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ภาคผนวก

ภาคผนวก ก

ค่าความหนาแน่นของแก้วระบบฟอสฟอรัสแคลเซียมโซเดียมออกไซด์ (P_2O5-CaO-Na_2O)

แสดงค่าความหนาแน่นของแก้วระบบฟอสฟอรัสแคลเซียมโซเดียมออกไซด์ (P_2O5-CaO-Na_2O)

Conditions	Density (g/cm^3)
$45P_2O_5$-32CaO-$23Na_2O$	2.6796
$45P_2O_5$-36CaO-$19Na_2O$	2.7175
$45P_2O_5$-40CaO-$15Na_2O$	2.780

ภาคผนวก ข

ค่าการหดตัวเชิงเส้น (Linear shrinkage)

แสดงค่าร้อยละการหดตัวเชิงเส้น (Linear shrinkage) ของเม็ดสารเซรามิกแคลเซียมฟอสเฟตที่มีรูพรุน ในอัตราส่วนต่างๆ

Conditions	**Sintering temperature**	**Linear shrinkage (%)**		
		Compositions (glass : camphor)		
		10:0	**7:3**	**5:5**
$45P_2O_5$-32CaO-23Na_2O	500°C	10.446 ± 0.147	15.144 ± 0.690	21.352 ± 0.256
	550°C	10.459 ± 0.226	14.985 ± 0.405	22.563 ± 0.902
	600°C	10.737 ± 0.394	19.058 ± 0.263	27.368 ± 0.701
	650°C	10.205 ± 0.130	18.034 ± 0.688	26.086 ± 1.195
$45P_2O_5$-36CaO-19Na_2O	500°C	9.043 ± 0.219	4.500 ± 0.204	4.451 ± 0.200
	550°C	10.722 ± 0.137	4.518 ± 0.177	4.267 ± 0.334
	600°C	10.966 ± 0.189	12.788 ± 0.624	14.589 ± 0.304
	650°C	10.021 ± 0.190	11.482 ± 0.284	10.972 ± 0.324
$45P_2O_5$-40CaO-15Na_2O	500°C	3.774 ± 0.392	6.850 ± 0.367	7.037 ± 0.695
	550°C	7.736 ± 0.123	9.056 ± 0.362	11.037 ± 0.505
	600°C	9.846 ± 0.168	7.201 ± 0.691	6.746 ± 0.178
	650°C	9.281 ± 0.452	5.539 ± 0.205	6.051 ± 0.258

ภาคผนวก ค

ค่าความหนาแน่น (Density)

แสดงค่าความหนาแน่น (Density) ของเม็ดสารเซรามิกแคลเซียมฟอสเฟตที่มีรูพรุน ในอัตราส่วนต่างๆ

Conditions	**Sintering temperature**	**Density (g/cm^3)**		
		Compositions (glass : camphor)		
		10:0	**7:3**	**5:5**
$45P_2O_5$-32CaO-$23Na_2O$	500°C	2.665 ± 0.009	1.852 ± 0.059	1.616 ± 0.042
	550°C	2.572 ± 0.001	1.924 ± 0.035	1.692 ± 0.067
	600°C	2.557 ± 0.004	2.088 ± 0.005	1.959 ± 0.012
	650°C	2.484 ± 0.007	2.071 ± 0.002	1.806 ± 0.021
$45P_2O_5$-36CaO-$19Na_2O$	500°C	2.563 ± 0.024	1.324 ± 0.014	0.765 ± 0.213
	550°C	2.666 ± 0.004	1.429 ± 0.104	1.162 ± 0.061
	600°C	2.649 ±0.010	1.659 ± 0.034	1.189 ± 0.037
	650°C	2.574 ± 0.007	1.596 ± 0.044	1.013 ± 0.011
$45P_2O_5$-40CaO-$15Na_2O$	500°C	2.213 ± 0.015	1.509 ± 0.048	1.194 ± 0.099
	550°C	2.503 ± 0.018	1.634 ± 0.029	0.898 ± 0.086
	600°C	2.642 ± 0.011	1.501 ± 0.042	0.997 ± 0.089
	650°C	2.613 ± 0.017	1.417 ± 0.061	0.970 ± 0.109

ภาคผนวก ง

ค่าความพรุนที่ปรากฏ (Apparent porosity)

