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Effects of Micro- and Ultra ?ne Metakaolin on Compressive Strength and Water Sorptivity of High Volume Fly Ash Concrete Steve W. M. Supit and Febriane Makale w Abstr act The experi mental study on the effect of using mic ro- a nd ultr a ? ne metak aolin incor porated with Portland cemen t compo site for the imp rovement of compr essive strength and water sorptivi ty of high volum e ? y ash conc rete is pre- sente d in this paper.

Mixture propor tion s was prepared where metakaoli n in micro- and ultra ? ne sizes was used to replace cemen t in high volume ? y ash concret e mixtures (40 and 60% by wt .). Ph ase ident i ? cation and the hydration product s were obtain ed throu gh X-ray Diffract ion (XRD) and Fo urier Transfor m Infra red Spectr oscopy (FTIR) analys is.

The resul ts reveal that the incl usion of micro- and ultra ? n e metakaoli n offset s the inferior charact eristics of high volume ? y ash concret e by acceler ating the h ydration product s, contr ibutes the formati on of additional CSH through the react ion with calcium hydroxi de and densi ? e s the microst ructu re of sust ainable high v olume ? y ash mat rix, particula rly at the 7th day after wat er curing.

Furthe rmor e, the utilizat ion o f high volum e ? y ash containing micro- and ultra ? ne met akaolin is po tential for the appli cation of eco-friendl y concret e. Keyword s Meta kaolin _ Hig h volum e ? y ash _ Compre ssiv e stre ngth _ Water sorptivi ty _ XRD _ FTI R 1 Introd uction The utilization of suppl ementary cemen titious material s (SCMs) in mortar and concret e has b ecome one of the major research areas in the last twenty years.

The ability to repla ce cemen t partially in mortar and concrete mixtures due to the silicon and alum inium as the mai n constitu ents, has brought ? y ash, silica fume , blast furnac e slag, and met akaolin as suppl ementary cemen titious materials to be S. W. M. Supit (&) _ F. Makalew Department of Civil Engineering, Manado State Polytechnic Manado, North Sulawesi, Indonesia e-mail: stevewmsupit@gmail.com F. Mohamed Nazri (ed.), Proceedings of AICCE'19 , Lecture Notes in Civil Engineering 53, https://doi.org/10.1007/978-3-030-32816-0_27 415 alternati ves materials for const ruction indus try.

W ith respec t to the envir onmen tal impac t, the use of SCMs source d from natur al resources and by-pro duct materials in reduci ng Portland cemen t conten t in concret e is worth to pursue . It contr ibutes the reduct ion of greenho use gas emissi ons to the earth ' s atm osphere from the de-carb onation of limest one in the kiln durin g Portland cemen t product ion wher e the cemen t industry contr ibutes approxi mat ely 5 – 7% to global antrh opogeni c car- bon dioxide emissi ons [9 , 16].

One of the supplement ary cemen titious mat erials that has been inte nsively inves tigated is ? y ash as a by-pro duct mat erial source d from the coal- ? red po wer station that is abundant ly avail able in most of the countr ies in the world. Fly ash has been recogni zed as a pozzol anic material that can be used as a min eral admixtur e or as a component to replace cemen t in concret e.

So me experi mental studies have reporte d the ben e ? cial effect of using ? y ash in produci ng sust ainable high per- forman ce mortar a nd concret e on mecha nical and durability proper ties. Tan gpagasit et al. [15] reports the incre ase of early-age of stre ngth activity index of mortar contai ning 20% ? y ash (by wt. of cemen titious mat erials).

Fly ash with medi an particles sizes of 2.7 and 160 μ m when mixe d in cemen t morta r reached 22% higher of stren gth compared to control samp le. In this study , the packin g effect of ? y ash was found more effective than the pozzol anic react ion in improvi ng the streng th of cemen t mortar. Celik et al. [3] also conclu ded that the parti cle size distrib ution of ? y ash is very critical facto r in contr ibuting the increase of the compr essive strength of mortar, and not the chemical compo sition. On the other hand, the type of ? y ash used in mortar or concret e is also imp ortant to achiev e suitable stre ngth.

