A Study of the properties and microstructure of high-magnesium nickel slag powder used as a cement supplement

by Lisa Oksri Nelfia

IOP Conference Series: Materials Science and Engineering

PAPER • **OPEN ACCESS**

A Study of the properties and microstructure of high-magnesium nickel slag powder used as a cement supplement

To cite this article: L Oksri-Nelfia *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **829** 012007

IOP Conf. Series: Materials Science and Engineering **829** (2020) 012007 doi:10.1088/1757-899X/829/1/012007

A Study of the properties and microstructure of high-magnesium nickel slag powder used as a cement supplement

L Oksri-Nelfia[13], Reynaldi Akbar[1], and Sotya Astutiningsih[2]

[1]Departement of Civil Engineering, Faculty of Civil Engineering and Planning, Trisakti University, Jakarta 11440, Indonesia
[2]Departement of Metallurgical and materials Engineering, University Indonesia, Depok 16424, Indonesia

[3]E-mail: lisa@trisakti.ac.id

Abstract. This research aims to use high-magnesium nickel slag crushed into a powder as a replacement for cement and to study their properties and microstructure in concrete. The particle diameter of the nickel slag powder was obtained lower than 75 μm after a process of crushing and sieving. The specific areas, absolute dry densities and particle size distribution (PSD) were analysed to obtain the physical properties of the material. The techniques applied to study their chemical and mineralogical composition were the X-Ray Florescence (XRF), the Scanning Electron Micrograph (SEM). The OPC type 1 was used as a reference in comparison to the experimental material of this research. Without a chemical admixture, the compressive strength of concrete after 28 days is fc' 30 MPa and the specific surface area is 4360 cm^2/gr. The compressive strength of high-magnesium nickel slag powder is capable of replacing cement by 20% without altering properties of concrete. According to the XRF technique results, the nickel slag contains silica, calcium, magnesia, and alumina. Furthermore, the image of the SEM-EDS illustrated the slag particle to be sharp, irregular, and containing calcium, silicon, alumina, oxygen, and magnesium. This result confirmed that the XRF technique and SEM-EDS were accurate.

1. Introduction

Indonesia is a country that has many natural resources with numerous mining activities including nickel, iron, gold, coal. However, it is also the third source of environmental pollution in the country, so an environmental consciousness for mining expansion needs to be developed. This must start at the beginning of production and during the process, to enable a sustained establishment [1]. In several mining sites, there is slag waste and there are several cases of B3 pollution (harmful and toxic substance) released from mining sites and so far, no solution has been provided [2]. Currently, the nickel mining produces a redundant nickel slag and its fusion in the domestic sector ranging up to 5 million tons/year with the assumption of producing NPI or FeNi (Ni level is 10%). The standard input of Ni ore substance is approximately 40 million tons/year, which means that 30 million tons become slag [3].

Therefore, this research aims to explore the use of nickel slag and to prevent its effect on the environment. A nickel slag is a solid waste product that is obtained from the ore quarry process [4]. Therefore, the waste system needs to be organized properly to prevent environmental problems. Research has been undertaken to find solutions using the powdered slag obtained from nickel to minimize environmental problems. The compressive strength of the concrete has been improved by

IOP Conf. Series: Materials Science and Engineering **829** (2020) 012007 doi:10.1088/1757-899X/829/1/012007

replacing sourced materials for concrete with waste the nickel slag aggregates [5]. According to the recent research of using green construction (eco-friendly), the cement quantity can be modified to pozzolan substance such as fly ash [6]. The purpose of this type of concrete is to reduce environmental problems such as global warming, the overexploitation of natural resources, and the waste obtained from the quarries, households, or construction sites. A replacement substance for cement needs to be distinguished as a substitute material, so it can be used in the concrete production.