แสดงค่าร้อยละความพรุนที่ปรากฏ (Apparent porosity) ของเม็ดสารเซรามิกแคลเซียมฟอสเฟตที่มีรูพรุน ในอัตราส่วนต่างๆ

Conditions	**Sintering temperature**	**Apparent porosity (%)**		
		Compositions (glass : camphor)		
		10:0	**7:3**	**5:5**
$45P_2O_5$-32CaO-23Na_2O	500°C	1.414 ± 0.254	32.033 ± 1.989	41.710 ± 2.849
	550°C	8.967 ± 0.081	33.745 ± 1.307	41.726± 3.912
	600°C	3.197 ± 0.114	24.115 ± 1.029	32.267 ± 1.117
	650°C	4.213 ± 0.457	24.651 ± 1.334	36.927 ± 1.852
$45P_2O_5$-36CaO-19Na_2O	500°C	3.191 ± 0.847	49.253 ± 0.276	57.287 ± 14.39
	550°C	4.486 ± 0.111	50.254 ± 3.452	59.574 ± 5.784
	600°C	5.324 ± 0.210	40.709 ± 2.013	57.333 ± 1.320
	650°C	6.989 ± 0.297	42.192 ± 1.769	64.598 ± 0.222
$45P_2O_5$-40CaO-15Na_2O	500°C	19.687 ± 0.421	44.198 ± 1.142	56.261 ± 4.043
	550°C	8.141 ± 0.297	42.070 ± 3.203	67.872 ± 2.964
	600°C	5.33 ± 0.518	45.293 ± 0.657	64.172 ± 3.318
	650°C	7.742 ± 0.256	48.682 ± 2.027	67.116 ± 5.366

ภาคผนวก จ

เส้นผ่านศูนย์กลางของรูพรุน (Pores size)

แสดงเส้นผ่านศูนย์กลางของรูพรุนขนาดใหญ่ (Pore size) ของเม็ดสารเซรามิกแคลเซียมฟอสเฟตที่มีรูพรุน ในอัตราส่วนต่างๆ

Conditions	Sintering temperature	Pore size (μm)		
		Compositions (glass : camphor)		
		10:0	7:3	5:5
$45P_2O_5$-32CaO-23Na_2O	500°C	-	227.310 ± 72.062	200.449 ± 101.94
	550°C	-	239.803 ± 65.956	191.846± 56.449
	600°C	-	217.340 ± 64.523	187.83 ± 51.625
	650°C	-	325.646 ± 111.719	243.281 ± 88.303
$45P_2O_5$-36CaO-19Na_2O	500°C	-	297.136 ± 60.250	325.247 ± 117.595
	550°C	-	244.723 ± 66.119	241.006 ± 97.959
	600°C	-	228.465 ± 83.994	248.262 ± 97.426
	650°C	-	270.251 ± 119.126	245.762 ± 84.395
$45P_2O_5$-40CaO-15Na_2O	500°C	-	274.853 ± 142.298	373.612 ± 91.257
	550°C	-	238.698 ± 92.273	263.240 ± 78.766
	600°C	-	269.876 ± 97.186	290.432 ± 85.888
	650°C	-	284.563 ± 122.195	381.753 ± 102.917

ภาคผนวก ฉ

สมบัติเชิงกลแรงกด (Compressive stress)

แสดงค่าสมบัติเชิงกลแรงกด (Compressive stress) ของเม็ดสารเซรามิกแคลเซียมฟอสเฟตที่มีรูพรุนในอัตราส่วนต่างๆ

Conditions	**Sintering temperature**	**Compressive stress (MPa)**		
		Compositions (glass : camphor)		
		10:0	**7:3**	**5:5**
$45P_2O_5$-32CaO-$23Na_2O$	500°C	3.026	1.796	1.093
	550°C	10.077	1.419	2.017
	600°C	14.526	2.512	7.077
	650°C	7.809	2.096	2.929
$45P_2O_5$-36CaO-$19Na_2O$	500°C	3.110	1.250	2.503
	550°C	3.679	0.907	1.141
	600°C	4.019	2.641	1.459
	650°C	6.103	3.234	0.961
$45P_2O_5$-40CaO-$15Na_2O$	500°C	3.125	2.050	1.324
	550°C	5.734	1.713	0.550
	600°C	4.234	1.338	0.595
	650°C	4.414	1.972	0.778