Fo r example, Saka i et al. [10], studied the proper ties of two types of ? y ash consi sting different level of glass conten t which are 38.2 and 76.6%. It was found that the react ivity of ? y ash is controlle d by the glass conten t in ? y ash. Sampl es with high glass content wer e clari ? ed to h ave higher hydration rati o of ? y ash after curing for 270 days while almost identical values was found at an age of 360 days.

Despit e of some bene ? ts reported on the in ? uence of ? y ash in mortar and concret e, the limitatio n of using 15 – 20% by wei ght of cement conten t remains a probl em. Using this dosage u sually imp roves the wor kability to produce cost-effecti ve concret e but not suf? ciently effective on d urability enh ancement [7].

For this reason , the utilization of concrete with high volum e ? y ash (HVFA) for sustainabl e development h as been addres sed throu gh some research works. Experim ental study by Xu et al. [18] show ed the increase of carbona tion resistanc e of concret e due to the addition of 30 and 40% ? y a sh wi th wat er/binder ratio used was below 0.4.

The study found that the steel c orrosion resistanc e improved as meas ured from the steel weig ht loss ratio. Additi onally, incre asing the amoun t of ? y ash repla cemen t u p to 50% also exh ibited good steel corrosion resistanc e proper ties. Similar study from Moffatt et al. [8] also found the improvem ent of the chlor ide-ion penetrati on resistanc e in concret e with 56 and 58% ? y ash by wt .

Moreo ver, there was notable imp rovemen t in load carrying c apacity and cracki ng behavi or on mix tures containing 50% ? y ash [12]. For the applic ation, [4] conclu ded that the HVFA concrete when proper ly desig ned can be applied both 416 S. W. M. Supit and F. Makalew for stru ctural a nd non structura l applicati ons in c onstruction.

Despit e the extensive resear ch effort on the effect of ? y ash in cemen t compo sites, there is still a lack of unders tanding of compr essive strength and water sorptivi ty of high volum e ? y ash concret e wi th ultra ? ne metakao lin a ddition. This study aim s at inves tigatin g the e ffect ultr a ? ne-m etakaolin on compressi ve streng th development and water sorptivi ty resistanc e of Hi gh Volume Fly Ash (HVFA) concret e.

The results of HV FA concrete with ultra ? ne-metakao lin addition will be compa red with the results of concrete contai ning cemen t and micro-m etakao lin. In order to suppor t the ? ndings, microstruct ure analys is are also conduct ed and analys ed based on X-ray Diffraction (XRD) and Fourie r Tr ansform Infra red Spectr oscopy (FTIR) testing resul ts.

2 Mater ials Portlan d Compos ite Cem ent (PCC) , ? y ash (FA), mic ro- and ultra ? ne meta kaolin, ? ne and coarse aggrega te, and wat er are used in this ex periment . Class C of ? y ash is source d from the Amurang steam power plant whi le the metakaoli n from Toraget Villa ge, both locations are in Nor th Sulawesi Pr ovince, Indones ia.

The mic ro-metaka olin is o btained throu gh manua l grinding of metakaoli n after heatin g in ov en at temperat ure of 105 °C for 24 h while the ultra ? ne metakaoli n is produce d by grind ing met akaolin for a bout 2 h by using Hi gh Ene rgy Mil ling estab lished by CV. Nanotech Indonesia. The calcinati on of metakaoli n was con- ducted after re ? ning proces s at Bal ai Riset dan Standa rdisasi in Manado by using a ceram ic kilns furnac e for 6 h of heati ng perio d at a constant tem perat ure of 800 °C. Figure 1 shows the ima ge of ? y ash and metakaoli n used in this experiment .

Table 1 presents the chemical compo sition of each material based on the X-ray Fluore scence Anal ysis. The XRD spectra of ultra ? ne metakaoli n can be seen in Fig. 2 show ing the amor phous proper ties of the ultra ? ne met akaolin used in HVF A concret e mix tures. Fig.

