



DOI: 10.34910/MCE.109.9

## Effect of ferronickel slag in concrete and mortar

R. Suryaningrat Edwin\* , M. Kimsan , B. Pramono , F. Masud , R. Sriyani 

Department of Civil Engineering, Halu Oleo University, Anduonou Kendari, Indonesia

\*E-mail: [romy.edwin@uho.ac.id](mailto:romy.edwin@uho.ac.id)

**Keywords:** ferronickel slag, compressive strength, fine aggregate, cement replacement, heat treatment, pozzolanic activity

**Abstract.** Cement and nickel mining industry is facing challenges such as the depletion of natural resources and insufficient landfill disposals of ferronickel slag. These problems can be solved by upgrading the ferronickel slag in concrete production without loss of quality. In this research, the use of ferronickel slag as fine aggregate and cement replacement in concrete and mortar was investigated. The ferronickel slag was ground using a ball mill to achieve two levels of fineness. The performance of mortar was assessed under heat treatment at 75 °C as a comparison to that of the normal curing. The pozzolanic activity of ferronickel slag was determined by the Frattini test. The results obtained showed that the strength of concretes increased with increasing of ferronickel slag content in the concrete mixture up to 40 %; beyond that the strength of concrete decreased. A positive effect on the compressive strength of mortar was achieved by using the slag with a higher fineness. The use of heat treatment at 75 °C enhanced the compressive strength of mortar. Assessment of the pozzolanic activity by means of the Frattini test indicates the non-pozzolanic reaction of the slag after 28 days. The use of heat treatment partially hydrated unhydrated cement grains.

### 1. Introduction

In recent decades, concrete has become the main building material in construction projects throughout the world. In comparison to steel structure, concrete is easier to produce and relatively low cost, since the raw materials such as fine aggregate (sand) and coarse aggregate (stone and gravel) can be easily found near the project location and it does not require a high technology in its production. Besides the aggregates, cement is the essential “binder” which occupies about 15 % in the concrete matrix. Since the estimation of concrete production achieves about 25 billion tons worldwide annually [1], which will also demand about 6.25 billion tons of fine aggregate [2] and 2.8-4 billion tons of cement [3]. These numbers imply that massive exploitation of natural resources is on-going, leading to environmental degradation in the near future. In addition, the production of Portland cement consumes an enormous amount of energy from fossil fuels such as petroleum, coal, and natural gas, which will not be replenished for millions of years. Hence, burning fossil fuels for clinker calcination and grinding in cement manufacturing release a huge amount of CO<sub>2</sub> into the atmosphere, which directly upsets Earth’s balance carbon budget, leading to faster global warming. At the same time, the mining industry is facing challenges such as insufficient landfill disposal of waste material and the danger of heavy metals and other harmful elements from waste material on water quality and environment. Therefore, upgrading the waste material in concrete production is key to solve these problems regarding environmental degradation.

The primary source of ferronickel in Indonesia is saprolite nickel ore. After excavation and stockpile, the ore is then treated in the rotary dryer and rotary reduction kiln for sulfurization. Afterward, the treated ore is transferred to the smelting process for extracting and purification. In the final stage, the ferronickel which contains about 80 % iron and 20 % nickel is generated, followed by its by-product or slag. In

Suryaningrat Edwin, R., Kimsan, M., Pramono, B., Masud, F., Sriyani, R. Effect of ferronickel slag in concrete and mortar. Magazine of Civil Engineering. 2022. 109(1). Article No. 10909. DOI: 10.34910/MCE.109.9

© Suryaningrat Edwin, R., Kimsan, M., Pramono, B., Masud, F., Sriyani, R., 2022. Published by Peter the Great St. Petersburg Polytechnic University.



This work is licensed under a CC BY-NC 4.0

Indonesia, approximately 4 million tons of ferronickel slag is produced yearly, which needs a large landfill for storage the slag. Besides, ferronickel slag is still categorized as a hazardous and toxic material which contains harmful elements such as Pb, Zn, As, Cd, Co, and Cu [4], which can affect human health. Hence, a possible breakthrough to solve such problems is utilizing the ferronickel slag as a raw material (fine aggregate or cement replacement) within the concrete production without loss of quality. Looking into literature, ferronickel slag was investigated by several researchers as fine aggregate replacement in concrete and mortar [5–7]. The use of ferronickel slag as partial cement replacement was also studied by [4, 8, 9]. While applying heat curing to mortar mixture containing ferronickel slag was only reported by Li et al. [10].

In the current research, the effect of ferronickel slag as fine aggregate replacement in strength and workability of concrete was studied. The influence of heat treatment on the compressive strength of mortar containing ferronickel slag as the supplementary was also evaluated.

## 2. Materials and methods

### 2.1. Aggregates

Aggregates used in this study were purchased from Indonesian Stone Crusher Manufacturer. In Indonesia, aggregates that are used in concrete production are generally natural aggregates. The fine aggregate can be found in the river basin with a round texture and smooth surface. The gravel used in this study is obtained from the erosion of rocks, which is crushed using a stone crusher to obtain the desired gradation. The particle size distribution (PSD) and physical properties of aggregates are shown in Fig. 1 and Table 2.

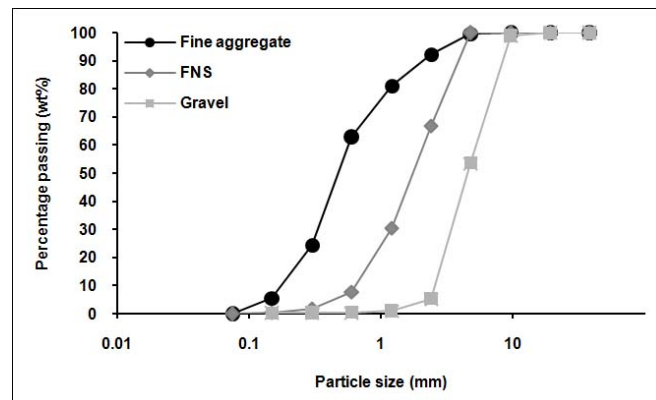


Figure 1. Particle size distribution of aggregates and FNS determined by sieving.

### 2.2. Ferronickel slag and cement

Ferronickel slag (FNS) used in this study is a by-product obtained from FeNi-IV Smelter Plant of one nickel mining company in Indonesia. This plant generates nickel ore and solid-granulated FNS. The slag has an angular particle with varying sizes and less porosity. As cement, an Ordinary Portland Cement I (OPC I) was used throughout all experiments. The physical properties and chemical compositions of OPC I and FNS are given in Fig. 1, Table 1 and 2.

Table 1. Physical characteristics of aggregates, OPC and FNS.

