PENGARUH *FILLER* KALSIUM OKSIDA DAN *PRECIPITATED CALCIUM CARBONATE* PADA SIFAT *WHITE MINERAL TRIOXIDE AGGREGATE* (WMTA) HASIL SINTESIS DARI BATU KAPUR DAN ABU SEKAM PADI

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INTISARI

White mineral trioxide aggregate (WMTA) telah disintesis menggunakan *precipitated calcium carbonate* (PCC) dari batu kapur dan silika dari abu sekam padi (ASP). Selain digunakan sebagai prekursor WMTA, PCC juga digunakan sebagai *filler* nonpozolanik pada WMTA hasil sintesis, beserta dengan kalsium oksida (CaO) untuk diketahui pengaruhnya terhadap sifat WMTA. Metode karbonasi digunakan untuk sintesis PCC dan dikaji pengaruh pelarut dan diameter *nozzle* karbonasi terhadap sifat PCC. Silika ASP diperoleh dengan metode sol-gel, setelah ASP didestruksi dengan NaOH dan dicuci sampai bebas ion natrium. Silika ASP dan PCC dikarakterisasi dengan XRD, SEM dan XRF. Sintesis WMTA dilakukan dengan mencampur prekursor-prekursor yaitu PCC dari batu kapur (60,000%), silika ASP (20,000%), dan Al_2O_3 **(**2,000%) kemudian dikalsinasi pada 1000 ºC, dan ditambahkan Bi_2O_3 (18,000%). Hasil sintesis WMTA ditambahkan *filler* **(**WMTA*f***)** nonpozolanik yaitu CaO dan PCC dari batu kapur dengan masing-masing 5,000 dan 10,000% berat WMTA. WMTA dan WMTA*f* dikarakterisasi dengan TGA-DSC, FTIR, XRD dan SEM-EDX. Selain itu terhadap produk yang dihasilkan dilakukan hidrasi dan diuji *in vitro* berupa uji kelarutan, pH, pelepasan ion Ca^{2+}, sifat mekanik dengan uji *diameter tensile strength* (DTS) dan radiopasitas.

Hasil menunjukkan bahwa PCC yang dibuat dengan pelarut HNO_3 dan *nozzle* berdiameter 0,800 mm memiliki fasa kristal kalsit berukuran kristal 2,420 ± 0,670 µm dan homogen, menghasilkan rendemen 96,010 %, serta kemurnian 99,992%. Silika ASP didapatkan berfase amorf dengan rendemen 91,870% serta kemurnian 92,825%. WMTA berhasil disintesis menggunakan PCC dari batu kapur dan silika ASP dengan temperatur dekomposisi 450-750 ºC, memiliki gugus fungsi, difraktogram dan morfologi serta komposisi yang mirip dengan ProRoot WMTA. Hasil uji *in vitro* menunjukkan bahwa *filler* CaO meningkatkan kelarutan WMTA dan menurun dengan *filler* PCC. *Filler* CaO meningkatkan pH menjadi lebih basa, pelepasan ion Ca^{2+} meningkat dengan adanya *filler* pada WMTA, pelepasan tertinggi pada *filler* CaO 10,000%. Nilai DTS tertinggi diperoleh pada *filler* CaO 5,000% (6,300±0,520 MPa) dengan media perendaman *artificial saliva*, sedangkan dengan media perendaman air deionasi diperoleh nilai DTS tertinggi dengan *filler* PCC 10% (9,770±0,400 MPa). Hasil uji radiopasitas didapatkan bahwa WMTA dan WMTA*f* sesuai ISO 6876:2001 dan spesifikasi ANSI/ADA nomor 57 bernilai di atas 3 mmAl.

Kata kunci: PCC, silika ASP, WMTA, *filler*, nonpozolanik

THE EFFECT OF CALCIUM OXIDE AND CALCIUM CARBONATE FILLER ON THE PROPERTIES OF WHITE MINERAL TRIOXIDE AGGREGATE (WMTA) SYNTHESISED FROM LIMESTONE AND RICE-HUSK-ASH

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ABSTRACT

White Mineral Trioxide Aggregate (WMTA) has been synthesised using precipitated calcium carbonate (PCC) from limestone and silica from rice-husk-ash (RHA). Besides being used as a precursor of WMTA, PCC was also used as a filler nonpozzolanic on WMTA, along with calcium oxide (CaO) to determine its effect on WMTA properties. PCC was synthesised by carbonation method and then studied the effect of solvent and carbonation nozzle diameter on the properties of PCC. Silica RHA was obtained by the sol-gel method after the RHA was digested with NaOH and washed until it was free of sodium ions. Silica RHA and PCC were characterized by XRD, SEM and XRF. WMTA synthesised by blend PCC from limestone (60.000%), silica (RHA) (20.000%), and Al_2O_3 (2.000%) then calcined at 1000 ºC, and added with Bi_2O_3 (18.000%). WMTA modified by adding filler (WMTA*f*) with a mass ratio of CaO and PCC, 5.000 and 10.000%, respectively. WMTA and WMTA*f* were characterized by TGA-DSC, FTIR, XRD and SEM-EDX. In addition, the resulting products were hydrated and tested *in vitro* in the form of solubility tests, pH, release of Ca^{2+} ions, mechanical properties and radiopacity.

The results showed that the PCC made with HNO_3 solvent and a nozzle with a diameter of 0.800 mm had a crystalline phase of calcite with crystal size of 2.420 ± 0.670 µm and was homogeneous, yield of 96.010%, and purity of 99.992%. Silica RHA was obtained in amorphous form with 91.870% yield and 92.825% purity. WMTA was successfully synthesised using PCC from limestone and silica RHA with a decomposition temperature of 450-750 ºC, has functional groups, difractogram, morphology and composition similar to ProRoot WMTA. *In vitro* test show that filler CaO increased the solubility of WMTA and decreased with the filler PCC. Calcium oxide (CaO) filler increases the pH to be more alkaline, the release of Ca^{2+} ions increase with filler in WMTA, and the highest release is the addition of 10.000% CaO filler. The highest DTS value was obtained in the addition of 5.000% CaO filler (6.300 ± 0.520 MPa) with artificial saliva immersion, while with deionized water immersion the highest DTS value was obtained with PCC (10.000%) filler (9.770 ± 0.400 MPa). The radiopacity test showed that the WMTA and WMTA fillers according to ISO 6876: 2001 and ANSI / ADA specification number 57 were above 3 mmAl.

Keyword: PCC, Silica RHA, WMTA, filler, nonpozzolanic