1 Images of a class F ? y ash, b Metakaolin Effects of Micro- and Ultra ? ne Metakaolin … 417 Acco rding to the Pa rticle Size Anal ysis (PSA), the parti cle sizes of mic ro- and ultra ? n e metak aolin used in this experiment is around 10 and 196 nm, respec tively. 3 Exper imental Me thod The experiment al work is condu cted in Concret e Laborato ry at Manado State Polyt echnic, Indones ia.

At the ? rst part, concret e samp les wi th h igh volum e ash contai ning micro- and ultra ? n e metak aolin wer e cast ba sed on the mixture pro- portions as seen in Tab le 2 . After casti ng and curin g for 7, 14 and 28 days, the samp les wer e then tested for compressi ve strength follow ing the AST M C39 stand ard [2] procedu re.

In this part, concret e with 40% ? y ash by wt. was selected Table 1 Chemical composition of materials Chemical analysis PCC Fly ash Micro-metakaolin Ultra ? ne-metakaolin SiO 2 8.43 18.77 40.48 47.00 Al 2 O 3 1.65 6.89 31.17 32.00 Fe 2 O 3 4.81 21.8 0.87 3.43 CaO 73.12 28.13 1.20 2.53 MgO – 4.65 3.65 – K 2 O – 1.38 0.73 1.10 Na 2 O – 7.41 12.32 – SO 3 2.71 6.65 2.59 1.9 Fig. 2 X-ray diffraction of ultra ? ne-metakaolin 418 S. W. M. Supit and F.

Makalew as contr ol samp le to evalua te the in? uence of ulra ? ne-m etakao lin addition. The dosage of 10% repla cemen t of cemen t by mic ro- and met akaolin was used based on some previ ous study conduct ed by Su pit et al. [14]. From this study, samp les with 10% metakaoli n exhibited highes t stre ngth compa red to other dosage such as 5, 15 and 20%.

At the second part, the sorpt ivity value was evaluated by condu cting sorptivi ty test a ccording to the procedu re in AST M C15 85 standard [1]. The cut-sampl e with size of 50 mm in height and 100 mm in diam eter was prepar ed and suspen ded above water at speci ? c time inte rval. The respec tive cumul ative absorp tion values was obtained throu gh the wei ght gain measured at several time incre ment such as 1, 5, 10, 20, 30 min , 1, 2, 3, 4, 5 and 6 h. Afterwards , the sorptivi ty value c an be obtained as the slope of the line that best ? ts the plot.

In order to evalua te and charact erize the cryst alline phases that form during hydration of the blended cemen ts, the XRD and FTIR analys is are also conduct ed for sample after water curing at the 7th day. 4 Result s and Discussions 4.1 Effect of Micro - and Ultra ? ne-metakaolin on Workability of HVFA Concrete The workabil ity of samples contai ning micro and ultra ? ne-m etakao lin is meas ured based on the slump test with the resul ts are presen ted in Fig. 3 . The results show that the u se of high volum e ? y ash (40 and 60% by wt.)

incre ase the slump value of the specimens compa red to the contr ol PCC concret e due to the spheri cal shape particles that is possible to incre ase ? ow. However , the addition of micro- and ultr a ? ne metakaoli n ca n reduce the slump value from 7. 5 to 5.5 mm for the FA30. MK10 sample wi th a lowe r value up to 4 mm when the ultra ? n e met akaolin is added into the mix tures.