Test	Fine aggregate	Gravel	OPC	FNS
Specific gravity (g/cm <sup>3</sup> )	2.62	2.37	3.15	2.93
Water absorption (%)	1.43	0.89	–	0.50

**Table 2. Chemical compositions and fineness of OPC and FNS.**

Constituents	OPC	FNS
SiO <sub>2</sub>	19.9	53.6
Al <sub>2</sub> O <sub>3</sub>	5.3	5.5
CaO	64.1	5.2
Fe <sub>2</sub> O <sub>3</sub>	3.0	12.7
MgO	2.4	20.9
SO <sub>3</sub>	1.9	0.2
MnO	ND	0.5
K <sub>2</sub> O	0.6	0.1
Na <sub>2</sub> O	0.2	ND
Cr <sub>2</sub> O <sub>3</sub>	ND	1.2
Blaine permeability (cm <sup>2</sup> /g)	3350	-

### 2.3. Grinding process and particle size distribution

Before using the FNS as a cement replacement, the slag has to be ground to reduce the size within the micron meter. In this study, at first, the FNS was ground for one hour in a ball mill machine using 12 balls. Afterward, the FNS was sieved using a sieve with a size of 1.18 mm. The sieved FNS was then ground using a ball mill machine with 24 balls for 1 hour and 2 hours to achieve two levels of fineness. The FNS finer 1 (FNS\_F1) and the FNS finer 2 (FNS\_F2) is the ground FNS for 1 hour and 2 hours, respectively.

After the grinding process, the FNS and OPC were measured their fineness using laser diffraction (LD). A wet method was chosen to measure the PSD of binders. As a dispersant, isopropanol was used instead of water in order to confirm that no hydration occurred during this measurement between binder and dispersant. An outline of the optical parameters used in this study is presented in Table 3.

**Table 3. Outline of the optical parameters used to determine the PSD of binders by LD.**

Optical parameters	OPC	FNS_F1	FNS_F2
Absorption coefficient [-]	0.003	0.500	0.500
Refractive index [-]	1.730	1.530	1.530
Dispersant [-]	1.390	1.390	1.390
Obscuration [%]	12.990	12.570	12.690
Stirrer rate [rpm]	1500	1500	1500
Sonication times (minutes)	5	5	5

### 2.4. Superplasticizer

In this research, a new generation of modified polycarboxylic ether (PCE) based superplasticizer (MasterGlenium Sky 8851) was used throughout all experiments. This superplasticizer has long side chains, which initiates some electrostatic dispersion mechanism at the beginning of the mixing process to separate the cement particles. In this mechanism, the slump life and the desired workability at a low water-to-cement ratio is obtained.

### 2.5. Mixtures proportions and mixing procedure

In this study, the mix design of high strength concrete (HSC) is based on Indonesian Standard [11]. This standard can be used for concrete mix design with a water-to-cement ratio of less than 0.5. In the current research, a water-to-cement ratio of 0.35 was chosen. FNS was used as a fine aggregate replacement with the proportions of 0 %, 10 %, 20 %, 30 %, 40 %, 50 % and 60 % by weight. Due to the different specific gravity between fine aggregate and FNS, in which FNS is heavier than fine aggregate, the total volume of concrete containing FNS will decrease. Therefore, other materials such as cement, coarse aggregate, and water will compensate to obtain 1 m<sup>3</sup> of the total volume of concrete containing FNS. The detail of concrete composition is shown in Table 4.

To study the effect of FNS as cement replacement, the mix design of high strength mortar (HSM) was prepared with FNS contents from 0 to 20 wt% in steps of 5 wt%. Similar to concrete, a water-to-cement ratio of 0.35 was also chosen for HSM in this study. The mortar compositions used in this study is shown in Table 5.

In the mixing process of concrete, a 70 liter intensive mixer with two stirring plates and a speed of 30 rpm was used in this study. The mixing process of concrete was seen in Fig. 2. For mortar mixing, a 5

liter intensive mixer with 5 speeds was used. The mixing procedure of mortar used in this study was based on [12].

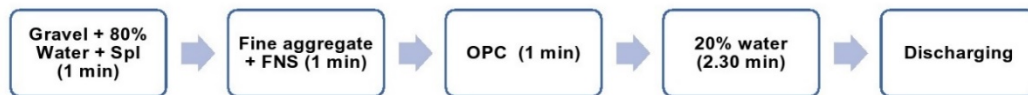


Figure 2. Mixing procedure of concrete.

Table 4. Mix composition of concrete with FNS as fine aggregate replacement ( $\text{kg/m}^3$ ).

Material	Ref	10%	20%	30%	40%	50%	60%
OPC	449.0	449.1	449.2	449.3	449.4	449.5	449.6
Fine aggregate	576.0	518.6	461.1	403.6	346.1	288.5	230.8
FNS	0.0	57.6	115.2	172.9	230.6	288.4	346.1
Coarse aggregate	1117.0	1117.5	1117.9	1118.4	1118.9	1119.4	1119.8
Water	157.0	157.4	157.7	158.1	158.5	158.9	159.2
Superplasticizer	9.9	9.9	9.9	9.9	9.9	9.9	9.9

Table 5. Mix composition of mortar with FNS as cement replacement ( $\text{kg/m}^3$ ).

Material	Ref	5%	10%	15%	20%
OPC	864.2	821.0	777.8	734.6	691.3
FNS	0.0	43.2	86.4	129.6	172.8
Fine aggregate	1108.9	1108.9	1108.9	1108.9	1108.9
Water	302.3	302.2	302.1	302.1	302.0
Superplasticizer	4.3	4.3	4.3	4.3	4.3

## 2.6. Heat treatment

In this study, after curing in room temperature for 24 h, the mortar samples were demolded and placed on the grid in the concrete curing box. The maximum temperature was set at 75 °C with a heating rate of 0.2 °C/min. Based on the literature, heat curing accelerates the hydration heat and simultaneously lead the porosity [13, 14]. The latter is due to the difference in the coefficient of thermal expansion of mortar ingredients [15]. Other literature reported that heat curing at higher temperatures also gives temporary-relaxation effect on strength at 28 days, which would again increase after longer curing periods as reported by [16]. So that, in this study a 75 °C was chosen to prevent the negative effect on the porosity and strength of mortar.

## 2.7. Frattini test

The Frattini test was used in this study to evaluate the pozzolanic activity of FNS. The procedure of this test was based on [17]. 20 grams of binder (cement + FNS) and 100 ml of distilled-decarbonized water were mixed in a polyethylene container. The carbonized water was prepared by boiling the distilled water to remove the carbon dioxide. Similar to the mortar composition as shown in Table 5, the FNS content varied from 0 wt% to 20 wt% of the binder in steps of 5 wt%. After curing in an oven at  $(40 \pm 2)$  °C for 8 days and 28 days, the solution was then vacuum-filtered using Whatman filter paper. To analyze the filtered solution, the acid-base titration was performed to determine the hydroxyl ion  $[\text{OH}^-]$  concentration and calcium oxide concentration  $[\text{Ca}^{++}]$ . The pozzolanic activity of FNS can be assessed if they are below the lime saturation curve [18].

