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Effects of nano-metakaolin on cement mortar and concrete properties Steve Wilben Macquarie Supit *a, Rilya Rumbayanb, Adriana Ticoaluc a,b Department of Civil Engineering, Manado State Polytechnic, Manado, Indonesia c Department of Civil Engineering, Sam Ratulangi University, Manado, Indonesia ABSTRACT This paper evaluates the effect of nano-metakaolin on compressive strength and durability properties of cement mortar and concrete. The Portland composite cement was replaced with 3%, 5% and 10% nano-metakaolin (by wt.). The compressive test results show that the optimum amount of nano-metakaolin in the mortar and concrete mixes is 10%.

There is also a great improvement on water sorptivity of concrete with nano-metakaolin. The result shows that concrete with nano-metakaolin 10% has sorptivity values of 6 and 5 (x10-4mm/min1/2) at 7 and 28 days, which is 76% and 68% lower when compared to the sorptivity of cement concrete. In this study, microstructure and phases identification results are also presented. Results show the effectiveness of nano-metakaolin in accelerating the pozzolanic activity by consuming calcium hydroxide (CH) and improving packing density.

Keywords: Nano-metakaolin, compressive strength, durability, microstructure, phase identification 1. INTRODUCTION The use of cement in concrete has raised concern of its sustainability, given the fact that the production of one tonne of Ordinary Portland Cement (OPC) releases approximately one tonne of carbon dioxide to the atmosphere, or in total roughly 6% of all man-made carbon emissions1. Therefore, it is important to reduce the consumption of OPC in concrete by incorporating industrial by-product materials which can be disposed at low economic and environmental costs.

This concern has led the use of various industrial by-products and pozzolan materials such as fly ash, blast furnace slag, silica fume, metakaolin, etc. as partial replacement of OPC in the concrete. Recently, the characterization of materials at the nanoscale has brought many ideas to study the properties of cement-pozzolan concrete due to the addition of nanoparticles. Based on some available results, the beneficial action of the nanoparticles on the microstructure and performance of cement based materials can be explained by some factors: 1) Nanoparticles contribute to formation of a dense structure of the aggregates' contact zone, resulting in a better bond between aggregates and cement paste2.

This matter can be originated from the crystallization effect of nanoparticles which can control the growth of CH (calcium hydroxide) and AFm (monosulphate) crystals and resulted in making the homogeneous matrix3. 2) Nanoparticles fill the voids between cement grains, resulting in the immobilization of “free” water2. This provides denser microstructure, reduces porosity and improves strength4. One of the advancement made on nano particles development in construction is the discovery that addition of metakaolin (in nano-size) can improve the mechanical properties of mortar and concrete.

The effect of incorporating nano- metakaolin to mortar and concrete on enhancing its properties are evaluated through some studies. From some conducted experiments, Morsy, et al.5 reported the increase of compressive and tensile strength of cement mortars was 49% and 8%, due to the addition of 8% nano-metakaolin. The results indicated that nano-metakaolin acts as a nano-fiber due to its morphology, and also an activator in promoting hydration process.

In cement concrete, it was found that the 28th day compressive strength increased around 30% at 10% replacement of cement with nano-metakaolin6. In another study, Shoukry, et al.7 reported the effectiveness of nano-metakaolin as a mineral additive in improving the microstructure and flexural strength of fiber reinforced cementitious composites. The study also found that the CH content of cement paste with 10% nano-metakaolin decreased to 42% due to the pozzolanic activity of nano-metakaolin and relatively remained constant when the nano-metakaolin replacement ratio was higher than 10%.

Despite the positive influence of micro and nano-metakaolin in mechanical properties of mortar and concrete, the effects of nano-metakaolin on water sorptivity, microstructural and phases identification analysis of Portland composite cement concrete is not intensively explored. *steve.macq@gmail.com; <mailto:%2Asteve.macq@gmail.com>phone 628134398901 In this study, the experimental investigation focuses on the compressive strength of cement mortar and concrete, and water sorptivity of cement concrete containing nano-metakaolin.

