

The 14th International Conference on QIR (Quality in Research)

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Dear Mr./Mrs. Setiawan,

The 14th International Conference on QIR (Quality in Research) 2015 Editorial Committee has completed the reviewing process and we are pleased to inform you that your manuscript;

Paper ID : 869
Title : SYNTHESIS OF PORTLAND NANO CEMENT USING SOL-GEL METHOD AS BINDER IN POROUS MEMBRANE FOR WATER FILTRATION PURPOSE
Author(s) : Heri Setiawan

has been **ACCEPTED** as **POSTER** presentation in the 14th International Conference on QIR (Quality in Research) 2015 Symposium on Metallurgy and Materials Engineering. For inclusion in the QIR 2015 Conference Proceeding we require **that you revise the full paper according to the reviewers' comment** if any and then submit the full paper camera ready by **30 April 2015** (use the QIR format as attached, **4 - 6 pages**).

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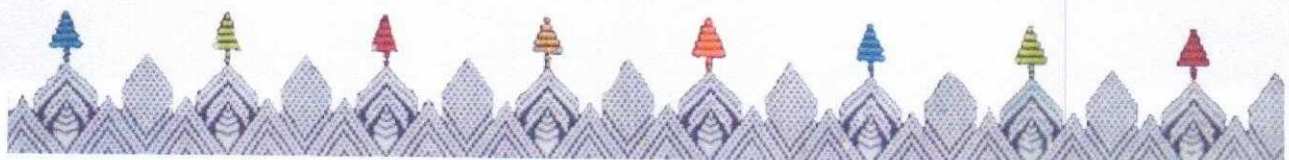
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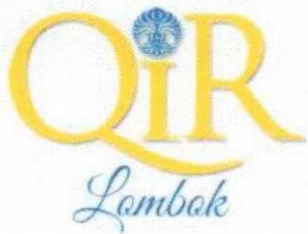
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If none of the authors will be able to attend and present the paper, corresponding author should notify us by replying this e-mail as soon as possible and request for withdrawal from the technical program. The conference will be disrupted by missing papers and gaps in oral or poster sessions. For all participants' convenience, we urge that all participants inform the QIR2015 secretariat about all cancellations as soon as possible.





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We highly appreciate your participation and look forward to welcoming you in QIR 2015 at Lombok, on 10-13 August 2015!

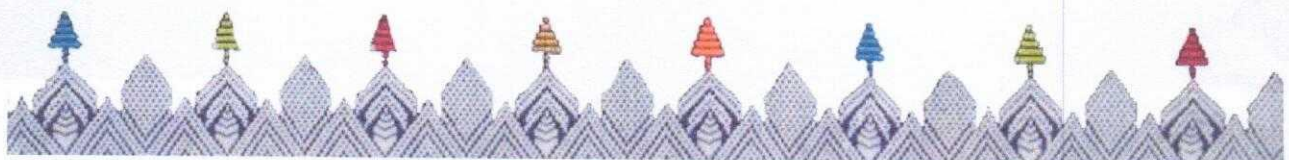
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SYNTHESIS OF PORTLAND NANO CEMENT USING SOL-GEL METHOD AS BINDER IN POROUS MEMBRANE FOR WATER FILTRATION PURPOSE

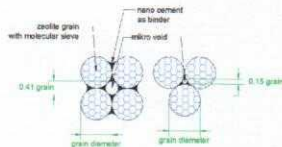
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Abstract

Portland nano cement has been made by sol-gel method. This will be aimed as a binder between zeolite aggregates on preparation of nano porous membrane for water filtration purpose, which normally conventional portland cement should be not used because of inhomogeneity of powder dimension. Synthesis of Portland nano cement began by mixing poly-ethyleneglicol (PEG) and distilled water for 10 minutes, then added calcium acetate as precursor and stirred for 10 minutes, and then added also other precursors material such as: Al_2O_3 , Fe_2O_3 and SiO_2 with a particular composition, stirred and heated for 30 minutes until gel is formed. The resulting gel then calcined at $1000^\circ C$ in a crucible furnace for 1 hour. Solid gel was formed which was then ground into nano powder. Analysis of X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and compressive test has been conducted to analyze the phase and degree of crystallization, to determine the morphology particle and mechanical properties of Portland nano cement.