The use of cement in building construction or other infrastructure work creates negative impacts on the environment. The literature [7] revealed that each ton of cement production generates nearly 0.8 tons of carbon dioxide, which is released into the atmosphere. It is caused from using a large use of energy and a high temperature of approximately 1450°C to produce the clinker, which is the main component of cement production [8, 9]. Hence, the industrial cement causes 5% of the emission from the greenhouse gases [10, 11, 12]. In some research, the cement was partially replaced by pozzolanic wastes product known as fly ash, which comes from coal-burning electricity generation, and ground granulated blast furnace slag from iron production waste, also used are gypsum, Limestone filler. Such methods will reduce the carbon dioxide emission that comes from its clinker production. It is used to produce the eco-friendly concrete by utilizing the industrial production residue (Green Concrete). The substitution of cement is effective for reducing the carbon dioxide emission during production, especially when it is combined with an additional substance [12, 13]. The present research [14, 15], the C-A-S ternary diagram approach is a reaction of the nickel slag powder with water. When it is mixed with water it becomes portlandite reaction $Ca(OH)_2$.

The objective of this research is to use high magnesium nickel slag powder as partial substitute for cement, with the maximum substitution rate up to 30%. The nickel slag powder undergoes a test to determine its physical, chemical, and mineralogical characteristics. The research illustrates the following tests: the pycnometer, the Blaine test, workability, compressive strength and the new concrete production made with a mixture of the nickel slag powder. The compressive strength were conducted in a time phase of 2, 7, 14 and 28 days, with substantial replacement composition of 5%, 10%, 20% and 25% to the weight of the cement. Concrete samples with different substitutions, were named successively NSP5, NSP10, NSP15, NSP20 and NSP25. The concrete with NSP samples was compared to concrete made with 100% OPC type 1 as a reference concrete.

2. Materials and methods

2.1. Preparation of high-magnesium nickel slag powder (NSP)

The nickel slag used was obtained from Southeast Sulawesi, Indonesia. This raw material was prepared by using industrial ball mill came from PT. Growth Java, Cilegon. Indonesia. In order to obtain a granular size less than 0.075 mm, the NSP (nickel slag powder) was filtered by sieving it with a machine strainer of #200 in a dry condition (room temperature between 20-25 °C). After the sieving and straining process that took 10 minutes, the NSP was produced (Figure 1). In addition, to determine the absolute dry density, the nickel slag should be in a dry condition; hence, it was kept in an oven for ± 24 hours with a temperature of 110 °C and it was later placed in desiccator's for ± 24 hours to ensure the nickel slag was in room temperature condition. After that process, the nickel slag can be used in the next test process.

Figure 1. Sample of NSP.

IOP Conf. Series: Materials Science and Engineering **829** (2020) 012007 doi:10.1088/1757-899X/829/1/012007

2.2. *Physical, chemical and mineralogical characterization*

The density of NSP was determined by using a pycnometer according to European standard EN 1097-6 [16]. Specific surface area was determined by Blaine test according to European standard EN 196-6 [17]. The particle size distribution and the median diameter (D_{50}) were measured by laser diffraction in dry suspension. The results of the physical characterization are shown in Table 1**.** Then**,** the chemical composition of OPC type 1 and the NSP were obtained from the X-Ray Florescence (XRF) by using EPSILON 5 analyzer from Panalytical, The result is presented in Table 2. According to these results, the main chemical components of nickel slag powder are silica, alumina, calcium, and magnesia. This results are confirmed by literature review [18, 19].

Table 1. Physical analysis of materials studied [20].

Material	Density (g/cm^3)	Blaine (cm^2/g)	D_{50} (μm)
NSP	2.75	4360	15.4
OPC Type 1 Tiga Roda	3.15	3670	14.90

Table 2. Chemical analysis of materials studied.

Chemical composition	OPC Type 1 Tiga Roda	NSP
CaO	63.2	23.58
Al_2O_3	4.96	8.85
SiO_2	18.45	42.03
Fe_2O_3	2.86	1.59
SO_3	2.18	-
MgO	3.52	19.51
K_2O	0.31	0.12
Na_2O	0.15	0.08
LOI	3.42	2.59
$CaCO_3$	-	-
C_3S	68.72	-
C_3A	8.30	-
C_4AF	8.70	-

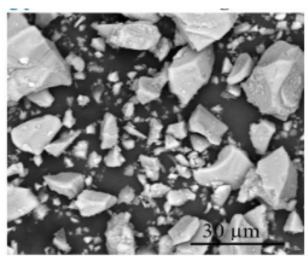


Figure 2. The microstructure of NSP.