Additionally, microstructural and phase identification analysis of cement pastes with nano-metakaolin was conducted based on Backscatterd Electron (BSE) images, X-Ray Diffraction (XRD), and Brunauer-Emmett-Teller (BET) surface area analysis. 2. MATERIALS Portland Composite Cement, manufactured by PT. Semen Tonasa and Nano Metakaolin, denoted by PC and NK, were used as main materials in all mixes. Kaolin from Toraget Village in North Sulawesi Province in Indonesia was calcined at 800°C with constant temperature for 6 hours and then pulverized to nano-size around 300 nm by using High Energy Milling at CV. Nanotech Indonesia.

The characteristics of nano-metakaolin from Toraget Village based on X-Ray Diffraction (XRD) analysis is shown in Figure 1. It indicates that nano-metakaolin is amorphous material and is relatively less crystal hence makes a good synthetic pozzolan. Figure 1. X-Ray Diffraction of nano-metakaolin 3. EXPERIMENTAL PROGRAM This experiment is divided into four parts. In the first part, the optimum dosage of nano-metakaolin was determined based on the compressive strength development of cement mortar at 7 and 28 days. In this part, Portland composite cement was replaced by nano-metakaolin at 3%, 5% and 10% (by wt.).

In the second part, the optimum content of nano- metakaolin that exhibited high compressive strength in mortar was used to evaluate the compressive strength development of cement concrete at 3, 7 and 28 days. The third part evaluated the water sorptivity of cement concretes at 7 and 28 days, based on ASTM C1585 standard8. The fourth part analysed the microstructure and phase changes on cement paste containing nano-metakaolin based on Backscattered Electron image, X-Ray Diffraction and BET surface area analysis. Table 1 presents the mix proportions of mortar and concrete.

In the mixing process, the raw powder materials including cement and nano-metakaolin were weighed and dry mixed by hand until a homogeneous consistency was obtained. A superplasticizer from PT. Sika Indonesia was used to maintain the consistency and workability of mortar and concrete mixes where the dosage was calculated as a percentage by mass of total binders. Table 1. Mix proportions of mortar and concrete (kg/m3) Type of Mixes Mix Proportions of Mortar (kg/m3) Mix Proportions of Concrete (kg/m3) C NK S CA W C NK S CA W PC 450 - 1125 - 207 553 - 565 976 223 NK-5 427.5 22.5

1125 - 207 526 28 565 976 223 NK-10 405 45 1125 - 207 498 55 565 976 223 C=Cement; NK=Nano-metakaolin; S=Sand; CA=Coarse Aggregate; W=Water 4.

RESULTS AND DISCUSSION 4.1 Workability The workability of mortar and concrete with different percentage of nano-metakaolin is measured in terms of flow table value and slump value according to ASTM C14379 and ASTM C14310, respectively. The results show that the addition of nano-metakaolin decreased the workability of Portland composite cement mortar from 180mm to 145mm as the increase of nano-metakaolin content up to 10% (see Table 2). Table 2.

Workability of cement mortar and concrete with nano-metakaolin Type of Mixes Flow Table (mm) Slump value (mm) PC 180 75 NK-5 155 50 NK-10 145 30 Similar trend can be also observed in concrete where is the slump value of cement concrete decreases from 75mm to 30mm due to 10% nano-metakaolin addition. It can be explained that nano-metakaolin has a clay properties that increased the water absorption thus increased the water demand during mixing. Additionally, the lower workability values of mortar and cement concrete with nano-metakaolin is suggested due to the smaller particle size and higher surface area of nano-metakaolin thus requires more water to wet the particles surface. 4.2

Compressive strength The effect of nano-metakaolin on the compressive strength of cement mortar and concrete is shown in Figures 2 and 3. The results revealed that the compressive strength increased at all ages when nano-metakaolin was added into cement mixtures. It can be seen in Fig.2 that the compressive strength of mortar containing 10% NK exhibits higher strength when compared to control mortar and mortars with 3% and 5% NK.

