superior properties compared to conventional concrete in terms of strength, performance and durability [2].

Based on research conducted [3], it is clear that the use of nickel slag in concrete mixtures increases the mechanical properties of concrete, especially its compressive strength. The increase that occurs can reach about 45%, 24% and 19%, respectively, for compressive strength, split tensile strength and modulus of elasticity. This good improvement is related to the physical form of nickel slag aggregates. Research [4] [3], concluded that nickel slag has a positive impact as a substitute for concrete aggregates, and increases the compressive strength of concrete. And also in research [5] [2], concluded that slag as a substitute has a good impact on the strength and durability of high-strength concrete. This previous research has encouraged us to make high-strength concrete using nickel slag as a supplementary cementitious material. Through this approach, the consumption of natural materials, thermal energy and electrical energy, along with CO₂ emissions can be reduced [6].

2 Experimental

Nickel slag used sifted nickel slag that was sieved using a sieve shaker machine with #200 sifter for ± 10 minutes, it produces very fine grain sizes (< 0.075 mm) which is called nickel slag powder (NSP). NSP will be as hydraulic as cement, and based on Trace Metal Analysis results conducted by Indocement, NSP was proven not to contain any dangerous heavy metal. With water cement ratio of 0.4, NSP concrete substitution can reach about $f_c' 30$ MPa up to a 25% substitution rate.



Fig. 1. Nickel Slag Powder (NSP).

Preparing samples and material testing were carried out at PT. Jaya Beton Indonesia. Samples were made in cylinders ($\varnothing = 200$ mm; H = 100 mm). Obtained data conducted from the testing process at Laboratorium PT. Jaya Beton Indonesia Laboratory includes the test results of coarse aggregates and fine aggregates, compressive strength, and concrete mixture workability test with water cement ratio of 0.3. NSP substitution of cement were 0%, 5%, 10%, 15%, 20%, 25% dan 30%, where 0% NSP substitution was Ordinary Portland Cement (OPC) as a Reference (Ref). Compressive strength was performed at the concrete age of 3 days, 7 days, 14 days, and 28 days. Samples were put in the water (water curing) until the age of concrete reached the age needed for the test. The total samples in this study were 91 samples, with 84 samples for compressive strength and 7 samples for scanning electron microscopy (SEM).

2.1 Materials

The cement which used as a comparison material is OPC Type 1 originated from PT. Indocement Tiga Roda. Cement has an adhesive and cohesive character, that is used for binders which react with water, and used as a concrete mixture with coarse aggregates and fine aggregates. Water was taken from the laboratory facility of PT. Jaya Beton Indonesia. The coarse aggregates come from Gn. Sindur and fine aggregates come from Gn. Sudamar. Coarse aggregates maximum size of 5 mm and fine aggregates maximum size of 20 mm. Specific gravity test of NSP and cement using the Le Chatelier method refers to ASTM C.188 at saturated surface dry (SSD) condition with the result of 2.797 for NSP and 3.14 for cement.

Material testing for coarse aggregates and fine aggregates refers to Japan Industrial Standards (JIS), with the following information:

Table 1. Materials test result.

Parameter	Coarse Aggregates	Fine Aggregates
Specific Gravity (SSD)	2.59	2.57
Unit Weight	1.49 gr/cm ³	1.66 gr/cm ³
Fineness Modulus		
Water Absorption	2.38 %	2.17 %
Water Content	3.41 %	9.20 %
Sludge Levels	-	1.95 %
Abrasion Test (Los Angeles)	17.76 %	-

NSP substitution of 0% (Ref), 5%, 10%, 15%, 20%, 25% and 30% from the mass of cement. Concrete samples of 0% were used as a reference (Ref) with a compressive strength result targeted to reach $f_c' = 63$ MPa at the age of 28 days. To reach great workability, superplasticizer type Sika Viscocrete 8015 was added as an admixture. Calculation of concrete mix design refers to SNI 03-6468-2000, 2000, although this regulation only regulates the mix design of high-strength concrete using fly ash, fly ash is one of a pozzolanic material, while NSP was categorized as a pozzolanic material which has been confirmed by the results of X-Ray Diffraction (XRD) analysis [8]. Based on the ratio of $W/(c+p)$ with superplasticizer, the water-cement ratio was set at 0.31.

Table 2. Mix design.