ภาคผนวก ช

ผลงานทางวิชาการที่เผยแพร่

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Department of Pharmaceutical Technology, Faculty of Pharmacy, Silpakorn University

HP-04 **Chlorhexidine Thermosensitive Gels**
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Watchara Chokwiwat
National Metal and Materials Technology Center

HP-06 **Antimicrobial Acitivty of Gentamicin Loaded 3DP Calcium Phosphate Sphere**
Phetrung Phanphiriya
National Metal and Materials Technology Center

HP-07 **Development of *Centella asiatica* (Linn) Urban Silicone Transdermal Patch for Wound Healing**
Panee Sirisa-ard
Faculty of Pharmacy, Chiang Mai University

HP-08 **Synthesis and Characterization of Glycolide - L-Lactide - ε-Caprolactone Terpolymers: Influence of Sequential Monomer Addition on Chain Microstructure**
Wichaya Kalaithong
Biomedical Polymers Technology Unit, Department of Chemistry, Faculty of Science, Chiang Mai University

HP-09 **Phase Formation in P_2O_5-CaO-Na_2O Glass Ceramics**
Sutatip Thonglem
Department of Physics and Materials, Faculty of Science, Chiang Mai University

HP-10 **Dynamic DSC Polymerization of ε-Caprolactone Using Stannous Octoate as Initiator**
Wanich Limwanich
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HP-12 **Effect of Chitosan Hydrochloride on *in vitro* Permeation of Theophylline through Porcine Colon**
Uraiwan Pongpiriyajit
Department of Pharmaceutical Technology, and Pharmaceutical Biopolymer Group, Faculty of Pharmacy, Silpakorn University

HP-13 **Novel Tin (II) Alkoxide-Oligoester Adducts as Macroinitiators for the Ring-Opening Polymerisation of Cyclic Esters**
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HP-14 **PET Surface Immobilized with N-Cadherin Mediated Cyclic Peptide for Stem Cell Culture**
Suttinee Poolsup
Program in Biotechnology, Faculty of Science, Chulalongkorn University

Phase formation in P_2O_5-CaO-Na_2O glass ceramics

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Abstract

This project is aimed at producing calcium phosphate glass ceramic for bone substituting applications. Effects of calcium content on the thermal parameters, physical properties, phase formation and microstructures of P_2O_5-CaO-Na_2O glass ceramics were studied. Three glass compositions of fixed P_2O_5 content (as shown in Fig.1) were prepared by conventional melt quenching method at 1200 °C. Thermal parameters of each glass were studied by differential thermal analysis (DTA). The glass powder was pressed into pellets and subsequently sintered at 550, 600 and 650 °C. After that, the density of the glass ceramic samples was measured by Archimedes's method. Phase investigation and micro structural study were performed by XRD and SEM, respectively. The DTA traces provided heat treatment conditions in a range of 500-650°C. XRD results revealed that β-$Ca_2P_2O_7$, CaP_4O_{11} and $NaPO_3$ were formed in the glass ceramics.

Keywords: *Calcium phosphate; Glass ceramic; bone substituting*

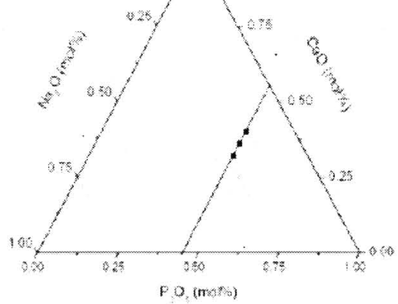

Fig.1. Ternary phase diagram of P_2O_5-CaO-Na_2O glass system.

1. Introduction

Calcium phosphate glasses have a potential use as bone substituting applications, because of their chemical composition is closely similar to that of natural bone. They have bioresorbable property enable this glass to be dissolved in physical fluids. The implant glass can be slowly replaced by regenerate tissue and have good biocompatibility and non toxicity [1-2]. Calcium phosphate glasses from a ternary P_2O_5-CaO-Na_2O have been also studied by Uo et al. [3]. They reported the properties and cytotoxicity of water soluble. Ahmed et al. [4-5] also studied about characterization of this glass and found the suitable composition of P_2O_5 more than 40 mol%, where the thermal parameters decreased with increasing CaO content and these glasses can dissolve in water. The good range of glasses was also found at 45 mol% of P_2O_5 content. It was easily melted and had good biocompatibility. Moreover it possessed low melting point, low glass transition, low softening point, low crystallization temperature and consequently low processing temperature [6]. In this project, effects of calcium content on the thermal parameters, physical properties, phase investigation and microstructure of P_2O_5-CaO-Na_2O glasses and glass ceramics with fixed amount of 45% P2O5 and various ratios of CaO:Na2O were investigated.

