SYNTHESIS OF Na_xCoO_2 THERMOELECTRIC VIA CITRATE-NITRATE AUTO COMBUSTION REACTION

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This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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I hereby, declared this report entitled "Synthesis of Na_xCoO_2 Thermoelectric Via Citrate-Nitrate Auto Combustion Reaction" is the result of my own research except as cited in references.

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Date : 1 July 2017

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee are as follow:

…….....

(Dr. Mohd Shahadan Bin Mohd Suan)

ABSTRACT

This study is regarding the synthesis of Na_xCoO_2 thermoelectric via citrate-nitrate auto combustion reaction method. To get a better purity of Na_xCoO_2 the composition of Na is being controlled which is ratio from x = 0.1 to 0.9M. Precursor gels of the composition of Na_xCoO_2 is being heated until it achieves the auto combustion reaction which turned the precursor gels into ashes. After that, the ashes are calcined at 900°C. Then the powder is continued to be pelletized by using compressive machine which is pressed at 14.2 MPa. The size of the pellet is being fixed which is about 10 mm thickness x 2mm diameter. The characterization of the Na_xCoO_2 is observed by using the x-ray diffraction (XRD) are used to get the crystallinity structure and study the composition of sodium in the Na_xCoO_2 and to analyze the surface morphology of the Na_xCoO_2 by means of scanning electron microscopy (SEM). At ratio x = 0.1, 0.3 and 0.5 shows the hexagonal structure while x = 0.7 and 0.9 look plate-like regions. Thermogravimetric analysis and differential thermal analysis (TG-DTA) were observed that the precursors gels with ratio x = 0.7 decomposed in a single-step reaction combusted at temperature of around 260 °C. Samples with ratio x = 0.7 appeared in this work to be electrically superconducting as measured using standard four-probe technique.

ABSTRAK

Kajian ini adalah mengenai sintesis Na_xCoO_2 termoelektrik melalui kaedah sitrat-nitrat tindak balas pembakaran auto. Untuk mendapatkan keaslian yang lebih baik Na_xCoO_2 komposisi Na harus dikawal iaitu nisbah dari x = 0.1-0.9M. Komposisi Na_xCoO_2 gel pelopor dipanaskan sehingga ia mencapai reaksi pembakaran auto yang ternyata menukar gel pelopor ke dalam abu. Selepas itu, abu dikalsinasi pada suhu 900 ° C. Kemudian serbuk terus pelletized dengan menggunakan mesin mampatan yang ditekan pada 14.2 MPa. Saiz pelet sedang tetap iaitu kira-kira 10 mm ketebalan x 2 mm diameter. Pencirian Na_xCoO_2 diperhatikan dengan menggunakan pembelauan sinar-X (XRD) digunakan untuk mendapatkan struktur penghabluran dan mengkaji komposisi natrium dalam Na_xCoO_2 dan untuk menganalisis morfologi permukaan Na_xCoO2 melalui mikroskop elektron pengimbas (SEM). Pada nisbah x = 0.1, 0.3 dan 0.5 menunjukkan struktur heksagon manakala x = 0.7 dan 0.9 struktur seperti bentuk kawasan plat. Analisis termogravimetri dan analisis terma pengkamiran (TG-DTA) diperhatikan bahawa gel pelopor dengan nisbah x = 0.7 reput dalam satu tindak balas satu langkah dibakar pada suhu sekitar 260 ° C. Sampel dengan nisbah x = 0.7 dalam eksperimen ini muncul sebagai superkonduktor elektrik seperti yang diukur menggunakan teknik biasa empat siasatan.

DEDICATION

To everyone that contributes to this research, my family and my friends that has been helping me all day along

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LIST OF ABBREVIATIONS

Na_xCoO_2	-	Sodium cobalt oxide
PbTe	-	Lead telluride
XRD	-	X-Ray Diffraction
TGA	-	Thermogravimetric Analysis
DTA	-	Differential Thermal Analysis
FESEM	-	Field Emission Scanning Electron Microscope
Bi_2Te_3	-	Bismuth Telluride
Na	-	Sodium
ZT	-	Figure of merit
SSR	-	Solid state reaction
CoO_2	-	Cobalt oxide
MSS	-	Molten salt synthesis
BDS	-	Broadband dielectric spectroscopy
CSD	-	Cambridge Structural Database
$NaNO_3$	-	Sodium Nitrate
$Co(NO_3)_2$	-	Cobalt Nitrate
$C_6H_8O_7$	-	Citric Acid
NH_4OH	-	Ammonia solution

LIST OF SYMBOLS

S	-	Seebeck coefficient
ΔT	-	Temperature changes between the ends of the material
ΔV	-	Potential change
$\mu V/K$	-	Micrometer Voltage per Kelvin
T_H	-	Temperatures at the hot side
T_C	-	Temperatures at the cold side
σ	-	Electrical conductivity
$\boldsymbol{\lambda}$	-	Thermal conductivity
g/mol	-	Gram per Mol

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter discussed about the general idea of the thermoelectric from which include the background study, problem statement, objective, scope of study and significant of research.