Scanning electron microscope (SEM) characterization was carried out with a Brücker-Zeiss EVO MA10 instrument in order to study the morphology of slag particles. The microstructures of nickel slag powder are shown in Figure 2. 28 day concrete samples (NSP 20) were crushed with a compressive strength machine. A fracture-shaped sample with a maximum thickness of 2.5 cm were tested by SEM. Chemical characterization were determined by testing heavy metal leachability (also called trace metal analysis with EPSILON 4, Malvern Panalytical instrumentation). Leaching tests consisted of subjecting a material to the action of a solvent in order to quantify chemical elements (pollutants or not) released by the material. The aqueous solution was placed with a liquid/solid ratio (water/powder) of 10 L/kg, according to the standard NF EN 12457-2 [21]. The leaching agent was demineralized water and the contact process was carried out over a period of 24 hours. At the end of this period, the liquid/solid separation was carried out by filtration. The purpose of these tests is to allow the quantification of heavy metals, arsenic (As), barium (Ba), cadmium (Cd), chromium (Cr), copper (Cu), nickel (Ni), lead (Pb), selenium (Se) and zinc (Zn) in the studied samples. Most metals are dangerous to the environment beyond a certain threshold. The samples were prepared according to the leaching method.

2.3. Composition of concrete with NSP and OPC type 1

To use the nickel slag as the supplementary cementitious materials (SCMs), the concrete was designed on the Japanese International Standard (JIS A 5004), with almost 56% sand in the total aggregate (volume of fraction of sand fixed) (S/A) and a cement water factor (W/C) of 0.47. For testing fine aggregate and coarse aggregate based on JIS A 5004, the fine aggregate passes the cleanliness test, (Sludge Level ≤ 7.0%). Fine aggregate with specific gravity (SSD) was 2580 kg/m^3 and has an absorption of 0.2%, coarse aggregate with specific gravity (SSD) was 2650 kg/m^3, absorption of 0.23%. Ordinary Portland cement (OPC) Type 1 used was Tiga Roda (Indocement Ltd). Composition of the concrete mixture was compared with the compressive strength target, $f_{c'}$ = 30 MPa). The concrete was made by gradually substituting the mass of cement by the nickel slag powder (NSP) from 0% to 25%. The total samples were 72 specimens. The comparison of water ratio to the cement mixture and NSP (water to binder ratio of mortars) (W/ (A/C) is constant and the water and pasta volume were constant. The details can be seen in Table 3. Compressive strength was tested at the ages 3, 7, 14, and 28 days.

Table 3. Mix proportions (in kg/m^3) and properties of the studied concrete.

	Ref	NSP				
		5	10	15	20	25
OPC type 1 Tiga Roda	400	380	360	340	320	300
Nickel slag powder (NSP)	0	20	40	60	80	100
Gravel 10/20	1030	1030	1030	1030	1030	1030
Sand 0/5	662	662	662	662	662	662
Water	195	195	195	195	195	195
W/C	0.47	0.47	0.47	0.47	0.47	0.47
W/(C+A)	0.47	0.47	0.47	0.47	0.47	0.47
Slump (cm)	2 ± 0	2 ± 0.6	4 ± 0.8	4 ± 0.8	6 ± 1	6 ± 1
28-day compressive strength (MPa)	41.7	39.8	36.1	34.2	30.2	28.4