Material	Ref	NSP 5	NSP 10	NSP 15	NSP 20	NSP 25	NSP 30
Water (kg)	96.32	84.86	85.14	85.42	85.70	85.98	86.26
Cement (kg)	591.94	562.34	532.74	503.15	473.55	443.95	414.35
NSP (kg)	0	29.60	59.19	88.79	118.39	147.98	177.58
Coarse (kg)	1109.38	1109.38	1109.38	1109.38	1109.38	1109.38	1109.38
Fine (kg)	590.85	587.54	584.22	580.90	577.58	574.26	570.94

^a Proportion per m^3 of the mixture (according to aggregate wetness condition)

The mixer capacity was up to $0.3 m^3$. The mixing methods start with 100% coarse aggregates and fine aggregates in saturated surface dry (SSD) conditions. Followed by 100% cementitious materials in dry condition. These materials were mixed for approximately 3 minutes. Next, add 50% water and 100% superplasticizer was slowly poured in. Thereafter, 50% water was added. According to the steps of the materials placed, the concrete was mixed for approximately 6 minutes. The workability of concrete was measured by the concrete slump test. Slump was allowed to reach ≥ 200 mm for high-strength concrete with superplasticizer according to SNI 03-6468-2000.

2.2 Methods

Compressive strength was performed at the concrete age of 3 days, 7 days, 14 days, and 28 days. Samples were put in the water (water curing) at a temperature of $20^\circ C$ until the concrete had reached the age needed for the test. At the compressive strength test, samples will be pressed with optimum strength.



Fig. 2. Concrete samples on preparation.



Fig. 3. Concrete samples on water curing.

Properties of Concrete. Workability test using slump test with Abram's cone where the mould size is height = 150 mm and Diameter is 100 mm. Testing the workability of concrete to know the slump value of each concrete made in cylinder size ($\phi=100\text{mm}$ and $H=200\text{mm}$). For testing the compressive strength is analyzed based on ASTM C470. Each of the concrete specimens was tested at 3, 7, 14 and 28 days using a PILOT Press digital compression device (Capacity 2000 kN). Before testing the compressive strength, the concrete must first soak at a water temperature of 20°C . Superplasticizers are not used in the concrete fabrication process. However, the addition of admixture in the concrete mixture will affect the concrete properties and ease of work in the field.

Characterization of Nickel Slag Powder (NSP) The density of NSP was measured using the Le Chatelier procedure according to the standard test method for the density of hydraulic cement [9]. Scanning Electron Microscopy (SEM) was conducted to determine the surface characteristics and object texture, to find out the shape and sizes of the constituent particles of the object, to observe the grains structures of the object, and to obtain quantitatively data of the elements and particles contained in the object. SEM was performed on one sample, namely 30% NSP for the age of 7 days. For the 30% NSP sample, cylindrical samples are cut using a bar cutter until the sample has dimensions $\pm 1.5 \times 1.5 \text{ cm}$ and $\pm 2 \text{ mm}$ thick. After the sample was cut, the sample using abrasive paper. First using CAMI 60 abrasive paper (rough), then using CAMI 80 abrasive paper (medium), then finally using CAMI 100 abrasive paper (fine). After the surface of the sample is very smooth, the sample is ready for SEM.

Concrete samples that have been prepared are entered into a vacuum and coating system. The samples must reach 2×10^{-1} millibar for the coating process. The thicker and larger the sample, the longer the vacuum process takes time. The sample should be made at least $\pm 2 \text{ mm}$ with an area of $2 \text{ cm} \times 2 \text{ cm}$. After the sample finishes the vacuum process, the sample will be coated with gold so that the surface can be read by an electron microscope. The coating process lasts for ± 2 minutes. After that, the coated sample is inserted into an electron microscope. The electron microscope used is brand Inspecttm

F. Zoom the sample 11 times to 500 times then specify several points to view the EDS (Energy-Dispersive X-Ray Spectroscopy). EDS serves to identify the mineral content in the sample being tested. Zoom the sample 2000 times to 3000 times at the specified point.

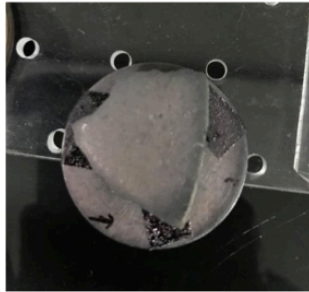


Fig. 4. 30% NSP Concrete sample for SEM.