In the concrete mix ture containing 60% replacemen t of ? y ash, the sim ilar trend of slump reduction can be also Table 2 Mixture proportions of cement concrete with different percentage of ultra ?ne-metakaolin (kg/m 3) Mix PCC HVFA MK UM FA CA W PCC 400 – — 724 1131 195 FA40 240 160 — 724 1131 195 FA60 160 240 — 724 1131 195 FA30.MK10 240 120 40 – 724 1131 195 FA50.MK10 160 200 40 – 724 1131 195 FA30.UM10 240 120 – 40 724 1131 195 Note: PCC Portland composite cement; HVFA high volume ?y ash; MK metakaolin; UM ultra ? ne-metakaolin; FA ? ne aggregate; CA coarse aggregate; W water Effects of Micro- and Ultra ? ne Metakaolin … 419 observ ed. The slump value is decreas ed from 8.5

to 6 mm after adding 10% micro-m etakao lin into concrete with 50% ? y ash as a cement replacemen t. Thi s can be explai ned that the use of met akaolin wi th its high reactivity, ? ner and irregular shape having mul tiple layer stru cture consumes water very early and increases the cohesi veness of concrete [5]. 4.2

Effect of Micro - and Ultra ? ne-metakaolin on Compressive Strength of HVFA Concrete The compr essi ve strength develo pment of HVFA concret es co ntaining micro- and ultra ? n e-metakaol in is show n in Fig. 4 . The compr essive strength results due to different sizes of metakaoli n addit ion are compa red with the resul ts of co ntrol concret e mixes with cemen t (PC C) and ? y ash only (F A40 and FA60).

Based on the ? gure, it can be seen that concret e sample contai ning 30% ? y ash and 10% micro-m etakao lin had higher compr essi ve strength at 7, 14 and 2 8 days, respec tively, compared to FA40 sample. The compre ssive streng th reached the value of 10, 1 3 and 16 MPa at 7, 14 and 28 days, whi ch are approxi mately 18 – 40% higher than the results of FA40 concret e.

The higher per- centag e c an be found at the age of 7 days indicating the in ? uence of micro-m etakao lin in incre asing the early -age compr essive strength of concret e contai ning ? y ash and cemen t only. The effecti veness of mic ro-metaka olin in high volum e ? y ash conc rete can be also o bserved in FA50- MK10 samp le.

However , the compr essive strength resul ts are slightl y lower when the repla cement c ontent is incre ased up to 60% (50% ? y ash a nd 10% mic ro-metaka olin) by wt . of ce ment. Since the maximum compr essive stre ngth of HVFA concrete was found in FA40 mix, the effect of 10% ultra ? ne-m etakao lin is evalua ted to repla ce cement and Fig.

3 Slump value of different type of concrete mixtures 420 S. W. M. Supit and F. Makalew combi ned with 30% ? y ash. Figure 3 show s that there is a signi ? cant imp rovement on compr essive strength resistanc e when 10% ultra ? ne met akaolin was invol ved into the mix. The compr essive strength resul ts of FA30-UM 10 concret e wer e found to be 15 , 21 and 23 MP a at 7, 14 and 28 days, respec tive ly.

These resul ts are even higher than the resul ts obtained by contr ol concret e (PCC) at the selec ted d ays. The high speci ? c surfa ce area of ultr a ? ne met akaolin is sugges ted bene ? ts in acti ng as a ? ller an d reducing the calciu m ions from the vicin ity of the cement particles , there fore, acceler ates the hydrat ion proces s and increases the amount of CSH that can compe nsates the loss of early strength of HVFA concret e [17]. 4.3

Effect of Micro - and Ultra ? ne-metakaolin on Water Sorptivity of HVFA Concrete The typi cal plot s of absorp tion rate agains t the square root and sorpt ivity values of HVFA concret e samp les containing 1 0% micro- and ultra ? ne-m etakao lin a t 7 and 28 days are presen ted in Fig. 5 a, b a nd Table 3 , respec tively. Concret es with PCC and 40% ? y ash are selec ted as control samp les to eva luate the effectiven ess of using 10% micro- and ultra ? ne met akaolin in HVFA concrete mix ture. In Fig.

5 a, b, it can be seen the typical plot s of cumulati ve water a bsorption taken from the two samp les from each type of mixture tested at the 7th and 28th day with correl ation Fig. 4 Compressive strength development of different type of concrete mixes Effects of Micro- and Ultra ? ne Metakaolin ... 421 coef? cient s great er than 0.98.

