# EFFECT OF CHEMICALLY PRETREATED PAPER MILL SLUDGE ASH ON THE PROPERTIES OF GEOPOLYMER MORTAR

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## SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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## STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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# ABSTRAK

Geopolimer telah dikenali sebagai pengikat alternatif yang kukuh kepada simen Portland biasa (OPC) kerana sifatnya seperti kekuatan mampatan yang tinggi, rintangan kimia dan haba yang baik dan komposisi toksik yang rendah. Dalam geopolimer, penggunaan abu terbang sebagai sumber aluminosilikat utama telah digunakan secara meluas kerana jumlah silika dan alumina yang tinggi. Walau bagaimanapun, terdapat banyak sisa industri yang dihasilkan dari pelbagai industri telah menimbulkan masalah dalam system pelupusan dan ianya menjejaskan alam sekitar. Oleh itu, pelbagai jenis buangan seperti abu kumbahan kilang kertas boleh dicampur dengan abu terbang sebagai bahan mentah utama didalam geopolimer. Gabungan ini akan memberikan kekuatan dan sifat ketahanan yang lebih baik atau setanding dan juga membantu dalam mengurangkan peratusan sisa industri. Walaubagaimanapun, sisa ini tidak boleh digunakan secara bersendirian dalam geopolimer kerana jumlah silika ($SiO_2$) dan alumina ($Al_2O_3$) yang tidak mencukupi dan jumlah Ca yang tinggi. Jumlah Ca yang tinggi dalam geopolimer menyumbang kepada masa penetapan yang cepat dan kebolehpasaran yang rendah. Oleh itu, kajian ini dilaksanakan untuk merawat PMSA dan menyiasat kesan PMSA yang telah dirawat dalam mortar geopolimer di bawah suhu pengawetan (30°C dan 90°C). Abu kumbahan kilang kertas dirawat dengan menggunakan asid hidroklorik (HCl) dengan pelbagai molariti iaitu 0.5 M, 1.0 M dan 1.5 M. Satu siri ujian dijalankan untuk menentukan sifat-sifat geopolimer yang mengandungi PMSA pra-rawatan pada pelbagai peratusan iaitu 5%, 10% dan 15% berat abu terbang. Ujian pengaliran dan penetapan masa telah dijalankan untuk menentukan sifat-sifat reologi manakala kekuatan mampatan, keliangan dan darjah ujian reaksi dilakukan untuk menentukan sifat-sifat mekanik. Sementara itu, XRF dan XRD diuji pada bahan abu terbang mentah, PMSA dan PMSA yang telah dirawat terlebih dahulu untuk menentukan komposisi oksida dan bahan kristal. Spektrometri jisim plasma yang digabungkan secara induktif (ICPMS) pula diuji pada cecair pra-rawatan PMSA untuk menentukan kepekatan logam berat. Sementara itu, ujian Inframerah Transformasi Fourier (FTIR) telah dijalankan untuk mengenalpasti ikatan kimia. Berdasarkan hasil eksperimen, PMSA yang dirawat menggunakan HCl, telah meningkatkan peratus oksida seperti $SiO_2$ (8.23% hingga 44.00%) dan $Al_2O_3$ (4.36% hingga 19.60%) dan mengurangkan jumlah CaO (52.61% hingga 22.00%). Jumlah $SiO_2$ dan $Al_2O_3$ yang tinggi memainkan peranan utama dalam pembentukan tulang belakang geopolimer (bon Si–O–Al dan Si-O-Si). Sementara itu, peratusan tambahan CaO dalam sumber aluminosilikat telah menghasilkan produk sekunder iaitu gel CSH dalam matriks geopolimer kerana ikatan Ca–O lebih mudah dipecahkan daripada Al–O dan Si–O. Setiap komposisi oksida juga berfungsi dalam pengeluaran struktur akhir dengan mengawal gel geopolimer dan pertumbuhan kristal dengan itu menyumbang kepada pembangunan struktur. Peratusan kemasukkan optimum PMSA yang telah dirawat di dalam geopolimer adalah 5% dengan 1.5 M dari HCl. Gabungan ini telah memanjangkan masa penetapan, kebolehkerjaan yang tinggi, kekuatan mampatan yang tinggi, mengurangkan keliangan dan menghasilkan mikrostruktur yang padat. Pendedahan kepada suhu pengerasan 90°C telah mempercepatkan proses geopolimerisasi dan meningkatkan pembentukan gel geopolimer dalam rangka kerja geopolimer. Oleh itu, penggunaan abu kumbahan kilang kertas di dalam geopolimer akan membantu menguruskan sisa yang banyak dan akan membawa kepada bahan binaan hijau lestari yang membantu memelihara sumber semula jadi dan meningkatkan kualiti persekitaran.