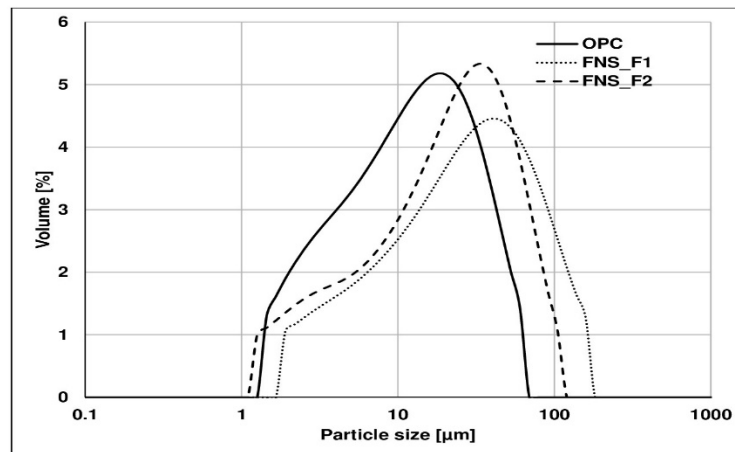
## 2.8. Workability and compressive strength

The workability of fresh concrete was determined using an Abrams cone with the following dimensions: diameter of the base ( $200 \pm 2$  mm); diameter of the top ( $100 \pm 2$  mm), and height ( $300 \pm 2$  mm) according to [19]. For fresh mortar, the flowability was measured using a mini-slump flow based on [20]. The slump test was conducted in a triplicate to evaluate the workability of concrete and mortar containing FNS as fine aggregate and cement replacement, respectively.

After curing in a water bath with a temperature of  $25 \pm 2$  °C, the concrete cylinder (100 mm diameter  $\times$  200 mm length) and mortar cylinder (55 mm diameter  $\times$  110 mm length) were taken out and then left at ambient temperature for two hours. The cylinders were tested in triplicate to evaluate the compressive strength. A compression machine with a capacity of 150 tons-force was used.

### 3. Results and Discussion

#### 3.1. Particle size distribution of different binders



**Figure 3. Particle size distribution of OPC and FNS.**

**Table 6. Specific gravity and  $d_{50}$  of the different binders.**

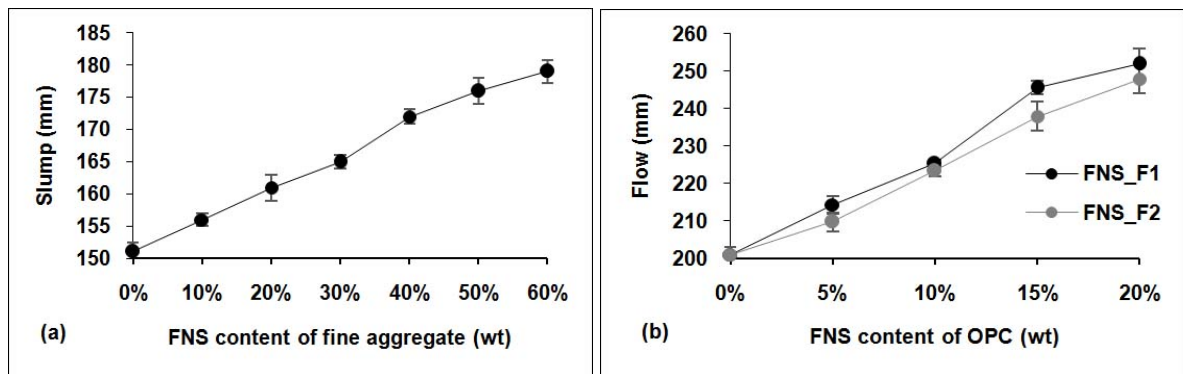
Materials	OPC	FNS_F1	FNS_F2
Specific gravity ( $\text{g/cm}^3$ )	3.151	3.107	3.107
Particle size $d_{50}$ ( $\mu\text{m}$ )	11.392	23.713	18.703

The results of the PSD of OPC and FNS determined by the wet method using laser diffraction was shown in Fig. 3. It seemed that the PSD of OPC was finer than FNS\_F1 and FNS\_F2. Meanwhile, the grain size distribution of FNS\_F2 was finer than FNS\_F1. By comparing the  $d_{50}$  of FNS as seen in Table 6, the FNS\_F2 which was ground for the longer time obtained finer particle size (18.703  $\mu\text{m}$ ) compared to FNS\_F1 (23.713  $\mu\text{m}$ ).

#### 3.2. Workability

The results of the workability of concrete and mortar are shown in Fig. 4. It can be seen that the slump of fresh concrete and mortar increased as the increase of the FNS content. For concrete, it can be explained by the fact that the FNS has a low water absorption compared to fine aggregate, as seen in Table 1. This different water absorption could lead to surplus water content in fresh FNS concrete. In addition to the water absorption, the differences in physical properties such as particle size and particle shape of materials composing concrete also influence the workability of fresh concrete [7]. In the current research, the particle size of FNS is coarser than fine aggregate as shown in Fig. 1, also resulting in lower the specific surface area of FNS than fine aggregate, therefore it will lead to less amount of un-wetting area in the FNS concrete matrix. This phenomenon can increase the amount of water trap, generating in enhancement the excess water in the fresh concrete matrix, and in the end, the workability of fresh FNS-concrete rises.

The flowability of mortar containing FNS coarser is higher than FNS finer as shown in Fig. 4b. This result corresponds with the result obtained of concrete workability as mentioned above. Since the FNS\_F2 has a higher specific surface area than FNS\_F1, in the mixing process, the air cavities of fresh mortar significantly decreased, which was effectively filled by the fine particles of FNS\_F2. So that, the higher viscosity of fresh mortar is obtained and reducing the flowability of fresh-FNS\_F2 mortar compared to mortar containing FNS\_F1. This result is in accordance with the finding of Humad et al. [21], who also found a decrease in the flowability of mortar using finer slags due to the effect of higher specific surface area.