3 Results and Discussion

3.1 Compressive Strength

The compressive strength test was applied after the concrete aged 3 days, 7 days, 14 days and 28 days of water curing at 20°C by using three cylinders for each test. Compressive strength data are listed in Table 3 and shown in Figure 5.

Table 3. Compressive strength result.

Material	Ref	NSP 5	NSP 10	NSP 15	NSP 20	NSP 25	NSP 30
fc' at 3 days (MPa)	66.17 ±0.63	66.04 ±1.41	59.80 ±0.97	58.03 ±0.89	55.68 ±0.98	53.68 ±0.86	51.82 ±3.06
fc' at 7 days (MPa)	71.47 ±1.16	66.55 ±1.20	59.98 ±0.46	59.39 ±0.61	58.01 ±0.84	56.33 ±0.54	52.31 ±1.37
fc' at 14 days (MPa)	72.83 ±2.21	72.52 ±8.14	72.45 ±4.68	65.70 ±0.67	59.37 ±1.78	58.91 ±2.87	58.06 ±10.55
fc' at 28 days (MPa)	80.49 ±0.26	82.94 ±7.79	82.28 ±0.58	79.98 ±0.39	77.89 ±2.45	77.26 ±0.75	73.80 ±6.27

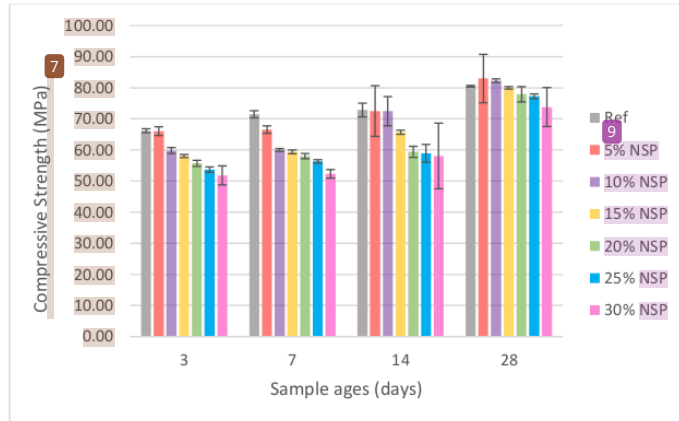


Fig. 5. Compressive Strength result.

Based on the results of the compressive strength obtained, it can be seen that the nickel slag powder gives increased strength to each of its substitutes. The compressive strength value increases until the concrete age of 14 days. At the concrete age of 28 days, 5% NSP and 10% NSP got a higher compressive strength result than the Reference. The higher strength result of the NSP substitution concrete is due to the pozzolanic reaction of Nickel Slag Powder (NSP), according to research [10] nickel waste is a material that has a pozzolanic effect, where the composition of the chemical compound is $\text{SiO}_2 + \text{Fe}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 70\%$ as regulated in the specifications ASTM C618-93. In this case, the nickel waste described was nickel slag powder which is used as a partial substitution material for cement. The use of slag can increase the compressive strength of concrete by 22%. It can be concluded that all NSP substitution concrete samples reach the compressive strength of $f_c' \geq 41.4$ MPa which can be categorized as high-strength concrete according to SNI 03-6468-2000.

3.2 Scanning Electron Microscopy (SEM)

It is well known that for a given substitution level with mineral admixtures, the early ages characteristic of high-strength concrete is influenced by the reactivity of mineral admixtures. The more reactive the mineral admixtures are, the higher the early strength of the concrete will be [11]. SEM was performed on 30% NSP concrete substitution at the age of 7 days. SEM aims to see and investigate the surface of objects directly. The magnitude for the SEM test starts from 150 times to 3000 times. Later Energy-Dispersive X-ray spectroscopy (EDS) will identify the percentage of mineral compound content in the specimens test, where the results are strengthened by the results of the X-ray fluorescence (XRF) [8].

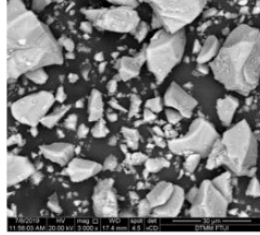


Fig. 6. Magnitude 3000 times of nickel slag powder (NSP)

It can be observed on the 3000 times magnified nickel slag powder, that the nickel slag powder has a sharp and irregular shape. Besides, the EDS results show that nickel slag powder predominantly contains silica, calcic, magnesia and alumina.