**ABSTRACT**

Geopolymer has been well known as a sustainable alternative binder to ordinary Portland cement (OPC) because of it properties such as high compressive strength, good chemical and thermal resistance and low toxic composition. In geopolymer, utilization of fly ash as the main aluminosilicate source has been widely used due to high amount of silica ($SiO_2$) and alumina ($Al_2O_3$). However, abundant of industrial waste are being released from various industries has become difficulties in their disposal and will affect to the environment. Therefore, it is possible to use different types of wastes such as paper mill sludge ash (PMSA) mixed with fly ash as raw materials. This combination would help to provide better or comparable strength and durability properties and also help in reducing the percentages of waste by product. However, this waste cannot be used alone in the geopolymer because of insufficient amount of $SiO_2$ and $Al_2O_3$ and high amount of Ca. The present of high amount of Ca in geopolmyer contributed to the rapid setting time and low workability. Therefore, this study was proposed to treat the paper mill sludge ash and investigate the effect of pretreated PMSA in geopolymer mortar at various curing temperature (30°C and 90°C). PMSA has been treated with hydrochloric acid (HCl) at various molarities which is 0.5 M, 1.0 M and 1.5 M. A series of test were conducted to determine the properties of geopolymer containing pretreated PMSA at various percentages of 5%, 10% and 15% by weight of fly ash. Flowability and setting time test were conducted to determine the rheological properties while compressive strength, porosity and degree of reaction tests were conducted to determine the mechanical properties. Meanwhile, XRF and XRD were tested on raw materials of fly ash, PMSA and pretreated PMSA to ascertain the oxide composition and crystalline material. Inductively coupled plasma mass spectrometry (ICPMS) was tested on solution of pretreatment PMSA to determine the heavy metal concentration. Meanwhile, Fourier Transform Infrared Spectroscopy (FTIR) testing was conducted to identify chemical bonds. Based on the experimental results, by treated PMSA in acid washing using HCl has significantly enhanced the oxide percentages such as $SiO_2$ (8.23% to 44.00%) and $Al_2O_3$ (4.36% to 19.60%) and reduce the amount of CaO (52.61% to 22.00%). High amount of $SiO_2$ and $Al_2O_3$ have a major role to play in the geopolymer backbone formation (Si–O–Al and Si–O–Si bonds). Meanwhile, addition percentages of CaO in aluminosilicate source have produced the secondary product which is CSH gel in geopolymer matrix as Ca–O bond is more susceptible to break the bonds than Al–O and Si–O. Each oxide composition is also functioning in the production of final structure by controlling the geopolymer gel and crystal growth hence contribute to the structural development. The optimum percentages inclusion of pretreated PMSA in geopolymer was 5% with 1.5 M of HCl. This combination has significantly prolonged the setting time, high workability, higher in compressive strength, reduce the porosity and produce denser microstructure. Exposure to 90 °C of curing temperature has accelerated the geopolymerization process and enhances the formation of geopolymer gel in geopolymer framework. Conclusively, the use of pretreated PMSA in geopolymer would reduce quantity of wastes thrown at landfill and promote the development of construction materials contributing towards cleaner environment for healthier community.

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