

EFFECT OF METAL OXIDES ON THE  
RELEASING OF NUTRIENT IONS FROM  
PHOSPHATE GLASS FERTILIZER SYSTEM

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**EFFECT OF METAL OXIDES ON THE RELEASING OF NUTRIENT IONS  
FROM PHOSPHATE GLASS FERTILIZER SYSTEM**

**SITI HAFIZAH BINTI MOHAMAD @ MD HUSSIN**

**Thesis Submitted in Fulfilment of the Requirement for the Degree of Master of  
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Submitted by: *[Name]*

For the degree of *[Degree]*

*Dedicated to*

*My beloved parents and siblings*

Abstract of thesis presented to the Senate of Universiti Malaysia Terengganu in fulfilment of the requirements for the degree of Master of Science

**EFFECT OF METAL OXIDES ON THE RELEASING OF NUTRIENT IONS FROM PHOSPHATE GLASS FERTILIZER SYSTEM**

**SITI HAFIZAH BINTI MOHAMAD @ MD HUSSIN**

**July 2016**

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**School : School of Fundamental Science**

Nowadays, the study of phosphate glass as potential glass fertilizer has been widely conducted due to the environmental consciousness of using chemical fertilizer that caused soil acidulation and water pollution. In this study, three phosphate glass systems of  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>,  $P_2O_5$ -CaO-Na<sub>2</sub>O-K<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub> and  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> with (0-20 wt. %) K<sub>2</sub>O, (0-20 wt. %) Na<sub>2</sub>O and (2-8 wt. %) Fe<sub>2</sub>O<sub>3</sub> have been prepared in the alumina crucible at 1300 °C via the conventional melting technique. The phosphate glass samples were characterized by using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Differential Scanning Calorimetry (DSC) to determine the physical and chemical properties. For solubility studies, all glass samples have been immersed in deionised water for 28 days under the static condition and the nutrient ions release were analysed using Ion Chromatography (IC) and Atomic Absorption Spectroscopy (AAS). The released of phosphate ion, potassium ion and calcium ion from the  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub> glass increased with the increases of K<sub>2</sub>O content from 0 to 15 wt. % and decreased with the addition of 20 wt. % K<sub>2</sub>O. FTIR analysis indicates the peaks around 1115 cm<sup>-1</sup> to 1119 cm<sup>-1</sup> were associated with the formation of metaphosphate and pyrophosphate

unit due to the rearrangement of the phosphate glass structure with the addition of  $K_2O$ . Meanwhile, the presence of pores on the glass surface indicates the corrosion of phosphate glass samples after several days of immersion in deionised water. Based on the conducted study, the addition of  $K_2O$  has increased the glass solubility while the addition of  $Na_2O$  with the presence of  $K_2O$  has decreased the glass solubility. The addition of 0 to 20 wt. %  $Na_2O$  to the  $P_2O_5$ - $CaO$ - $Na_2O$ - $K_2O$ - $Al_2O_3$  glass system have decreased the phosphate and potassium ion release but increased the sodium ion release. The  $T_g$  values of phosphate glass decreased from 510 °C to 453 °C with the addition of 5 to 15 wt. %  $Na_2O$  and increased to 486 °C with the addition of 20 wt. %  $Na_2O$  due to increase in the phosphate glass durability. Meanwhile, the solubility of  $P_2O_5$ - $CaO$ - $K_2O$ - $Na_2O$ - $Fe_2O_3$ - $Al_2O_3$  glass decreased with the addition of 2 to 8 wt. %  $Fe_2O_3$  due to the formation of P-O-Fe bond, which more hydration resistant compared to P-O-P bond. The  $T_g$  values also increased from 453 °C to 459°C with the incorporation of 2 to 8 wt. %  $Fe_2O_3$  that shows the phosphate glass structure become highly durable. Other than that, optimization of the  $45P_2O_5$ - $16CaO$ - $15K_2O$ - $14Na_2O$ - $4Fe_2O_3$ - $6Al_2O_3$  glass shows an increasing trend of nutrient ions release within four months of immersion with 8.3% of weight loss and pH 6.80-6.58, which nearly to the soil pH of 6.5. Thus, this glass has a great potential to be used as a glass fertilizer. Based on the conducted studies, the addition of  $K_2O$  and  $Na_2O$  contributed to the increase of the glass solubility due to the break up of phosphate glass structure. However, the ionic cross-linking between  $Fe_2O_3$  and phosphate glass chain has improved the phosphate glass durability.

