PROCCESSING CERATE-ZIRCONATE NANOPOWDER AT SUPERCRITICAL CONDITIONS VIA BATCH-WISE REACTOR SYSTEM

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Contents

2. Letter of Offer (Research Grant)

UNIVERSITI TEKNOLOGI MARA

Surat Kami : 600-RMI/ERGS 5/3 (8/2013)
Tarikh : 29 Mei 2013

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Puan

KELULUSAN SKIM GERAN PENYELIDIKAN EKSPLORATORI (ERGS) FASA 01/2013

Tajuk Projek	:	*Processing Cerate-Zirconate Nanopowder At Supercritical Conditions Via Batch-Wise Reactor System*
Kod Projek	:	600-RMI/ERGS 5/3 (8/2013)
No. Rujukan Penaja	:	ERGS/1/2013/STG05/UITM/02/03
Bidang	:	*Pure and Applied Science (Materials Science)*
Tempoh	:	01 Jun 2013 - 31 Mei 2016 (36 bulan)
Peruntukan Diluluskan (KPT)	:	RM113,000.00
Peruntukan Pengurusan	:	RM5,650.00 (5%)
Peruntukan Pengoperasian	:	RM107,350.00 (95%)
Ketua Projek	:	Dr. Nafisah Osman

Dengan hormatnya perkara di atas adalah dirujuk.

2. Sukacita dimaklumkan pihak Kementerian Pengajian Tinggi melalui surat JPT.S(BPKI)2000/09/01/018Jld.6(12) yang bertarikh 9 Mei 2013 telah meluluskan kertas cadangan penyelidikan puan untuk dibiaya di bawah Skim Geran Penyelidikan Eksploratori (ERGS) Fasa 01/2013.

3. Bagi pihak Universiti kami mengucapkan tahniah kepada puan kerana kejayaan ini dan seterusnya diharapkan berjaya menyiapkan projek ini dengan cemerlang.

4. Peruntukan kewangan akan disalurkan melalui tiga (3) peringkat berdasarkan kepada laporan kemajuan serta kewangan yang mencapai perbelanjaan lebih kurang 50% dari peruntukan yang diterima.

Peringkat Pertama	20%
Peringkat Kedua	40%
Peringkat Ketiga	40%

5. Untuk tujuan mengemaskini, pihak puan adalah diminta untuk menandatangani perjanjian ERGS, melengkapkan semula kertas cadangan penyelidikan, mengisi borang setuju terima projek penyelidikan dan menyusun perancangan semula bajet yang baru seperti yang diluluskan. Sila lihat lampiran bagi tatacara tambahan pengurusan projek.

Sekian, harap maklum.

"SELAMAT MENJALANKAN PENYELIDIKAN DENGAN JAYANYA"

Yang benar

PROFESOR DR. ABU BAKAR ABDUL MAJEED
Penolong Naib Canselor (Penyelidikan)

: Dilampirkan

Penolong Naib Canselor (Penyelidikan) : 603-5544 2094/2095
Bahagian Penyelidikan : 603-5544 2097/2091/2098/2101/5521 1462/1463
Bahagian Perundingan : 603-5544 2100/2787/2092/2093
Bahagian Inovasi : 603-5544 2750/2747/2748
Bahagian Penerbitan : 603-5544 1425/2785
Bahagian Sokongan ICT : 603-5544 3097/2104/5521 1461
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Penolong Pendaftar : 603-5544 2090
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5.2 Enhanced Executive Summary

This study reports on the reactivity study of parameter that comprises different reaction temperature, reaction pressure and solvent in synthesizing of $BaCe_{0.54}Zr_{0.36}Y_{0.1}O_{2.95}$ (BCZY) using a supercritical fluid method (SCF). Then the best parameter was selective prepared by a sol-gel assisted supercritical fluid method (SGSC) using Brij97 as surfactant. In this work, five samples with different reaction pressure and reaction temperature of BCZY were used and denoted as sample S4, S8, C6, P4 and P5 respectively. Furthermore, six different of solvent namely ethanol, methanol, hexane, pentane, dichloromethane and acetone were used as reaction medium and denoted as A1, A2, A3, A4, A5 and A6. Ethanol solvent was selective as reaction medium prepared BCZY using SGSC with constant reaction pressure and reaction temperature (2MPa, 150°C) with different heating processes (150 °C, 200 °C & 325 °C) respectively denoted as sample B1, B2 and B3. The powders of all samples were calcined at temperature of 1100 °C for 10 hours in an air. The phase formation of the samples was identified by X-ray diffractometer (XRD). The analysis of room temperature XRD data revealed that all samples using SCF method exhibit a mixture of respective $BaZrO_3$, $BaCeO_3$, CeO_2, BaO_2 and $BaCO_3$. However, SGSC method showed a well develops single-phase perovskite-type oxide of BCZY compound. The peaks can be indexed to (110), (200), (211), (220), (310) and (222) that belong to the BCZY phase. No additional reaction products or secondary phases were observed indicating that up to 1100 °C. Thus, it found that the SGSC method is successful produce a single phase of BCZY compare to SCF method.

5.3 Introduction

Protonic conductors of ceramic perovskite-type oxides (ABO_3) have captured attention of researchers worldwide for technological purpose in fuel cells, batteries, steam electrolyser, etc. The best candidate amongst them is the perovskite-type oxides of $Ba(Ce,Zr)O_3$ which is known to exhibit high mechanical and chemical stability along with good conductivity. This proton conductor is the best applicant as electrolyte for solid oxide fuel cells (SOFC) in the presence of hydrogen and/or water vapour at intermediate temperatures (400–750 °C)

There are very limited information available regarding the durability of cells based on the cerate-zirconate, for example, Y-doped $Ba(Ce,Zr)O_3$ electrolyte. Indeed, synthesizing cerate-zirconate electrolyte especially supercritical fluid method is a critical challenge as previous report has found that only 2-3 metal element can be obtained a single perovskite phase. However, to the best of our knowledge this route has not yet been carried out to synthesize ceramics compounds with four metal elements

In the presence work, high-pressure–high-temperature (HP-HT) batch-wise reactor system was used to synthesise $BaCe_{0.54}Zr_{0.36}Y_{0.1}O_{2.95}$ (BCZY) perovskite powder using different parameter such as reaction pressure, reaction temperature and solvent as SCF media. Considering the advantages of Wet Chemical Method (WCMs) and SCF properties, an approach towards the combination of both synthesis routes was used in this work. BCZY was synthesise by sol-gel assisted supercritical fluid method using different heating proceses. The phase formation was studied to investigate the formation of any secondary using X-ray diffractometer (XRD).