Introduction

Synthesis of portland nano cement consisting of aqueous solutions as a solvent and PEG as organic template. As precursor for sol-gel process used $Ca(CH_3COOH)_2$. Additional materials were also added such as Al_2O_3 , SiO_2 , and Fe_2O_3 . This Portland nano cement will be used as binder in the future work on manufacture of water filtration membranes, where the zeolite grain will be bonded by Portland nano cement.



Above Figure illustrates the formation of a nanoporous layer. Gaps among zeolite grains bounded by nano cement. Zeolite grains is formed through top down method (ball milled grinding). Above figure did not have any unit size, just to show a comparison size between zeolite grain and pore (several micron in size). In this illustration shown that the formed pore size between zeolite grains were smaller compared with the zeolite grain.

Experimental details

Portland nano cement powder was produced by sol-gel process. $Ca(CH_3COOH)_2$ obtained from Merck was used as a precursor. As addition material were used Al_2O_3 , SiO_2 , and Fe_2O_3 . Distilled water was used as a solvent, while PEG was used as dispersant. Portland nano cement was synthesized using a sol-gel method. Step of experiments are described as follows:

Synthesis of tricalcium silica (C_3S): 4 grams of PEG dissolved in 15 ml of distilled water then was stirred for 10 minutes by a magnetic stirrer. In to solution was gently added 3.2 grams precursor of $Ca(CH_3COOH)_2$, then stirred for 10 minutes. 0.6 grams of SiO_2 was added in to solution then stirred as well. Aging process in solution then performed at temperature of $100^\circ C$ for 30 minutes at the same time stirred until gel is formed and then calcined at temperatures of $1000^\circ C$ for 1 hour.

Synthesis of tricalcium alumina (C_3A): same as above procedure but SiO_2 replaced by Al_2O_3 .

Synthesis of tetra calcium alumina ferite (C_4AF): same as above procedure but 0.6 grams of SiO_2 replaced by 1.6 gram Al_2O_3 and Fe_2O_3 .

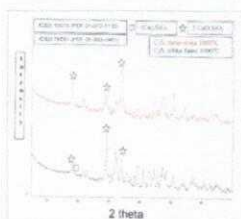
Specimen preparation of sample A; was done by mixing 4.23 g Portland nano cement with 6 ml of water, then mixed well until slurry formed. Slurry then poured into a cylindrical mold of PVC, which has 1.25 cm in diameter and 2.5 cm in length. Inside of the PVC was softly coated by a lubricant. This aims to make easier while releasing product. In other to remove the water content, specimen was dried in an oven for 1 day by temperature of $27^\circ C$. Dried specimens further dried in an oven for 6 days. Specimens then removed from the mold to conduct the test.

Specimen preparation of sample B; same procedure as preparation of sample A but 4.23 g Portland nano cement with 6 ml of water replaced by 6 g portland nano cement with 8 ml of water.

Results and discussion

This study shows the comparison of X-RD results between C_3S using additive materials nano silica and silica fume. Which C_3S greatly affects the compressive strength of the cement. Sample A is C_3S using additives of nano silica and sample B is C_3S using additives of silica fume. Both samples calcined at temperatures of $1000^\circ C$.

Below figure shows X-RD results from sample A and sample B. This figure shows X-RD results from C_3S powder with additional silica nanoparticle, size range 100 up to 400 nm by temperature range of $1000^\circ C$. From the figure can be seen that crystallization of minerals C_3S has occurred at temperature $1000^\circ C$. X-RD image below shows crystal structure of C_3S in monoclinic form. Identification based on X-RD analysis obtained some compositions phase such as: $Ca(OH)_2$ and $2CaO.SiO_2$.



From these data can be analyzed that for synthesis C_3S using nano silica as additive materials can be produced calcium hydroxide. This is caused by incomplete synthesis process, occurred in the formation of $CaSiO_3$. By this composition can be also estimated that the use of C_3S mineral, the synthesized Portland nano cement will have poor strength. The compressive strength of cement is strongly influenced by the condition of C_3S . C_3S is very important in terms of final strength. From this phase also produced crystals of $Ca(OH)_2$, which can reduce compressive strength of the cement. Hydration reaction of $Ca(OH)_2$ will produce voids that can also reduce compressive strength.