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**KESAN OKSIDA LOGAM TERHADAP PEREMBESAN ION NUTRIEN  
DARIPADA SISTEM KACA BAJA FOSFAT**

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**Pusat Pengajian : Pusat Pengajian Sains Asas**

Kebelakangan ini, kajian penggunaan kaca fosfat sebagai kaca baja telah dijalankan dengan meluas disebabkan kesedaran penggunaan baja kimia terhadap alam sekitar yang mengakibatkan pengasidan tanah dan pencemaran air. Dalam kajian ini, tiga sistem kaca fosfat iaitu  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>,  $P_2O_5$ -CaO-Na<sub>2</sub>O-K<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub> dan  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Fe<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> dengan (0-20 wt. %) K<sub>2</sub>O, (0-20 wt. %) Na<sub>2</sub>O dan (2-8 wt. %) Fe<sub>2</sub>O<sub>3</sub> telah dihasilkan di dalam mangkuk pijar pada suhu 1300 °C menggunakan teknik peleburan konvensional. Kaca yang diperolehi dilakukan pencirian menggunakan Spektroskop Inframerah Transformasi Fourier (FTIR), Pembelauan Sinar-X (XRD), Mikroskop Imbasan Elektron (SEM) dan juga Kalorimetri Pembezaan Pengimbasan (DSC) bagi menentukan sifat kimia dan sifat fizikal kaca. Bagi ujian keterlarutan, sampel-sampel kaca direndam ke dalam larutan air ternyah ion dalam keadaan statik selama 28 hari dan kepekatan ion yang terembes dari kaca fosfat dianalisa menggunakan Kromatografi Ion (IC) dan juga Spektroskopi Penyerapan Atom (AAS). Kadar perembesan ion fosfat, ion kalium dan juga ion kalsium dari kaca  $P_2O_5$ -CaO-K<sub>2</sub>O-Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub> meningkat dengan penambahan K<sub>2</sub>O sebanyak 0 hingga 15 wt. % dan menurun dengan penambahan 20 wt. % K<sub>2</sub>O. Analisa spektrum Inframerah menunjukkan kehadiran puncak sekitar

1115  $\text{cm}^{-1}$  dan 1119  $\text{cm}^{-1}$  menunjukkan pembentukan unit metafosfat dan pyrofosfat ekor an penyusunan struktur kaca dengan penambahan  $\text{K}_2\text{O}$ . Sementara itu, kehadiran liang pada permukaan kaca menunjukkan hakisan berlaku selepas beberapa hari kaca direndam di dalam larutan air ternyah ion. Berdasarkan kajian yang telah dijalankan, penambahan  $\text{K}_2\text{O}$  telah meningkatkan kadar keterlarutan kaca manakala penambahan  $\text{Na}_2\text{O}$  disamping kehadiran  $\text{K}_2\text{O}$  telah menurunkan kadar keterlarutan kaca fosfat. Penambahan 0 hingga 20 wt. %  $\text{Na}_2\text{O}$  ke dalam sistem kaca  $\text{P}_2\text{O}_5\text{-CaO-Na}_2\text{O-K}_2\text{O-Al}_2\text{O}_3$  telah menurunkan kadar perembesan ion fosfat dan ion kalium namun meningkatkan kadar perembesan ion natrium. Nilai  $T_g$  bagi kaca fosfat telah menurun daripada 510  $^\circ\text{C}$  ke 453  $^\circ\text{C}$  dengan penambahan 5 hingga 15 wt. %  $\text{Na}_2\text{O}$  dan meningkat ke 486  $^\circ\text{C}$  dengan penambahan 20 wt. %  $\text{Na}_2\text{O}$  disebabkan oleh peningkatan kestabilan kaca. Sementara itu, keterlarutan kaca  $\text{P}_2\text{O}_5\text{-CaO-K}_2\text{O-Na}_2\text{O-Fe}_2\text{O}_3\text{-Al}_2\text{O}_3$  menurun dengan penambahan 2 hingga 8 wt. %  $\text{Fe}_2\text{O}_3$  dengan pembentukan ikatan P-O-Fe yang mempunyai ketahanan hidrasi lebih kuat berbanding ikatan P-O-P. Nilai  $T_g$  juga meningkat daripada 453  $^\circ\text{C}$  ke 459 $^\circ\text{C}$  dengan penambahan 2 hingga 8 wt. %  $\text{Fe}_2\text{O}_3$  yang menunjukkan struktur kaca fosfat menjadi lebih kuat. Kajian pengoptimaan kaca  $45\text{P}_2\text{O}_5\text{-16CaO-15K}_2\text{O-14Na}_2\text{O-4Fe}_2\text{O}_3\text{-6Al}_2\text{O}_3$  menunjukkan trend perembesan ion selama empat bulan meningkat dengan kehilangan berat sebanyak 8.3% serta pH sekitar pH6.80-6.58 yang menghampiri pH tanah. Oleh itu, kaca ini mempunyai potensi yang tinggi untuk dijadikan kaca baja. Berdasarkan kajian yang telah dijalankan, penambahan  $\text{K}_2\text{O}$  dan  $\text{Na}_2\text{O}$  ke dalam sistem kaca telah meningkatkan kadar keterlarutan kaca disebabkan oleh pemutusan ikatan kaca fosfat. Namun begitu, ikatan silang antara  $\text{Fe}_2\text{O}_3$  dan rantaian kaca fosfat telah meningkatkan kestabilan kaca fosfat.