2. Experimental procedures and method

2.1 Glass melting

The calcium phosphate glasses were prepared using $(NH_4)_2HPO_4$, $CaCO_3$ and Na_2CO_3 in three compositions of $45P_2O_5$-32CaO-$23Na_2O$, $45P_2O_5$-36CaO-$19Na_2O$ and $45P_2O_5$-40CaO-$15Na_2O$ as shown in Fig.1. The glasses were prepared by conventional melt quenching method at melting temperature of 1200°C for 1 hour in alumina crucible and glass melts were quenched between stainless steel plates at room temperature. The phosphate glass powders were prepared by milling machine and this powder were studied by differential thermal analysis (DTA) using Al_2O_3 as reference.

2.2 Glass ceramic preparation

The powders were pressed into pellets of 15 mm in diameter and 1 gram in weight and were subsequently sintered at 500-650°C for 2 hours. The density of glass ceramics was measured via Archimedes's method using ethyl-alcohol as a medium because this glass system can be dissolved in water. Phase formation and microstructures of the resulting glass ceramics were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively.

3. Results and discussion

Calcium sodium phosphate glasses and glass ceramics sintered at different temperatures are shown in Fig. 2. All glasses are transparent while all glass ceramics are opaque having white in color.

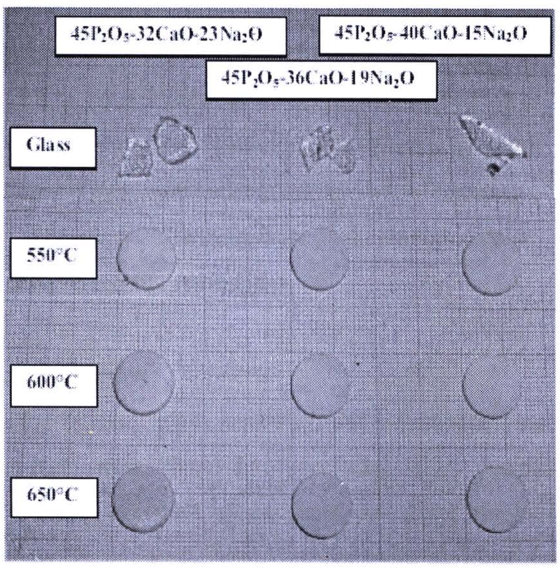

Fig.2. Glasses and glass ceramics from P_2O_5-CaO-Na_2O system sintered at 550-650°C.

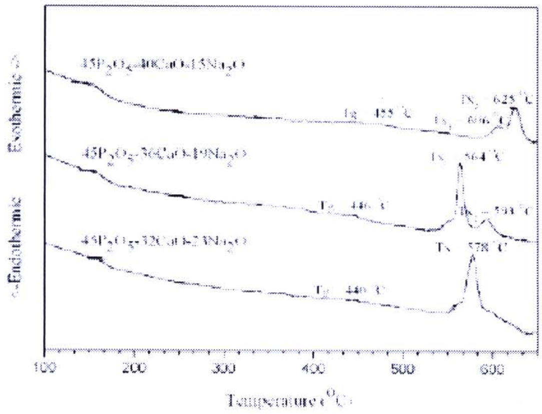

Fig. 3. DTA traces of P_2O_5-CaO-Na_2O glasses.

DTA traces in Fig 3 reveals that these glasses have low glass transition temperature and low crystallization temperature. Crystallization temperatures were found in a range of 550-650°C depending on each composition.