2.4. Properties of concrete

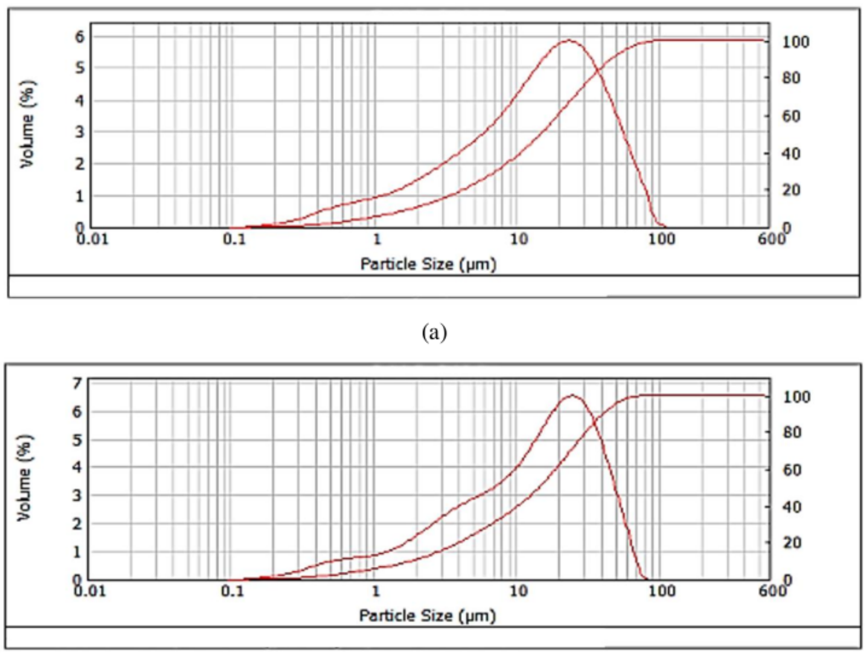
The research analyses the workability by using the slump test with Abrams cone (H = 150 mm, Ø = 100 mm). Furthermore, a test was conducted on the workability of concrete to determine the value of slumps from each concrete produced in a cylinder form (Ø=100mm and H=200mm). The compressive strength was analysed based on the ASTM C470; within a period of 3, 7, 14 and 28 days with digital compression machine PILOT Press (capacity 2000 kN). Before testing it, the concrete should be cured in a water temperature of 20 °C. In making concrete samples, it should be underlined that concrete was made without the addition of superplasticizer. Indeed, it is known that chemical mixing can modify the hydration kinetics [22].

3. Results and discussion

3.1. Properties of NSP

3.1.1. Physical properties

The result obtained from the absolute dry density, specific surface area and median diameters are shown in Table 1. The absolute dry density of nickel slag powder was 2.75 t/m^3 and cement was 3.15 t/m^3. The absolute dry density NSP was smaller than the density of cement. In addition, the NSP specific surface area was higher than cement. Therefore, the nickel slag can be a supplementary cementitious materials (SCMs). It also influences the compressive strength and workability result [23]. Figure 3(a) shows that the particle size distribution (PSD) with median diameter (D_{50}) of nickel slag powder (NSP) at 15.4 μm was almost the same as the median diameter of OPC type 1 at 14.90 μm in figure 3(b).



(a)

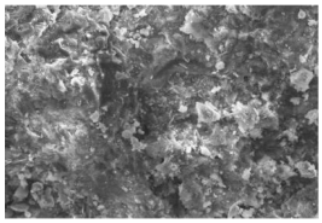
(b)

Figure 3. Particle size distribution of (a) NSP (b) OPC type 1.

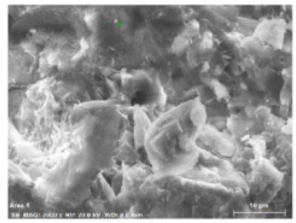
3.1.2. Chemical and mineralogical composition

According to trace metals testing for raw material analysis, the assay showed that the arsenic, cadmium, and mercury level are below the threshold of nickel slag powder (NSP) as shown in literature [20]. Hence, this substance is very safe for the environment and can be implemented as the new substitute of cement [4]. Cement industry (PT Indocement Ltd) has been accepted criteria for trace element solid waste in this case for this sample.