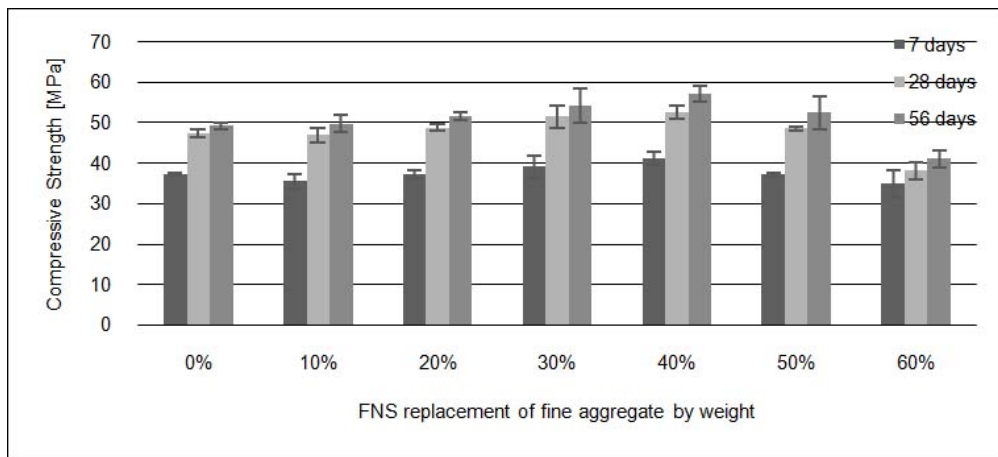
**Figure 4. Workability of: (a) concrete and (b) mortar (error bars represent standard errors; the average values represent three replicates).**

### 3.3. Compressive strength of concrete

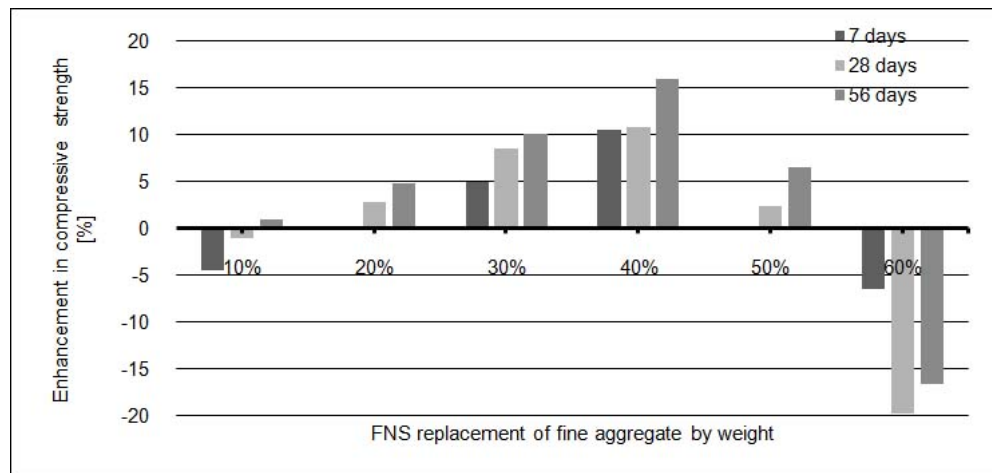
The development of strength of concrete containing FNS as fine aggregate replacement is seen in Figure 5. It can be seen that the strength of concrete increased with the increasing FNS content in the concrete mixture up to 40 %, beyond that the strength of concrete decreased. The highest compressive strength was achieved for 40 % FNS at later ages, which increased by about 16 % compared to the reference mixture, as shown in Fig. 5. This finding is in accordance with the finding of Saha and Sarker [7], who also found in an increase the compressive strength up to 50 % FNS and then decrease at a higher replacement level of FNS. This result can be explained by the fact that FNS has sharp angular edges and rough surfaces, which can improve cohesion and slip resistance between aggregate and paste. Besides, it is mentioned in the literature that fine aggregate has good abrasion properties due to its irregular surface [22]. However, this irregular surface will change with time to have a round surface due to the physical weathering. Since the hardness of FNS is similar to copper slag [23] and higher than the fine aggregate [24], a good abrasion of FNS is still obtained even though they are exposed in the open area for many years. Another reason is that the grain size of FNS is bigger than fine aggregate as seen in Fig. 1, which cannot effectively fill the concrete pores. Otherwise, the filler effect is obtained from the finer grain size of fine aggregate. So that, the combined fine aggregate-FNS grading at 40 % FNS gives the optimal packing fraction in the concrete matrix. For higher replacement level, the optimum packing fraction does not obtain due to the reduced the finer fraction from the fine aggregate which cannot be compensated by the grain size of FNS. This is the reason for the decreased compressive strength of concrete at 50 % and 60 % FNS substitution, as shown in Fig. 5 and Fig. 6.

Concerning the influence of water absorption on strength development, Sun et al. [6] noticed that the use of FNS as a sand replacement with higher water absorption gives a positive effect on the compressive strength of concrete. Regarding this, it seems that this current finding is in contrast with the findings of Sun et al. [6] since the water absorption of FNS is lower than the fine aggregate. However, it should be noted that the higher strength of concrete obtained in this study is only achieved at a lower replacement level. In addition, the excess of water in the fresh concrete matrix due to lower water absorption of FNS will leave more air cavities. This weakness might be compensated by the packing effect of FNS.

From Fig. 6, it can be observed that for early curing days (7 days), the concrete containing 10 % and 50 % FNS achieved slightly lower or similar strength compared to the control mixture, respectively. Compared to the result obtained of compressive strength at 7 days for 10 % and 50 % FNS, better results were achieved at longer curing periods (28 and 56 days), which was similar and slightly higher compared to the reference mixture, as seen in Fig. 6. It also can be observed that the strength enhancement for mixes 20 %, 30 %, and 40 % increased in the function of curing time as seen in Fig. 6. From these results, it can be noticed that strength enhancement of concrete up to 50 % FNS tends to be low at early curing periods (7 days) but relatively high at longer curing periods (28 and 56 days). This higher strength development at the later ages might be due to the increased interlocking of the aggregates combined with the filled porosity by the gel calcium-silicate-hydrate (CSH gel) generated during the hydration process.



**Figure 5. The strength evolution of concrete containing FNS as sand replacement (error bars represent standard errors; the average values represent three replicates).**