Bas ed on the ? gures, the wat er absorp tion of HVFA concret es at the 7th and 28th d ay reduces due to the incor poration of micro- and ultra ? n e met akaolin. The u se of ultra ? ne met akaolin in FA40 concret e signi ? cantly decreas es the sorptivi ty values from 241 to 123 (_ 10 - 4 mm /s 1/2) at 7 days and 225 to 118 (_ 10 -4 mm /s 1/2) at 28 days, as seen in Tab le 3 . These values are also much lower when compa red to the concret e mixture with PCC only.

In compa rison to the concret e sample wi th 10% mic ro-metaka olin , the sorptivity value of concret e contai ning 10% ultra ? ne metakaoli n is around 40 – 48% lower after the curin g days. This less water absorp tion indi cates the high pozzol anic react ion and ultra ? ne pore ? ller effects of ? y ash combined with ultra ? n e-metakaol in.

[9], also found a sim ilar results wher e the presen ce of met akaolin as a much thinner and reactive mat erial compa red to ? y ash, decreas ed the large pores and provided more compa ct matrix and therefore less permeable mixtures . Fig. 5 Water sorptivity results of different type of concrete mixes at a 7 days and b 28 days 422 S. W. M. Supit and F. Makalew 4.4

X-ray Diffraction Analysis Figure s 6 a – d show the phase compo sition that forms du ring hy dration of the paste samp les containing 10 % micro- and ultra ? ne met akaolin combined with 30% of ? y ash. Since the inves tigation is focuse d on the in ? uence of ultra ? ne-metakao lin at the early -age charact eris tics of the sample, there fore, the polished paste samples were tested after water curing at the 7 th day o nly.

In the XRD patt erns, it can b e seen that the mai n peak form ed are Quartz, Calcite , Calciu m Silicate (CS) and Calcium Hydroxid e (CH) . The peak intensity of CH at 2-the ta angle of 18° and 34° can be an indi cator to explain the hydration perfor mance of all type of paste samples. However , the peak of CSH is overlap with Calcite , therefore, its presen ce cannot be identi ? ed clear ly and needs further analys is to quantitat ively deter mine the percentage amoun t of Cal cite and CSH phases .

Based on the ? gure, the formation of Calciu m Silicate Hydr ate can be observ ed in samp les with 40% ? y ash (FA40) and combined 30% ? y a sh and 10% micro- or ultra ? ne met akaolin (FA3 0.MK10 and FA30. UM 10). Regardi ng to the presen ce of CH, the intensity p eaks of CH in FA40 paste samp les wer e found lower than in PCC samp le (see Fig. 6).

The inte nsities were even much lower when micro- and ultr a ? ne-me takaolin were added to parti ally repla ce cemen t in 30% ? y ash paste samples. When the inte nsity peaks of CH in FA30. 10UM sample is compa red with FA30.MK 10, it can be noticed that the presen ce of CH at 2-the ta angle of 18° in samp le wi th ultra ? ne met akaolin was disap peared indi cating that the ? ner size of metakaoli n bene ? ts in accelerati ng the consum ption of CH thus incre ased the inten sity of CSH after curing at the 7th day.

Shaikh and Supit [13] also commente d that the silicat e conten t in suppl ementary cemen ting system has an ability to react with CH thus reduces the inte nsity counts of CH and form additional CSH for the imp rovement of cemen t paste binder s proper ties. Table 3 Sorptivity value of various concretes containing micro- and ultra ?ne metakaolin Type of mixes Sorptivity value (_10 -4 mm/s 1/2) 7 days 28 days PCC 280 104 FA40 247 225 FA30.MK10 241 203 FA30.UM10 123 118 Effects of Micro- and Ultra ? ne Metakaolin … 423 4.5

Fourier Transform Infrared Spectroscopy (FTIR) Analysis Figure 7 presen ts spect ra o f FTIR of the b lended cemen t paste s at 7 days wher e the different compo sition in e ach type of paste can be evalua ted based on the peaks arising at different wavenumb ers wi th different intensities . Based on some studi es, the hydroxy l vibra tion s of Calcium Hydr oxide can be detecte d at wavenumb er around 3400 – 3600 cm - 1 , the absorp tion peaks in the range 1420 – 1480 cm - 1 refers to the vibra tions of carbona te assigned to O – C– O bond vibra tion, and the peaks at around 1000 cm - 1 corres pondin g to the formati on of CSH gel resul ted from the reaction of C 3 S and C 2 S during hydrat ion proces s [6 , 11].