1.2 Background of Study

Many of waste energy such as home boiler, motorized exhaust, and manufacturing developments can be produced using thermoelectric which is convert heat energy to electrical energy. However, a revolution of courtesy in thermoelectric started popular in the middle of 1990s after theoretical estimates that thermoelectric effectiveness might be significantly improved over nanostructure engineering. Thus, the experimental hard work is done to show the proof-of-principle and high-efficiency materials (Chen, 2003). Therefore, this energy can be used to conduct the electricity by using the thermoelectric materials. In this study, ceramic oxide is used as the raw materials which is sodium cobalt oxide (Na_xCoO_2). These materials show the

behaviour of non-toxicity compared to the conventional thermoelectric material which using toxicity materials such as lead telluride (PbTe). By considering the time and cost factors, suitable synthesis method is important to synthesis Na_xCoO_2. Thus, the suitable method that involve in this project is using the citrate-nitrate auto combustion reaction. One of the benefits of this technique were the materials have improved the limitation of stoichiometry. Next, the final oxide results of crystalline dimensions are invariably in the nanometer range which having high contact of surface area. Characterisation of the Na_xCoO_2 materials is test in variable type of characterisation machine such as X-Ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Differential Thermal Analysis (DTA), and Scanning Electron Microscope (SEM). Therefore, the behaviours of Na_xCoO_2 is justified.

1.3 Problem Statement

Thermoelectric materials have attracted more attentions due to their functions of transforming thermal energy into electrical energy. Thus, it will be resulting from their Peltier and Seebeck effects. The conventional thermoelectric materials such as bismuth telluride (Bi_2Te_3) and lead telluride (PbTe) could have a negative influence on the environment because of their toxicity. Thus, Na_xCoO_2 which is ceramic oxide has been selected. However, the thermoelectric properties of Na_xCoO_2 is very much influenced by the compositions of Na. Hence, the suitable synthesis method needs to be employed to achieved Na_xCoO_2 with expected compositions. Therefore, an appropriate technique is required in controlling Na compositions to get better resistivity. Various method was used to synthesis Na_xCoO_2 such as solid state reaction, hydrothermal and where the auto-combustion reaction attracted much attention. However, conventional process such as solid state reaction (SSR) is difficult to synthesis a sintered body with high crystallographic and orientation due to higher reaction temperature. Therefore, the advantage of using the auto-combustion reaction were low cost and low temperature process. It's also have a better control of stoichiometry which is more purity compared to the others method.

1.4 Objective

1. To synthesis Na_xCoO_2 thermoelectric via citrate-nitrate auto combustion reaction.
2. To evaluate the thermal and electrical properties of Na_xCoO_2.
3. To investigate the effects of Na composition in Na_xCoO_2 towards thermoelectric properties**.**

1.5 Scope of The Study

This research scopes are synthesizing and characterizing of Na_xCoO_2 produced by citrate-nitrate auto combustion reaction. The important parameter of this project is Na composition which is from 0.1 to 0.9M. Next, the combustion reaction of the sample was evaluated by thermal behaviour of the Na_xCoO_2 of as prepared gels at increasing temperature. It is determining by using the DTA and TGA machine. Moreover, the structural properties of Na_xCoO_2 sample such as lattice constants, crystallite size, elements, microstructure and surface morphology were characterized by using multiple technique to investigate each parameter of the materials. It is used the SEM and XRD analysis to study the properties of Na_xCoO_2.

1.6 Significant of Research

This research presents a method for synthesizing the thermoelectric materials. The method of citrate-nitrate auto combustion reaction consumed less time and energy compared to other conventional method for processing of composite superconductor oxides. As consequence, the properties of Na_xCoO_2 become better.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This section discussed about the fundamental of Na_xCoO_2 thermoelectric via citrate-nitrate auto combustion reaction process. In this topic, it will elaborate more about the structure and properties that need to achieve the objective and relate to the method that used in this experiment. Thus, the chapter is divided into some subchapters that required to make the preparation of sample and other related process involved.

2.2 Thermoelectric

Currently, the mankind claim used for energy is causing affected growth of social also politically conflict. Then, the ecological effect of worldwide weather changes due to the burning of remains oils rapidly increases. A method to recover the renewable of our power base is over the seeking of unused heat energy with thermoelectric makers. A large amount of unused heat such as automotive exhaust, industrial processes and home heating can be produced using thermoelectric which will convert heat energy to electrical energy. Thermoelectric makers remain solid-state tools with no moving component which is noiseless, consistent and accessible that

creating them perfect for delivered power generation (Rowe, 1995). By now many attempt is in progress to change the alternator in automobiles with a thermoelectric maker attached on the exhaust part so that developing fuel effectiveness. Developments in thermoelectric might also allow the restoration of compression-based refrigeration by solid-state Peltier coolers (DiSalvo, 1999) . Thus, thermoelectric keep on as well efficient to be useful in most applications (Rowe, 1995) as shown in Figure 2.1. Yet, a change of attention in thermoelectric started in the middle of 1990s after theoretic expectation recommended that thermoelectric effectiveness might be significantly improved completely by nanostructure engineering, which related to experimental works to show the proof-of-principle and high-efficiency materials (Chen, 2003). Simultaneously, complex bulk materials such as skutterudites (Uher, 2001), clathrates (Nolas, 2006) and zintl phases (Kauzlarich, 2007) have been discovered. Its discovered that high productivities can be gotten. At this point, we analysed these new developments used for seeing in what way disarrangement and complication in the unit cell together with nanostructured materials can guide to improve effectiveness of the materials. Thus, it allows us to discover mutual behaviours in these materials and extract realistic project strategies used for the finding of materials with great thermoelectric effectiveness.