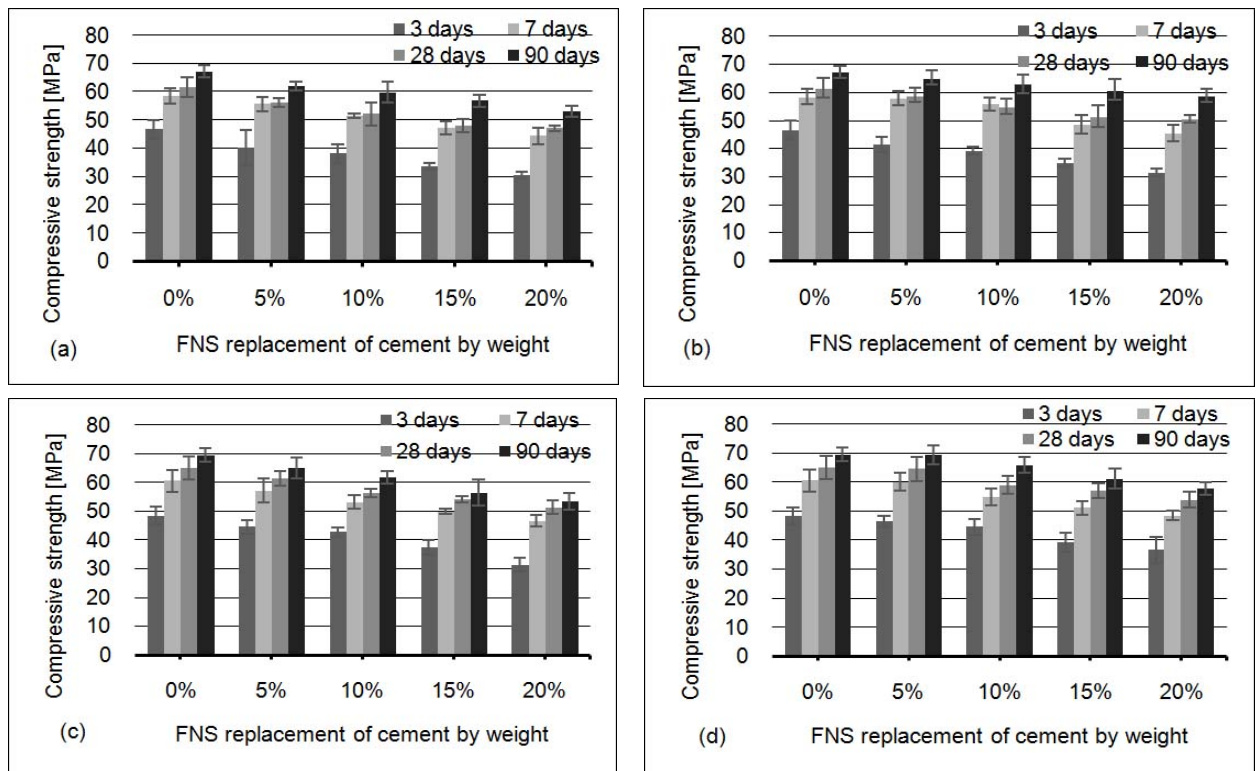


**Figure 6. Enhancement in strength (concrete containing FNS versus reference) at 7, 28, and 56 days.**

### 3.4. Compressive strength of mortar

In Fig. 7, the results of the compressive strength of mortar are presented. It is clear that the strength of mortar decreased as increasing the FNS content for all curing periods. At 3 days of normal curing, the compressive strength of mortar containing the lower replacement level of FNS\_F1 decreased by about 14 % compared to reference mixture, whereas the mortar mixture with 5 % FNS\_F2 showed a decrease by about 11 %. For normal curing at 90 days, it can be observed that the reduction of the compressive strength of mortar containing 5 % of FNS\_F2 and FNS\_F1 was about 3 % and 8 %, respectively compared to the reference mixture. These phenomena also occurred for the compressive strength of mortar after applying heat treatment at 75 °C. It can be seen in Fig. 7(c) and (d) that the mortar mixture with FNS\_F2 gave a smaller reduction in compressive strength in comparison to that of mortar with FNS\_F1 for both early and later curing periods. From these results, it can be concluded that the use of FNS as cementitious material gives a negative effect on the compressive strength of mortar. However, it should be noted that the relative strength enhancement of mortars mixed with FNS at later curing periods is higher than that of mortar FNS at early curing periods, indicating a gradually reducing the gap in strength between reference and mortar FNS. These phenomena were also reported by other researchers [4, 9]. The low reactivity of FNS is due to the low amount of Calcium content and inadequate fineness which does not speed up the reaction degree of cement hydration in the short period. The pozzolanic reactivity of FNS starts relatively late which will gradually consume the excess of  $\text{Ca}(\text{OH})_2$  from cement hydration to generate CSH gel.

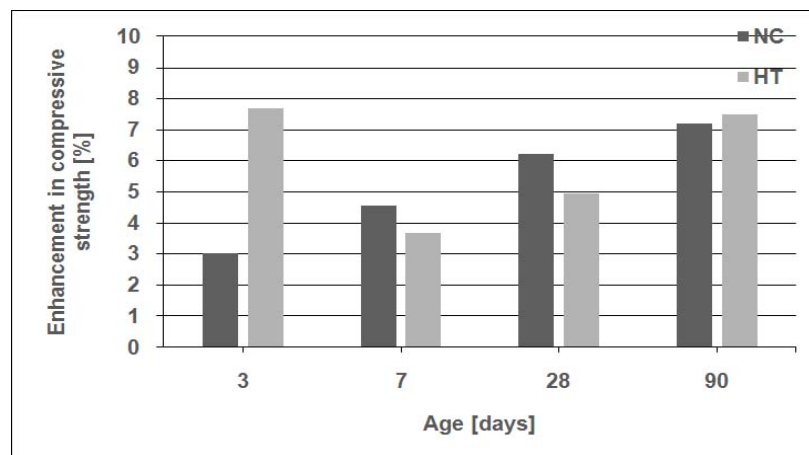
According to the methods explained above, the FNS was ground intensively to achieve two levels of fineness. Besides this, the heat treatment was applied to the specimens. So that, these aspects will be emphasized in the next sections.



**Figure 7. Mortar compressive strength results of difference fineness and treatment: (a) FNS\_F1 + NC, (b) FNS\_F2 + NC, (c) FNS\_F1 + HT, (d) FNS\_F2 + HT (error bars represent standard errors, the average values represent three replicates).**

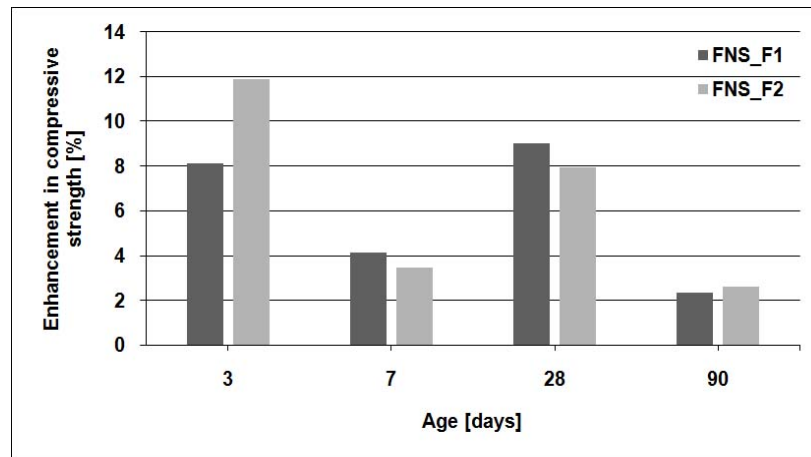
#### 3.4.1. Effect of fineness of FNS as cement replacement

The influence of the fineness of FNS as cement replacement on the compressive strength of mortar is shown in Fig. 8. It can be seen that the use of finer slag (FNS\_F2) enhanced the compressive strength of mortar for both NC and HT compared to coarser slag (FNS\_F1). It seems that a positive effect on the compressive strength of mortar is achieved by using the FNS with a higher fineness. These observations are in accordance with the findings of Kim et al. [8], who also found the positive effect of the finer FNS on the compressive strength of concrete. These results also confirm the finding of other researchers [25–29] even though they used different SCMs. This achievement might be due to the filler effect of finer FNS, which enhances the heterogeneous nucleation of cement hydration products. In addition to the filler effect, the higher fineness of FNS promotes the pozzolanic reactivity, which consumes more portlandite compared to the coarser FNS.