As an example, the compressive strength of NK-10 mortar is increased from 14 to 31 MPa and 20 to 40 MPa at 7 and 28 days, respectively. The increase is about 121% at 7 days, and at 28 days the strength improvement is about 100% higher than PC mortar. An increase of strength development is also observed on mortars containing 3% and 5%, after 7 and 28 days of water curing. In concrete, the compressive strength due to 5% and 10% nano-metakaolin addition is also investigated. As shown in Fig. 3 the compressive strength of concretes containing NK increases with increase in NK content from 5% to 10%.

This confirms that the optimum dosage of NK in this mixes is 10% by weight of cement content, reaching the value of 29, 37 and 46 MPa at 3, 7, 28 days, respectively. The improvement in compressive strength is about 123% at 3 days, 85% at 7 days and 53% at 28 days, respectively. This is attributed to the liberation of free calcium hydroxide as a result of initial hydration from PC, the silica and alumina of NK. These compounds react to form additional calcium silicate hydrate together with formation of hexagonal calcium aluminate hydrate which are precipitated as soon as saturation is approached11,12.

The indication of more CSH gel results in improving the compressive strength and densification of microstructure of mortar and concrete. Figure 2. Compressive strength of cement mortars with nano-metakaolin at 7 and 28 days Figure 3. Compressive strength of cement concretes with nano-metakaolin at 3, 7 and 28 days 4.3 Water sorptivity According to the summarized sorptivity results on Table 3, the water sorptivity of concrete with 10% NK is 6 and 5 (x104mm/min1/2) at 7 and 28 days, respectively, which is 76% and 68% lower when compared to the sorptivity value of cement concrete. The similar trend is also found on concrete with 5% NK. This is an indication that the usage of nano- metakaolin in cement concrete densified the microstructure and increased the density of concrete.

In addition, the pozzolanic reaction of nano-metakaolin with free calcium hydroxide (CH) resulted in the creation of more CSH which fill the internal capillary pores and make the structure uniform, compact and dense13. Morsy et al.5 also reported that nano particles enhanced the interface between paste and aggregates. The results are also confirmed by microstructure analysis through backscattered scanning electron images showing the less pores initiated in cement paste containing nano-metakaolin (see section 5.1). Table 3.

Sorptivity value of different type of concretes after 7 and 28 days of curing Type of Mixes Sorptivity Value (x10-4mm/sec1/2) 7 days 28 days PC 25 16 NK-5 7 5 NK-10 6 5 5. MICROSTRUCTURAL AND PHASE IDENTIFICATION 5.1 Backscattered Electron Image Figure 4 shows the backscattered electron images of cement pastes with and without nano-metakaolin after 28 days of curing. The analysis was conducted through backscattered electron image using 10 micron magnification. The brightest (or white) parts can be classified as un-hydrated cement particles, the dark (or black) parts as voids and cracks and grey to dark grey parts can be classified in the order of CH, other hydration products and C-S-H14.

It can be observed that many white and black areas in cement paste sample indicating the un-hydrated cement particle and voids, respectively, whereas less pores can be observed in the sample containing 10% nano-metakaolin (Fig.4b). Large number of grey to dark areas which is composed mainly of amorphous hydration products around the remaining un-hydrated parts of cement grains in NK-10 sample indicating the hydration products and the consumption of CH hence triggering the strength development of cement mortar and concrete containing nano-metakaolin. Moreover, the nano-metakaolin particles fill the internal capillary pores and make the microstructure uniform, compact and dense. Figure 4.