Above Figure also shows the X-RD results from C_3S powder with additional silica fume, size range 100 to 400 nm at temperatures of $1000^\circ C$. In the picture above is seen that crystallization of C_3S minerals has occurred at that temperature. In addition, the X-RD image showed crystal structure in the form monoclinic. Based on X-RD analysis obtained some compositions phase such as: $Ca(OH)_2$, $2CaO.SiO_2$, Ca_2SiO_4 , Ca_2SiO_5 , Ca_2SiO_6 , Ca_2SiO_7 , Ca_2SiO_8 , Ca_2SiO_9 , Ca_2SiO_{10} , Ca_2SiO_{11} , Ca_2SiO_{12} , Ca_2SiO_{13} , Ca_2SiO_{14} , Ca_2SiO_{15} , Ca_2SiO_{16} , Ca_2SiO_{17} , Ca_2SiO_{18} , Ca_2SiO_{19} , Ca_2SiO_{20} , Ca_2SiO_{21} , Ca_2SiO_{22} , Ca_2SiO_{23} , Ca_2SiO_{24} , Ca_2SiO_{25} , Ca_2SiO_{26} , Ca_2SiO_{27} , Ca_2SiO_{28} , Ca_2SiO_{29} , Ca_2SiO_{30} , Ca_2SiO_{31} , Ca_2SiO_{32} , Ca_2SiO_{33} , Ca_2SiO_{34} , Ca_2SiO_{35} , Ca_2SiO_{36} , Ca_2SiO_{37} , Ca_2SiO_{38} , Ca_2SiO_{39} , Ca_2SiO_{40} , Ca_2SiO_{41} , Ca_2SiO_{42} , Ca_2SiO_{43} , Ca_2SiO_{44} , Ca_2SiO_{45} , Ca_2SiO_{46} , Ca_2SiO_{47} , Ca_2SiO_{48} , Ca_2SiO_{49} , Ca_2SiO_{50} .

Synthesis of C_3S with additive materials silica fume generated calcium silicate phase, in the form of larnite and olivine. Moreover is seen that $Ca(OH)_2$ in sample B was not found. This is due to a perfect synthesis reaction in the formation of $2CaO.SiO_2$. Using C_3S mineral in synthesized Portland nano cement will have good strength because it contains no minerals $Ca(OH)_2$. The final strength of cement is strongly influenced by the conditions of the mineral C_3S . By using Scherrer equation [7] will be obtained a crystals size for sample A and B.

Press Test Analysis

The compressive strength of Portland nano cement reach 0.27 MPa (below figure), actually this would be not enough to be used as binder on zeolite membrane. Compressive strength curve diagram also shows increasing and decreasing value. These were caused by the absence of hydration on preparation of Portland nano cement. As noted in the X-RD results, the compositions of the C_3S mineral generated olivine minerals which affect the cement hydration process. Olivine significantly hindering cement hydration process so that the desired minerals are not fully formed or hydration processes were not occurred in some place. That's why the cement has low compressive strength.



Comparing stress vs. strain curves, Portland cement is much higher than Portland nano cement. This is due to the absence of olivine in the cement composition, wherein the mineral inhibit the hydration process between Portland nano cement and water.

SEM Characterization

Parts of Portland nano cement that does not undergo hydration process. It is shown in SEM characterization result image (below figure).



Below Figure shows the fracture part area of the Portland nano cement. This SEM characterization aims to see the fracture section of the specimen. Formed pore size is quite uniform but there are some clumps of binder caused by a less homogeneous mixing process. This causes compressive strength of Portland nano cement becomes lower.



Pore size was also not uniform, that why the compressive strength of Portland nano cements becomes lower. Moreover, it can be seen also in Figure, there are parts of the Portland cement were undergoing the hydration process and almost all minerals tend to undergo hydration process. No mineral was found to inhibit the cement hydration process. This was causing compressive strength of Portland cement becomes higher.

Conclusions And Suggestions

Some Conclusions are noted as follows:

1. The synthesis of Portland nano cement by sol-gel method has been done. This will be used in the future work as binder in a membrane for water filtration purpose. Compressive strength result of portland nano cement which is needed for mechanical properties of the membrane was achieved 0.27 MPa. This would be not enough to be used as binder on zeolite membrane. This because of hydration process of Portland nano cement was not taking place completely. Compressive strength was influenced by cement mineral composition, and the ratio between water and cement (W/C).
2. From X-RD result was found that there has been a crystallization of C_3S minerals with additional nano silica material at temperature of $1000^\circ C$. Crystals size of 13.2 nm have been obtained. Where by crystallization of C_3S minerals with additional silica fume material at temperature of $1000^\circ C$ was obtained crystal size of 21 nm.

Suggestion:

Studies on the synthesis and characterization of Portland nano cement should be further developed, especially the effect of olivine minerals that inhibit reaction of cement hydration.

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