**Figure 8. The change in strength development of mortar by increasing the fineness of FNS for NC and HT (The values represent the average of all replacement levels of FNS).**





**Figure 9. Enhancement in strength of mortar (HT versus NC) for FNS\_F1 and FNS\_F2 (The values represent the average of all replacement levels of FNS).**

### 3.4.2. Effect of heat curing

The effect of heat curing on the strength enhancement of mortar for FNS\_1 and FNS\_2 is shown in Fig. 9. In general, the use of heat curing at 75 °C enhanced the compressive strength of mortar compared to that of normal curing for all curing days and both FNS\_F1 and FNS\_F2. It can be observed that a significant improvement of mortar strength with FNS\_F2 after applying heat curing was obtained at early curing days (3 days), which increased almost 12 % compared to mortar strength applied normal curing. The latter result is not the case for mortar with FNS\_F1, which achieved a higher strength development at later curing days (28 days) after applying heat treatment. For later curing periods (90 days), the use of heat curing seems beneficial in strength enhancement for both mortar FNS\_F1 and FNS\_F2 although this achievement is lower than for early curing periods as shown in Fig. 9. The positive effect of heat curing was also reported by other researchers [30–32]. They also found that heat treatment has a significant contribution to compressive strength at early age, which also corresponds to the result presented for mortar mixed with FNS\_F2. It seems that heat curing speeds up the hydraulic and pozzolanic reactivity of binders (cement + FNS\_F2) in the early curing periods, which force the FNS\_F2 to release more amount of hydroxyl ion ( $\text{OH}^-$ ) to consume calcium ion ( $\text{Ca}^{2+}$ ) from cement. In the case of FNS\_F1, a significant contribution of heat treatment in the reaction rate between a pozzolan and  $\text{Ca}^{2+}$  does not obtain due to the lower fineness of FNS\_F1.

### 3.5. Frattini test

Fig. 10 depicts the results of the Frattini test for 8 days and 28 days. From Figure 10(a), it can be seen that all of the samples cured for 8 days are above the non-pozzolanic zone. For longer curing times, all of the data are still located in the non-pozzolanic zone. The results obtained for 8 days and 28 days indicated non-pozzolanic activity of both FNS\_F1 and FNS\_F2. However, it can be observed that all of the data of FNS\_F2 at 28 days of curing move further and near the lime saturation curve as seen in Fig. 10(b). So that, it can be estimated that the pozzolanic activity of FNS determined by the Frattini test at 40 °C will be obtained for a curing day longer than 28 days. In literature, it is often mentioned that besides the chemical composition and the active phase content, a higher fineness also influences the rate of the pozzolanic reaction [33, 34]. This is the reason why the finer FNS used in this study consumed more amount of calcium ion ( $\text{Ca}^{2+}$ ) than the coarser FNS, which led all of the plotted data of FNS\_F2 to approach the saturation curve of lime as seen in Fig. 10(b). Hence, for future research, a longer grinding time combined with increasing the number of balls for FNS should be considered to behave as a pozzolan in a shorter period.

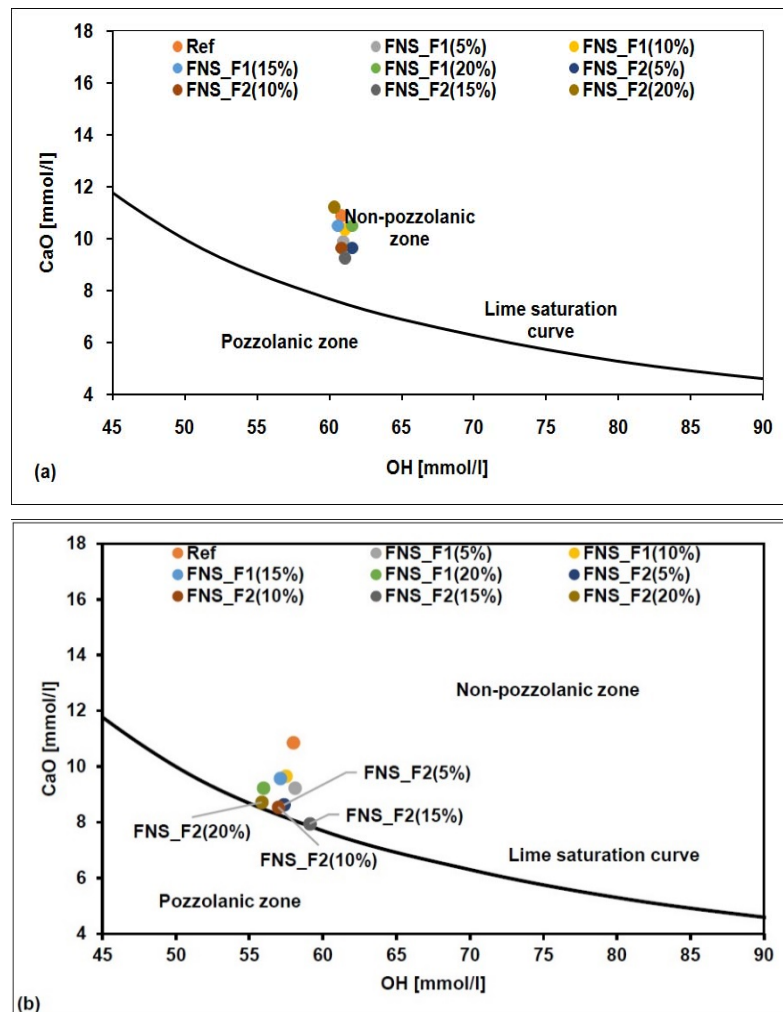
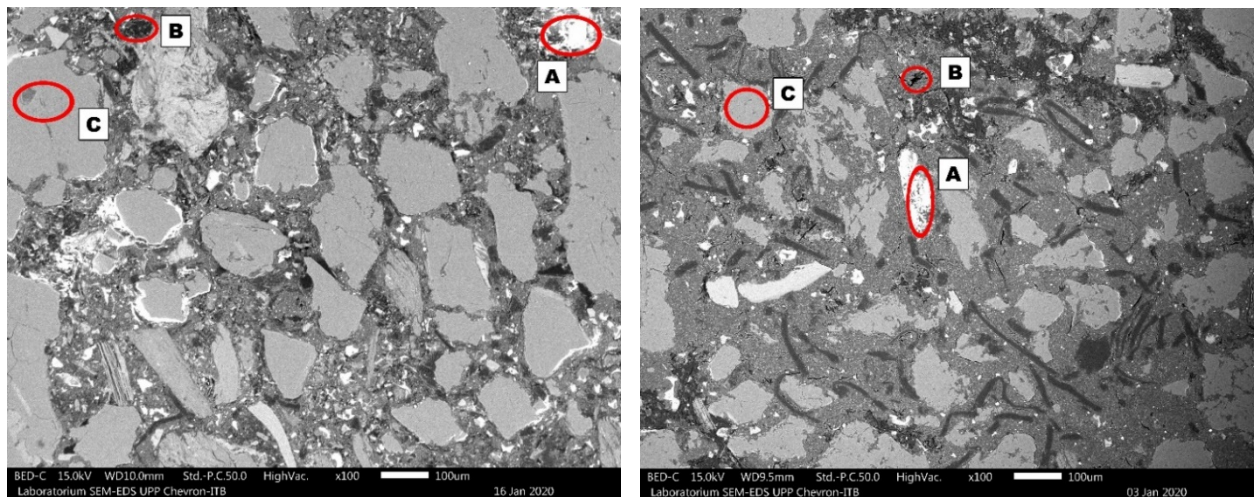


Figure 10. Frattini test results of FNS\_F1 and FNS\_F2 at: (a) 8 days and (b) 28 days.