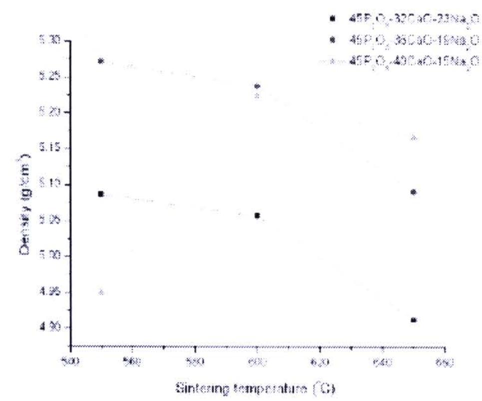

Fig. 4. Density of P_2O_5-CaO-Na_2O glass ceramics at different sintering temperatures.

The relationships between density and sintering temperature of the glass ceramic with different compositions are graphically illustrated in Fig. 4. It can be seen the trends of the glass ceramics with 32 and 36 mol%CaO are similar while that of the glass ceramic with 40 mol%CaO exhibits differently. For the glass ceramic with lower CaO content (32 and 36 mol%), the density decreases with increasing sintering temperature. This implies that the crystallization processes of these glasses at the temperature between 550-600°C, involves the mass transport of many atoms for formation of crystal phase, leaving many free volumes and pores in the glass matrix, thus caused the reduction in bulk density of the sintered pieces. While the glass ceramics with 40 mol%CaO content, has a higher range of crystallization temperatures between 606-630°C, therefore at 600°C the sintering process had no effect from crystallization, giving rise to the proper mechanism in densification of the sintered piece with the highest value of density. Further increase of sintering temperature to 650°C then minimized the density value of the glass ceramics as a result of crystallization.

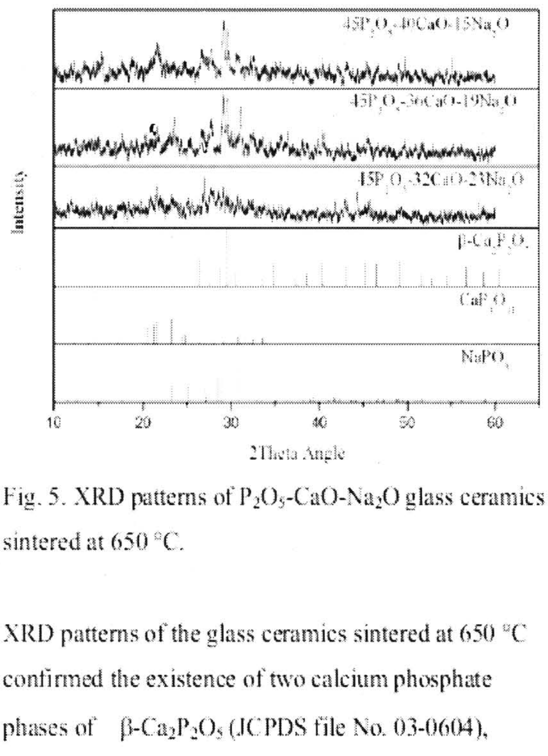

Fig. 5. XRD patterns of P_2O_5-CaO-Na_2O glass ceramics sintered at 650 °C.

XRD patterns of the glass ceramics sintered at 650 °C confirmed the existence of two calcium phosphate phases of β-$Ca_2P_2O_5$ (JCPDS file No. 03-0604), CaP_4O_{11}(JCPDS file No. 70-2085) and one sodium phosphate phase of $NaPO_3$ (JCPDS file No. 11-0650). These phases were found to increase when increasing CaO content which make these glass ceramics suitable in bioresorbable applications.

5. Conclusion

Glasses and glass ceramics from P_2O_5-CaO-Na_2O system were successfully produced. DTA analysis revealed that these glasses have low glass transition temperature, crystallization temperature and melting temperature due to the high content of P_2O_5 which acts as a good glass former in the glass structure. Phase identification study revealed the formation of β-$Ca_2P_2O_5$, CaP_4O_{11} and $NaPO_3$ phases which play an important part in controlling the ceramic and bioresorbable properties of these glass ceramics.

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ผลงานทางวิชาการ

National Conference

1. **S. Thonglem**, N. Raengthon, K. Pengpat, G. Rujijanagul and T. Tunkasiri, Fabrication of Artificial Bone from Calcium Sodium Phosphate glass system, The 35th Congress on Science and Technology of Thailand (STT 35), Chonburi (Bangsaen), Thailand, 15-17 October (2009). (poster presentation)

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