UNIVERSITI SAINS MALAYSIA
GERAN PENYELIDIKAN UNIVERSITI PENYELIDIKAN
LAPORAN AKHIR

SYNTHESIS OF NANOHYDROXYPATITE-SILICA HYBRID BY IN-SITU SOL-GEL FOR FABRICATION OF GLASS LONOMER CEMENT (GIC) DENTAL NANOCOMPOSITES

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PROJECT ACHIEVEMENT *(Prestasi Projek)*

B

ACHIEVEMENT PERCENTAGE			
Project progress according to milestones achieved up to this period	**0 - 50%**	**51 - 75%**	**76 - 100%**
Percentage (please state #%)			100%

RESEARCH OUTPUT		
	Indexed Journal	***Non-Indexed Journal***
Number of articles/ manuscripts/ books *(Please attach the First Page of Publication)*	(i) Marahaini Musa, Thirumulu Ponnuraj Kannan, Sam'an Malik Masudi & Ismail Ab Rahman. *Mol. Cell. Toxicol.* (2012) 8:53-60. (ii) Rayees Ahmad Shiekh, Ismail Ab Rahman, Maqsood Ahmad Malik, Norhayati Luddin, Sam'an Malik Masudi, Shaeel Ahmed Al-Thabaiti. *Int. J. Electrochem. Sci.*, 8 (2013) 6972 - 6987 (iii) Ismail Ab Rahman*, Sam'an Malik Masudi, Norhayati Luddin, Rayees Ahmad Shiekh. *Bull. Mater. Sci.* 37(2), 2014, 213–219. (iv) Rayees Ahmad Shiekh, Ismail Ab Rahman, Sam'an Malik Masudi, Norhayati Luddin. *Ceramics International 40 (2014) 3165–3170*	Tengku Yasmin Tengku Azama, Quah Xin Ying, Ismail Ab Rahman, Sam'an Malik Masudia, Norhayati Luddin, Rashita Abd Rashid. *Arch Orofac Sci* (2013), 8(2): 54-59
Conference Proceeding *(Please attach the First Page of Publication)*	**International**	***National***
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Intellectual Property *(Please specify)*	-	

RESEARCH ABSTRACT – Not More Than 200 Words *(Abstrak Penyelidikan – Tidak Melebihi 200 patah perkataan)*	
G	In this work, an hybrid of nanohydroxyapatite-silica (nano-HA-silica) has been successfully synthesized by in-situ sol-gel process. The hybrid was incorporated into a glass ionomer cement (GIC) to form a new GIC composite with superior hardness properties. It was found that the presence of higher degree of cross-linking of silyl species between silica and GIC, which makes the Nano-HA–Silica–GIC composite much stronger. In addition, the improvement in the hardness of new composite is attributed to the higher packing density of the cement and homogeneous distribution of the nano-HA-silica hybrid in the GIC matrix. Application of HA-silica-GIC with improved hardness property might lead to extended clinical indications, especially in stress bearing areas.

Date : 26 June 2014
Tarikh

Project Leader's Signature:
Tandatangan Ketua Projek

COMMENTS, IF ANY/ENDORSEMENT BY RESEARCH MANAGEMENT CENTER (RMC)
(Komen, sekiranya ada/Pengesahan oleh Pusat Pengurusan Penyelidikan)

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Date:
Tarikh:

1. INTRODUCTION

Glass ionomer cements (GICs) have been widely used as restorative dental materials due to the biocompatibility property that able to form direct bonding to the tooth structure and the release of fluoride ions that protect against dental caries and able to regenerate hard tissues including bones and tooth structure. Among the disadvantages, GICs are chemically soluble in saliva water environment that need a protective layer of methacrylate based gloss, the setting reaction affected by water, abrasive, weak in mechanical properties such as strength and hardness. Thus, it is required to develop new composition and the studies on the improvement of the mentioned aspects in order to find a better product for future restorative dental material. Modification of commercial GICs with hydroxyapatite (HA) stainless steel, zirconia, glass, HA/zirconia, HA/glass have been reported in literatures to improve mechanical properties. However, the improvement on mechanical properties was not significant. In all cases, they used commercial powders with particle size in micro range. In addition, a fundemental studies on the effect of powder morphologies and the interaction between additive and GIC that affect mechanical and chemical and biological properties were not elaborated. In our knowledge, the work on the addition of HA/silica nanoparticle is not yet reported in literature.

This work is aimed to produce a new composition of GIC by incorporation of nano HA-silica hybrid to obtain nanocomposites with superior properties. The HA-silica hybrid is derived from *in-situ* sol-gel process that has been developed in our laboratory. Therefore, incorporation of HA-silica inside the GIC has improved the hardness due to the better interaction between HA-silica-GIC and homogeneous distribution of additives.

2. RESEARCH METHODOLOGY

2.1. Synthesis of hydroxyapatite

Calcium hydroxide and phosphoric acid were the sources of calcium and phosphorus. Initially, calcium hydroxide was mixed into 100 ml of distilled water in a glass beaker and stirred for 30 min using a magnetic stirrer, until all calcium hydroxide powder was well mixed to form suspension. After that, phosphoric acid was added drop wise to the suspension. The pH of the suspension was adjusted to the range of 11-12 using ammonia. The suspension was stirred for 48 h to obtain a white viscous sol. The sol was filtered, freeze dried and calcined at 600 ^{0}C for 1 h.

2.2. Synthesis of hydroxyapatite-silica-nanocomposite

First, the nanohydroxyapatite (HA) was prepared by the procedure in section 2.1. A quantity of TEOS (5, 10, 20 ml) that had dissolved in 10 ml of ethanol was added drop wise into the sol at 12 h. The sol was centrifuged, dried using a freeze dryer and calcined at 600 ^{0}C for 1 h. The same procedure was repeated for addition of 10 ml and 20 ml TEOS. Based chemical equations, the theoretical composition of HA-silica and HA in the composites were calculated as HA-11%SiO_2, HA-21%SiO_2 and HA-35%SiO_2.

2.3. Preparation of nano-HA-silica-added GIC

A commercial GIC was used as the control and base material. HA, HA-11%SiO_2, HA-21%SiO_2 and HA-35%SiO_2 and silica were mixed into the GIC at various percentages