### 3.6. Microstructure observation

To study the microstructure of mortar containing FNS, observation from literature is needed. According to [35–37], during hydration reaction, anhydrous cement grain reacts with water to generate hydration products such as CSH gel, ettringite, and calcium hydroxide (CH). While unhydrated cement grains (UCG) and pores still exist in the cementitious system in white and black parts, respectively. The UCG and pores can be clearly detected in this observation at a magnification of 100x as seen in Fig. 11, while CH and CSH need a microscope with a higher magnification (i.e. 350x).

For 10 % FNS\_F2 + NC at 28 days, hydration reaction continued in which UCG with the particle size lower than 20  $\mu\text{m}$  were encircled by thick rims of CSH and CH. The UCG with higher particle size tends to agglomerate and appearing between aggregates or surrounding aggregates as seen in Fig. 11(a). After applying heat treatment, UCG was partially hydrated, reducing the amount of UCG as visualized in Fig. 11(b). At young ages during the period of heat treatment, the UCG was partially hydrated to form the lighter CSH, while the darker CSH was gradually formed during the period of curing in water. At this phase, the amount of pore was reduced, filled by CSH, generating a denser paste.



**Figure 11. Visualization of the selected mortar mixture at 28 days by SEM-BSE: 10 % FNS\_F2 + NC (left) and 10 % FNS\_F2 + HT (right) (A = unhydrated cement grain; B = pore; C = fine aggregate).**

#### 4. Conclusions

In this study, the use of ferronickel slag (FNS) as a fine aggregate replacement and cement replacement was investigated. The following conclusions can be drawn based on the results obtained:

- The workability of fresh concrete and mortar increased as the increase of the FNS content. The higher specific area of FNS decreased the flowability of mortar. This is due to the filling effect of FNS finer in the air cavities, which increases the viscosity of fresh mortar.
- The use of FNS for up to 40 % fine aggregate replacement enhanced the compressive strength of concrete, beyond that the strength of concrete decreased.
- By increasing the fineness of the FNS, a higher compressive strength of mortar can be achieved for both normal curing and heat treatment.
- The use of heat treatment gives a positive effect on the strength development of mortar containing finer FNS or coarser FNS for all curing ages. However, for early curing days, a significant effect of heat treatment is only achieved for mortar containing FNS finer, while the effect of heat treatment is limited for mortar containing FNS coarser.
- The results of the pozzolanic activity assessed by the Frattini test show that all of the mixtures with FNS indicate no pozzolanic activity after 28 days of curing. However, the FNS finer consumes more portlandite than the FNS coarser.
- Based on the microscope observation, the use of heat treatment partially hydrated unhydrated cement grains.

#### 5. Acknowledgements

The authors would like to thank the Ministry of Research, Technology and Higher Education of the Republic of Indonesia for providing the financial support to this research project (National Competitive Grant 2019-2021 with contract number: 171/SP2H/LT/DRPM/2019). The authors would also like to acknowledge Research and Development Strengthening Agency of the Ministry of Research, Technology and Higher Education of the Republic of Indonesia for Grant for participation in a conference abroad with contract number: B/2795/E5.3/KI.03.01/2019. Special thanks are addressed to the Construction and Material Laboratory at Halu Oleo University (staff and student) for their contribution to this research.

#### References

1. Klee, H. The Cement Sustainability Initiative: Recycling Concrete. World Business Council for Sustainable Development. 2009. (July).
2. Edwin, R.S., Ngii, E., Talanipa, R., Masud, F., Sriyani, R. Effect of nickel slag as a sand replacement in strength and workability of concrete. IOP Conference Series: Materials Science and Engineering. 2019. 615(1). DOI: 10.1088/1757-899X/615/1/01-2014
3. Schneider, M., Romer, M., Tschudin, M., Bolio, H. Sustainable cement production-present and future. Cement and Concrete Research. 2011. 41 (7). Pp. 642–650. DOI: 10.1016/j.cemconres.2011.03.019.