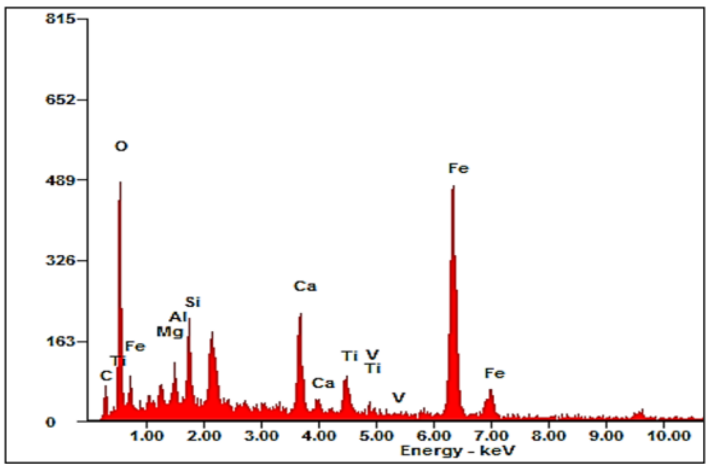
The figure 4 shows the results obtained from the SEM images and EDS test on the mineral composition for NSP 20. The EDS data results was carried out to identify dominant element of NSP. In figure 4(a) the SEM image was magnified 500 times. Furthermore, it uses a 20 kV SE detector as the age image obtained from a concrete sample for NSP25 at 28 days. From the SEM, there was a 20μm particle size of NSP; the image forms a particle that contains each mineral element, which was explained in the EDS result in figure 4(c), Magnesia, Alumina, calcia, Silica, iron and Oxygen dominate the EDS data result [19]. In addition, figure 4(b) shows that the SEM images were magnified 2000 times (microscope scale magnification) with a 20 kV SE detector. This Figure shows that Silica and Oxygen dominate the EDS results. The colour indicated that it contains dominant Iron (Fe), and it is lighter than the Silica (Si) [24].



(a)



(b)



(c)

Figure 4. SEM images NSP20 concrete sample at 28 days in (a) with 500 x of magnification and (b) with 2000x magnification (c) EDS analysis.

3.2. Properties of concrete prepared with NSP

The influence of NSP on concrete consistency is a higher result obtained in the workability test on NSP, whatever the substitution rate, than OPC Type 1 as shown in Table 3. According to literature [25], the workability result in each substitution level is always higher than the material of reference, it means that nickel slag does not absorb water (low water absorptions). This result is surprising considering the fineness of NSP particles finer than cement. In fresh state, recycled material tend to affect the rheology of concretes because it absorbs less water and increases the compactness of the granular skeleton, because of their nature and shape.

The influence of NSP on the compressive strength is based on the data provided in figure 5, the research concludes that the compressive strength for the partial cement substitution is 20% of the cement mass at f'_c 30 MPa at 28 days. Above 25% substitution, the compressive strength is below 30 MPa and so on, the compressive strength of the design target. Based on the research [23], it stated that there was a power improvement in each age due to its high-density level and low porosity. It is influenced by a higher nickel refinement. The higher the cement substitution of the nickel slag is, the lower the compressive strength is obtained with different curing times. Based on European standard EN 206-1, the substitution of cement level must not exceed 25% because it will affect the mechanical characteristics and durability of the concrete.

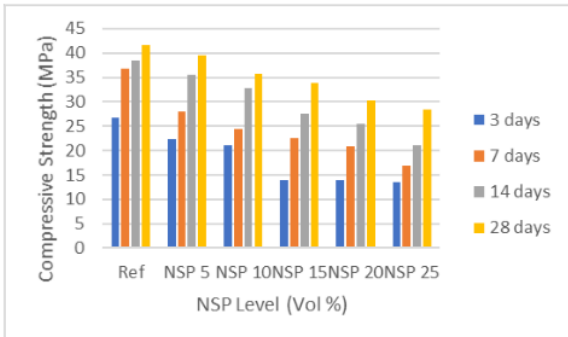


Figure 5. Effect of NSP on compressive strength of concrete.