The broad peaks at this wave number is attribut ed to Si – O, Si – O – Si and Si – O – Al bond s in CSH [6]. Acco rding to the ? gure, it can be report ed that the addit ion of micro- and ultra ? n e metakaolin into the mixtures co ntaining PCC and ? y ash accelera ted the consum ption of CH a t the 7th day, wher e the peak intensit ies of FA30. MK10 and FA30.

UM10 speci mens is 3434 and 3439 cm - 1 for, respec tively, lower than the Fig. 6 XRD spectra of a PCC, b FA-40, c FA30-MK10, d FA30-UM10 paste samples at 7 days after water curing days 424 S. W. M. Supit and F. Makalew peak inte nsities of PCC and FA40 speci mens . The h igh p eak of wave number also appear in FA30.

UM10 samp le at 1038 cm - 1 as an indi cation of the CSH form ation due to ultra ? ne meat akaolin. The intensity in this samp le was found higher than FA40, which shifted from 965 to 1038 cm - 1 . It can be explained that because of the very ? ne particles of ultr a ? ne metakaoli n, the hydrat ion process at the 7th day can be acceler ated, hence, improved the compr essive stre ngth of samp le containing 40% ? y ash, as discu ssed earli er.

5 Concl usions The follow ing conclusions could be drawn according to the experiment al results as follow s: 1. Micr o- and ultra ? ne meta kaolin source d from Tor aget Village in North Sulaw esi, Indones ia are very effective to be one of the potential natural resour ces in Indones ia to improve the perfor mance of sust ainable high v olume ? y ash (HVF A) c oncrete. 2.

The resul ts of compr essive stre ngth of concret e containing 40 and 60% ? y ash by wt . was imp roved after the add ition of 10% micro- and ultra ? ne-m etakao lin. The inclusio n of ultra ? ne-metakao lin is found more effecti ve in improvi ng the HVFA co ncrete proper ties at all testing days. Fig.

7 FTIR spectra of blended cement pastes containing micro- and ultra ? ne metakaolin at the 7th day Effects of Micro- and Ultra ? ne Metakaolin … 425 3. The presen ce of ultra ? ne metakaolin in FA40 concret e mixture signi ? c antly decreas ed the sorptivi ty values from 241 to 123 (_ 10 - 4 mm/s 1/2) at the 7th day and 225 – 118 (_10 - 4 mm/s 1/2) at the 28th day. The se values are much lower, compa red to the contr ol concret es incl uding PCC an d FA40 concrete mixes.

4. The analys is from XRD and FTI R also con ? rmed the ability of ultr a ? ne meta kaolin in acceler ating the hydrat ion proces s p articularly at the 7th day, hence, imp roved the compressi ve strength and wat er sorpt ivity of HVFA concret es. 5.

The production met hod of ultra ? ne-metakao lin is sti ll chall enging, therefore, furth er experi mental inves tigatio ns are still needed to develop the milli ng pro- cess from mic ro to ultra ? ne size of met akaolin. 6. The applica tion of high volum e ? y ash concret e combined with mic ro- and ultra ? ne metaka olin is very promisin g for stru ctural and non-struct ural element of b uilding const ruction.

Acknowledgements The activity presented in the paper is part of the internal research grant from Manado State Polytechnic in Manado, North Sulawesi, Indonesia. The contribution of Mr. Fikramsyah Vurman for data collection is also acknowledged.
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