4. Katsiotis, N.S., Tsakiridis, P.E., Velissariou, D., Katsiotis, M.S., Alhassan, S.M., Beazi, M. Utilization of Ferronickel Slag as Additive in Portland Cement: A Hydration Leaching Study. *Waste and Biomass Valorization*. 2015. 6 (2). Pp. 177–189. DOI: 10.1007/s12649-015-9346-7
5. Saha, A.K., Sarker, P.K. Expansion due to alkali-silica reaction of ferronickel slag fine aggregate in OPC and blended cement mortars. *Construction and Building Materials*. 2016. 123. Pp. 135–142. DOI: 10.1016/j.conbuildmat.2016.06.144.
6. Sun, J., Feng, J., Chen, Z. Effect of ferronickel slag as fine aggregate on properties of concrete. *Construction and Building Materials*. 2019. 206. Pp. 201–209. DOI: 10.1016/j.conbuildmat.2019.01.187.
7. Saha, A.K., Sarker, P.K. Sustainable use of ferronickel slag fine aggregate and fly ash in structural concrete: Mechanical properties and leaching study. *Journal of Cleaner Production*. 2017. 162. Pp. 438–448. DOI: 10.1016/j.jclepro.2017.06.035.
8. Kim, H., Hong, C., Yong, K. Feasibility of ferronickel slag powder for cementitious binder in concrete mix. *Construction and Building Materials*. 2019. 207. Pp. 693–705. DOI: 10.1016/j.conbuildmat.2019.02.166.
9. Huang, Y., Wang, Q., Shi, M. Characteristics and reactivity of ferronickel slag powder. *Construction and Building Materials*. 2017. 156. Pp. 773–789. DOI: 10.1016/j.conbuildmat.2017.09.038.
10. Li, B., Huo, B., Cao, R., Wang, S., Zhang, Y. Sulfate resistance of steam cured ferronickel slag blended cement mortar. *Cement and Concrete Composites*. 2019. 96 (December 2018). Pp. 204–211. DOI: 10.1016/j.cemconcomp.2018.12.001.
11. BSN. SNI-03-2834-2000. Indonesia Standard for Mix Design of Normal Concrete 2000.
12. EN-196-1, Methods of testing cement – Part 1: Determination of strength. 2005, CEN: Brussels, Belgium. Work. 2007. (april). Pp. 0–90.
13. Kjellsen, K.O., Detwiler, R.J., Gjrv, O.E. Pore structure of plain cement pastes hydrated at different temperatures. *Cement and Concrete Research*. 1990. 20 (6). Pp. 927–933. DOI: 10.1016/0008-8846(90)90055-3
14. Edwin, R.S., Gruyaert, E., Dils, J., De Belie, N. Influence of Vacuum Mixing on the Carbonation Resistance and Microstructure of Reactive Powder Concrete Containing Secondary Copper Slag as Supplementary Cementitious Material (SCM). *Procedia Engineering*. 2017. 171. DOI: 10.1016/j.proeng.2017.01.367
15. Kjellsen, K.O. Heat curing and post-heat curing regimes of high-performance concrete: influence of microstructure and C-S-H compositions. *Cement and Concrete Research*. 1996. 2. Pp. 295–307.
16. Heinz, D., Ludwig, H.M. Heat treatment and the risk of DEF delayed ettringite formation in UHPC. *International symposium on ultra high performance concrete*. 2004. Pp. 717–730.
17. EN-196-5, Methods of testing cement – Part 5: Pozzolanic test for pozzolanic cement. 2005, CEN: Brussels, Belgium. 2005.
18. Donatello, S., Tyrer, M., Cheeseman, C.R. Comparison of test methods to assess pozzolanic activity. *Cement and Concrete Composites*. 2010. 32. Pp. 121–127.
19. BSN. SNI-1972-2008. Indonesia Standard for Slump Test of Concrete 2008.
20. EN-1015-3, Methods of test for mortar for masonry – Part 3: Determination of consistence of fresh mortar (by flow table).2006, CEN: Brussels, Belgium.2006.
21. Humad, A.M., Habermehl-Cwirzen, K., Cwirzen, A. Effects of fineness and chemical composition of blast furnace slag on properties of Alkali-Activated Binder. *Materials*. 2019. 12 (20). Pp. 1–16. DOI: 10.3390/ma12203447
22. Al-Jabri, K.S., Al-Saidy, A.H., Taha, R. Effect of copper slag as a fine aggregate on the properties of cement mortars and concrete. *Construction and Building Materials*. 2011. 25 (2). Pp. 933–938. DOI: 10.1016/j.conbuildmat.2010.06.090.
23. U.S. Department of Transportation – Federal Highway Administration Research and Technology. *User Guidelines for Waste and Byproduct Materials in Pavement Construction*, Publication Number: FHWA-RD-97-148. (1) 2016.
24. OBE, R.K.D., Brito, J. De, Mangabhai, R., Lye, C.Q. *Sustainable construction materials: Copper slag*. Woodhead Publishing, 2016.
25. Edwin, R.S., De Schepper, M., Gruyaert, E., De Belie, N. Effect of secondary copper slag as cementitious material in ultra-high performance mortar. *Construction and Building Materials*. 2016. 119. DOI: 10.1016/j.conbuildmat.2016.05.007
26. Sajedi, F. Mechanical activation of cement-slag mortars. *Construction and Building Materials*. 2012. 26 (1). Pp. 41–48. DOI: 10.1016/j.conbuildmat.2011.05.001
27. Khan, K., Amin, M.N. Influence of fineness of volcanic ash and its blends with quarry dust and slag on compressive strength of mortar under different curing temperatures. *Construction and Building Materials*. 2017. 154. Pp. 514–528. DOI: 10.1016/j.conbuildmat.2017.07.214
28. Liu, S., Zhang, T., Guo, Y., Wei, J., Yu, Q. Effects of SCMs particles on the compressive strength of micro-structurally designed cement paste: Inherent characteristic effect, particle size refinement effect, and hydration effect. *Powder Technology*. 2018. 330. Pp. 1–11. DOI: 10.1016/j.powtec.2018.01.087
29. Mirzakhosseini, M., Riding, K.A. Influence of different particle sizes on reactivity of finely ground glass as supplementary cementitious material (SCM). *Cement and Concrete Composites*. 2015. 56. Pp. 95–105. DOI: 10.1016/j.cemconcomp.2014.10.004
30. Zdeb, T. An analysis of the steam curing and autoclaving process parameters for reactive powder concretes. *Construction and Building Materials*. 2017. 131. Pp. 758–766. DOI: 10.1016/j.conbuildmat.2016.11.026
31. Urbonas, L., Heinz, D., Gerlicher, T. Ultra-High Performance Concrete Mixes with Reduced Portland Cement Content. *Journal of Sustainable Architecture and Civil Engineering*. 2013. 3 (4). DOI: 10.5755/j01.sace.3.4.4569
32. Helmi, M., Hall, M.R., Stevens, L.A., Rigby, S.P. Effects of high-pressure/temperature curing on reactive powder concrete microstructure formation. *Construction and Building Materials*. 2016. 105. Pp. 554–562. DOI: 10.1016/j.conbuildmat.2015.12.147
33. Snellings, R., Scrivener, K.L. Rapid screening tests for supplementary cementitious materials: past and future. *Materials and Structures/Materiaux et Constructions*. 2016. 49 (8). Pp. 3265–3279. DOI: 10.1617/s11527-015-0718-z
34. Mertens, G., Snellings, R., Van Balen, K., Bicer-Simsir, B., Verlooy, P., Elsen, J. Pozzolanic reactions of common natural zeolites with lime and parameters affecting their reactivity. *Cement and Concrete Research*. 2009. 39 (3). Pp. 233–240. DOI: 10.1016/j.cemconres.2008.11.008. URL: <http://dx.doi.org/10.1016/j.cemconres.2008.11.008>
35. Famy, C., Scrivener, K.L., Atkinson, A., Brough, A.R. Effects of an early or a late heat treatment on the microstructure and composition of inner C-S-H products of Portland cement mortars. *Cement and Concrete Research*. 2002. 32 (2). Pp. 269–278. DOI: 10.1016/S0008-8846(01)00670-6

36. Gallucci, E., Zhang, X., Scrivener, K.L. Effect of temperature on the microstructure of calcium silicate hydrate (C-S-H). *Cement and Concrete Research*. 2013. 53. Pp. 185–195. DOI: 10.1016/j.cemconres.2013.06.008.
37. Scrivener, K.L. Backscattered electron imaging of cementitious microstructures: Understanding and quantification. *Cement and Concrete Composites*. 2004. 26 (8). Pp. 935–945. DOI: 10.1016/j.cemconcomp.2004.02.029

**Contacts:**

*Romy Suryaningrat Edwin, romy.edwin@uho.ac.id*

*Masykur Kimsan, masykur.kimsan@uho.ac.id*

*Bambang Pramono, Bambang.pramono@uho.ac.id*

*Fitriah Masud, fitriah.ecek@uho.ac.id*

*Rini Sriyani, rini.sriyani@uho.ac.id*