Backscattered Electron images of (a) cement paste and (b) cement paste with nano-metakaolin at 28 days 5.2 X-Ray Diffraction The XRD analysis of cement paste with and without 10% nano-metakaolin at 7 and 28 days is shown in Fig. 5. In this study, the CH peak was considered to be the main indicator of performance in cement pastes samples. On the XRD scale, it can be noticed that CH has a strong peak located at 2-theta angle of 18?, 34°, and 47° and 50°, while the peak at 2-theta = 29° is related to CSH phase.

The XRD results show that the intensity of CH peaks in cement paste containing 10% NK was decreased compared to the intensity in cement paste without NK. The significant reduction can be seen in at 2-theta angle of 18.18° and 34.24° where the intensity of CH in cement paste at 7 days decreased from approximately 225 to 60 counts and 320 to 100 counts, respectively (see Fig. 5a). The same trend was also found in cement pastes at 28 days (Fig.5b), where the addition of 10% NK decreased the intensity of CH at 2-theta angle of 18.18° and 34.24° from 260 to 80 and 250 to 90, respectively.

As a product of cement hydration, CH reacts with silica in supplementary cementing system and forms CSH resulting in improved mechanical properties of mortars and concretes. (a) (b) Figure 5. XRD analysis of cement paste with and without nano-metakaolin at (a) 7 days and (b) 28 days 5.3 BET surface area Brunauer-Emmett-Teller (BET) theory was used to analyse the gas adsorption data and calculate the surface areas which is directly related to the high porous phase. Nitrogen was used as the adsorbate gas.

Table 4 shows the surface area and pore volume of each type of mixes while Fig.6 illustrates the cumulative pore volume versus the pore size. Evidently lower surface area means low porosity and cumulative pore volume can be seen on samples containing 10% nano- metakaolin after 7 and 28 days of curing. The surface area and pore volume of NK-10 paste at 7 days were 45.948 m2/g and 0.156 cc/g while at 28 days decreased to 42.420 m2/g and 0.13 cc/g (approximately 35% and 18% lower when compared with the surface area and pore volume of cement paste sample).

This indicates that the particle size distribution is modified due to the addition of nano-metakaolin thus refines the pore structure of cement paste. Table 4. Surface area and pore volume of cement pastes with 10% nano-metakaolin Type of Mix Surface Area (m2/g) Pore Volume (cc/g) PC-7 days 71.569 0.164 PC-28 days 55.276 0.160 NK10-7 days 45.948 0.156 NK10-28 days 42.420 0.130 (a) (b) Figure 6. Cumulative pore volume vs pore radius of cement paste with and without nano-metakaolin at (a) 7 and (b) 28 days 6. CONCLUSIONS The following conclusion can be drawn from the experimental results: 1.

The usage of nano-metakaolin in cement composites was effective in increasing the compressive strength at 3, 7 and 28 days, respectively, where the highest compressive strength was achieved by mortar and concrete containing 10% nano-metakaolin (by wt.). The improvement of compressive strength in Portland cement concrete with nano-metakaolin is approximately 123%, 85% and 53% higher compared to the compressive strength of control concrete after 3, 7 and 28 days of water curing. This indicates the positive influence of nano-metakaolin in promoting the hydration process thus improving the compressive strength at early and later ages. 2.

In term of water sorptivity, the addition of 5% and 10% nano-metakaolin has shown a positive contribution increasing the density of cement concrete. The result shows that NK10 concrete has sorptivity values of 6 and 5(x10-4mm/min1/2) at 7 and 28 days, respectively, which is 76% and 68% lower when compared to the sorptivity of cement concrete. 3. The results form BSE and XRD analysis confirm the influence of nano-metakaolin in improving the microstructure of cement pastes.

The pore structure of NK-10 paste is denser than control hardened paste as also confirmed by surface area and pore volume of NK-10 paste. The reduction of CH indicates the formation of more CSH in the system thus increase the compressive strength of nano-metakaolin mortar and concrete. Moreover, nano-metakaolin refines the pores structure thus increase the resistance of cement concrete on water sorptivity.

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