4. Conclusions

The results from the study showed that NSP can be used as substitution for OPC type 1 in concrete mixture. It is expected to become an alternative construction material. The NSP with a size of 0.075 mm has finer properties and lower water absorptions rate compared to OPC type 1. In general, the obtained affirmative results revealed that NSP can replace up to 20% of the cement in the concrete mixture without altering properties of concrete and it has compressive strength of 30 MPa at 28 days of the concrete. This nickel slag contains a high magnesium level, where the results are proven by XRF and SEM-EDS. However, we need to do further tests of the compressive strength at age of 56, 90 and 180 days to see the pozzolanic activity of this material. It causes a higher compressive strength than normal concrete (without NSP substitution) long term conditions.

5. References

[1] Zainul R, Oktavia B, Dewata I, Efendi J 2018 Study of Internal Morphology on Preparation of Cu_2O Thin-Plate using Thermal Oxidation. *Int Conf on Science and Technology*

[2] Arino A M & Mobasher B 1999 Effect of ground copper slag on strength and toughness of cementitious mixes. *ACI Materials Journal,* **96**(1), 68-73

[3] KEPEMENPERIN 2018 Support of Slag Utilization Policy for Downstream Industry and Infrastructure Sectors

[4] Salain, I, Made A, Sudarsana, K, and Mustika W 2015 Mechanical properties of concrete using nickel slag as coarse mechanical properties of concrete, *International Journal of Engineering Inventions*, Vol 4, (9), p 7-16

[5] Achmadi A L I 2009 Slag Sebagai Agregat Halus Dan Agregat kasar Program Pascasarjana (*Slag as a Fine Aggregate and Coarse Aggregate*) post graduate program report

[6] Azeez Bahedh M, Saleh Jaafar M 2018 Ultra High-Performance Concrete Utilizing Fly Ash as Cement Replacement under Autoclaving Technique, Case studies in construction materials journal, vol 9

[7] Bastier, R 2000 Fours de cimenterie, ateliers de cuisson du clinker. Tech. L'ingénieur

[8] Mehta PK 1999 *Concrete technology for sustainable development*. Concr Int 47–52

[9] World business council for sustainable development (WBCSD) 2009 Cement industry energy and CO performance getting the Numbers Right (GNR)

[10] Humphreys K and Mahasenan M 2002 Toward a sustainable cement, substudy 8: Climate Change, World Business Council for Sustainable Development, Cement Sustainability Initiative

[11] Dalgleish T, Williams J M G, Golden A M J, Perkins N, Barrett L F, Barnard P J, Yeung C, Murphy V, Elward R, Tchanturia K, Watkins E 2007 Handbook of Low Carbon Concrete, Elsevier L. ed, *J. of Experimental Psychology: General*. *Joe Hayton*

[12] Hendriks A, Worrell D, Jager D, Block K and Riemer P 2000 Emission reduction of greenhouse from the cement industry. *Tech report*, IEA greenhouse gas R&D Program

[13] Flower D J M and Sanjayan J G 2007 Greenhouse gas emissions due to concrete manufacture. Int. J. Life Cycle Assess. **12**, 282–288

[14] Ashad H 2008 Optimisasi Bubuk Slag Nikel dengan Sistem Ternary C-A-S (*Optimization of Slag Powder Nickel with Ternary Systems C-A-S*) Vol 15, No 3.

[15] CIMBéton 2004 La durabilité des bétons. T48. Bétons ouvrages d'art 93

[16] NF EN 1097-6 2014 Essai pour déterminer les caractéristiques mécaniques et physiques des granulats - partie 6 : Détermination de la masse volumique réelle et du coefficient d'absorption d'eau

[17] EN 196-6 1990 Method of testing cement. Part 6: Determination of fineness

[18] Astutiningsih S and Suharno B 2019 NI SLAG VALORISATION IN INDONESIA : A CASE STUDY Slag characterization, *Int Slag Valorisation Symp* (Mechelen, Belgium)