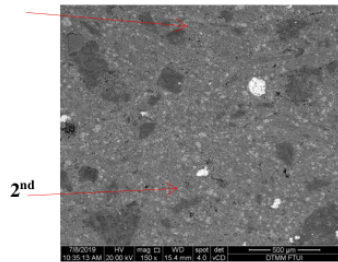


Fig. 7. Magnitude 150 times of 30% nickel slag powder concrete aged 7 days.

Magnification of 150 times as a global image on a sample of 30% NSP concrete that is 7 days (Fig. 7). Observations were also made at several points because the global image shows differences in the shape, texture, size and colour of the object's constituent particles. The solid black colour indicates voids due to air voids in the concrete. Referring to research (Rahman et al, 2016), the formation of a black gap between the fibre and matrix on the object indicates the presence of pores in the object. For the point being reviewed, a magnification of 3000 times is carried out so that the particles making up the object can be seen more clearly.

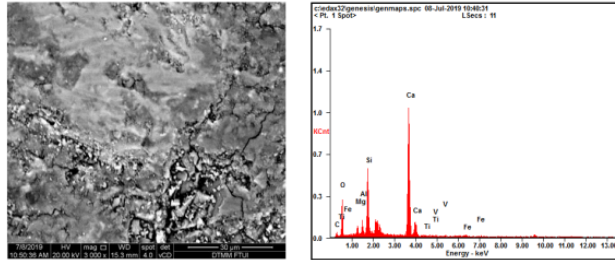


Fig. 8. Magnitude 3000 times of 30% nickel slag powder concrete aged 7 days with EDS 1st spot.

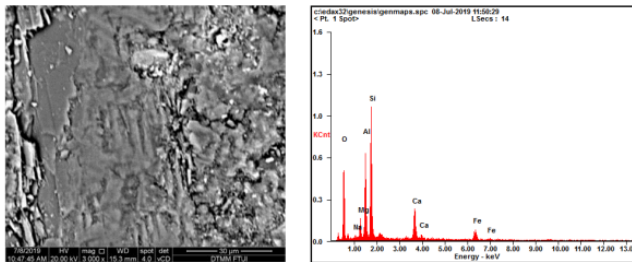


Fig. 9. Magnitude 3000 times of 30% nickel slag powder concrete aged 7 days with EDS 2nd spot.

It can be seen from the results of the SEM test that has been carried out, that the first spot is the point where the nickel slag powder gathers, indicated by the high levels of silica (Si) and magnesium (Mg). The second spot shows high silica (Si) where the compound is the same as the compound contained in cement and can form a pozzolanic reaction [13].

4 Conclusion

Nickel slag powder can be categorized as an alternative construction material as Supplementary Cementitious Materials (SCMs), which can minimize environmental damage due to nickel slag waste. With a water-cement ratio design of 0.31 plus an admixture of superplasticizer type, the compressive strength of NSP substituted concrete at the age of 3 days is relatively high at 66.04 MPa, 59.80 MPa, 58.03 MPa, 55.68 MPa,

53.68 MPa and 51.82 MPa. The results of the compressive strength are almost equivalent to reference concrete (Ref) at 66.17 MPa. The 30% NSP substitution concrete can be used as an alternative concrete because it can be categorized as high-strength concrete and is more environmentally friendly.

It is proven that nickel slag waste can increase the result of compressive strength, the shape and texture of the slag also affects the density of concrete so that it can increase the result of compressive strength. The compressive strength of 5% and 10% NSP substituted concrete obtained higher results than 100% OPC (Ref) concrete at the age of 28 days, this was due to the pozzolanic reaction of nickel slag powder. NSP reacts for a longer period than cement which reacts in the short term. NSP has been shown to achieve adequate compressive strength at an early age while maintaining higher long-term strength than OPC. This research illustrates that high-strength concrete innovation can be made from waste; it can be proven in this study with nickel slag waste. In addition, the use of nickel slag powder as a supplementary cementitious material can minimize nickel slag waste and reduce CO₂ emissions produced by clinker from cement production.

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Acknowledgements

We would like to thank all contributors from Jaya Beton Indonesia Ltd for their help in preparing samples and supporting materials, PT. Indoferro for supplying raw materials and DTMM-FTUI for the help in testing materials. We are grateful to Universitas Tri-sakti for their support, and generous assistance.

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