[19] Wu Q, Wu Y, Tong W and Ma H 2018 Utilization of nickel slag as raw material in the production of Portland cement for road construction. *Constr Building Materials* 193, 426–434

[20] Oksri-Nelfia L, Akbar R, Astutiningsih S 2019, KoNTekS 13 Proc, Aceh, Indonesia (To be published)

[21] NF EN 12457-2 2002 Lixiviation - essai de conformité pour lixiviation des déchets fragmentés et des boues

[22] Siler P, Kolarova I, Kratky J. Havlica J and Brandstetr J 2014 Influence of superplastisticizers on the course of hydration of Portland cement hydration Chem.pap.**68**, 90-97

[23] Sugiri, S 2005 Penggunaan Terak Nikel sebagai Agragat dan Campuran Semen untuk Beton Mutu Tinggi (Use of Nickel Slag as an Aggregate and Cement Mixture for High Quality Concrete), *J. infrastructure and Built Environment*, Vol 1 No 1

[24] Sujiono E H, Zharvan V, Yunita S N and Samnur S 2018 Influence of PT Vale's Granulated Gradation Nickel Slag Aggregate on Compressive Strength of Concrete *IOP Conf. Series: Materials Science and Engineering*

[25] Oksri-Nelfia L, Mahieux P Y, Amiri O, Turcry P, Lux, J 2016 Reuse of recycled crushed concrete fines as mineral addition in cementitious materials. *Mater. Struct. Constr.* **49**, 3239–3251

Acknowledgements
The author would like to acknowledge Budi Hartono (PT Indocement Ltd, Citereup) for chemical analyses by heavy metal leachability and PT Jaya Beton Indonesia (JBI) for testing compressive strength and also PT Growth Java, Cilegon Indonesia to provide nickel slag powder.

igh-magnesium_nickel_slag_powder_used_as_a_cement_suppl...

ORIGINALITY REPORT

13%	11%	14%	6%
SIMILARITY INDEX	INTERNET SOURCES	PUBLICATIONS	STUDENT PAPERS

PRIMARY SOURCES

1	Submitted to Aston University Student Paper	3%
2	P Siler, I Kolarova, J Bednarek, M Janca, J Masilko, R Novotny, T Opravil. "The effect of zinc, water to binder ratio and silica fume on the hydration and mechanical properties of Portland cement mixtures", IOP Conference Series: Materials Science and Engineering, 2019 Publication	2%
3	Bordy, Arthur, Akli Younsi, Salima Aggoun, and Bruno Fiorio. "Cement substitution by a recycled cement paste fine: Role of the residual anhydrous clinker", Construction and Building Materials, 2017. Publication	1%
4	iopscience.iop.org Internet Source	1%
5	trijurnal.lemlit.trisakti.ac.id Internet Source	1%

6 "ICCOEE2020", Springer Science and Business Media LLC, 2021 1%
Publication

7 Yoon-moon Chun, Peter Claisse, Tarun R. Naik, Eshmaiel Ganjian. "Sustainable Construction Materials and Technologies", Taylor & Francis/Balkema, 2020 1%
Publication

8 Mariane Audo, Pierre-Yves Mahieux, Philippe Turcry. "Utilization of sludge from ready-mixed concrete plants as a substitute for limestone fillers", Construction and Building Materials, 2016 1%
Publication

9 Jakob C Larsson, Carmen Vogt, William Vågberg, Muhammet S Toprak, Johanna Dzieran, Marie Arsenian-Henriksson, Hans M Hertz. "High-spatial-resolution x-ray fluorescence tomography with spectrally matched nanoparticles", Physics in Medicine & Biology, 2018 1%
Publication

10 www.mdpi.com 1%
Internet Source

11 Hsiang-Ning Wu, Chia-Hao Chang, Yu-Ching Ni. "An X-ray fluorescence experimental method for photon-counting detector in 1%

computed tomography system", Journal of Instrumentation, 2024
Publication

Exclude quotes	On	Exclude matches	< 17 words
Exclude bibliography	On		