ENHANCED THERMOELECTRIC PROPERTIES OF P-TYPE CALCIUM COBALT OXIDE VIA ALUMINUM, Al-Ni AND POROUS Al-Ni CO-DOPINGS

MOHAMMED MUSTAFA ABEDLHAFD

UNIVERSITI TEKNOLOGI MALAYSIA

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MOHAMMED MUSTAFA ABEDLHAFD ALHWANI

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Faculty of Engineering
Universiti Teknologi Malaysia

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Oxide-based thermoelectric (TE) materials are considered as one of the most eco-friendly materials for power generation applications. They seem more stable and less toxic compared to the conventionally used metals and semiconductors such as PbTe. Among the TE materials, the p-type $Ca_3Co_4O_9$ is the most commonly investigated due to its high potential to be used in high temperature power generation. Many attempts have been carried out to further increase the generating power of this TE material which includes doping and co-doping with other elements. However, most of the literature reported that their figure of merit (*ZT*) is still lower than 0.12. This research aimed to enhance the thermoelectric properties of $Ca_3Co_4O_9$ via doping and co-doping with metal elements and increasing its bulk porosity. The stoichiometric mixtures of $Ca(NO_3)_2.4H_2O$, $Co(NO_3)_2.6H_2O$ and starch were first dissolved in the distilled water. The resultant gel was decomposed at temperature 673K until it changed into a black precursor. The black precursor was then calcined in the furnace at 773, 873, 973, and 1073 K for 4, 6, 8, 10, 12 and 14 h to obtain $Ca_3Co_4O_9$ powder. The $Ca_3Co_4O_9$ pellet was prepared using a 5 tons hydraulic press, then sintered in air at 1173K for 20h. The best properties of $Ca_3Co_4O_9$ powder was then doped with Aluminum (Al). The amount of doping was varied at x = 0.1, 0.2, 0.3 and 0.4 on $Ca_{3-x}Al_xCo_4O_9$ powder. The figure of merit, *ZT* values were measured on doped $Ca_3Co_4O_9$ powders and compared. The highest *ZT* value of the doped $Ca_3Co_4O_9$ was selected for the next co-doping with Nickel (Ni) varied at y = 0.05, 0.1, 0.15 and 0.2. The highest *ZT* value obtained from Ni co-doped powder was selected and then their porosity was varied by adding starch at different weight percentages of 3, 5 and 7 wt%. $Ca_3Co_4O_9$ synthesis and treatments were analyzed using a thermogravimetric analyzer (TGA), scanning electron microscope equipped with energy dispersive spectrometry (SEM-EDS), x-ray diffractometer (XRD), X-ray photoelectron spectroscopy (XPS) and transmission electron microscope (TEM). The changes in thermoelectric properties were measured from 300 to 700K. The calcination process that produced the best lattice parameters ($b_1/b_2 = 1.613$) and crystal size (458 ˚A) were observed at 12h. The highest *ZT* for Al-doped was 0.139 for x = 0.1 at 700 K. While the highest *ZT* for $Ca_{3-x}Al_xCo_{4-y}Ni_yO_9$ was 0.145 at x = 0.1 and y = 0.05. An investigation was conducted on the increasing structure porosity with Al-Ni co-doping. The highest figure of merit *ZT* was 0.161 at 700K, for the Al-Ni co-doped sample with 5wt%. The results revealed that the doping elements have a significant effect on the microstructure properties and morphology of polycrystalline of $Ca_3Co_4O_9$, such as grain size reduction up to 1.76 µm for the best co-doped. These changes improve the overall thermoelectric properties.

ABSTRAK

Bahan termoelektrik (TE) berasaskan oksida dianggap sebagai salah satu bahan yang paling mesra alam sekitar untuk aplikasi penjanaan kuasa. Ia didapati lebih stabil dan kurang bertoksik jika dibandingkan dengan logam dan semikonduktor yang digunakan secara konvensional, seperti PbTe. Dari kalangan bahan TE, bahan jenis-p $Ca_3Co_4O_9$ adalah yang paling banyak dikaji disebabkan ia mempunyai potensi yang tinggi untuk digunakan dalam penjanaan kuasa bersuhu tinggi. Banyak usaha telah dijalankan untuk meningkatkan lagi kuasa penjanaan bahan TE ini, termasuklah pengedopan dan pengedopan-bersama dengan unsur-unsur lain. Walau bagaimana pun, kebanyakan literatur telah melaporkan bahawa angka merit (*ZT*) bagi bahan ini masih rendah iaitu kurang daripada 0.12. Penyelidikan ini bertujuan untuk meningkatkan sifat thermoelektrik $Ca_3Co_4O_9$ melalui pengedopan dan pengedopan-bersama dengan unsur logam dan meningkatkan keliangan pukalnya. Campuran stoikiometri $Ca(NO_3)_2.4H_2O$, $Co(NO_3)_2.6H_2O$ dan kanji dilarutkan terlebih dahulu dalam air suling. Gel yang diperoleh diurai pada suhu 673K sehingga ia bertukar kepada prapenanda hitam. Prapenanda hitam tadi kemudiannya dikalsin di dalam relau pada suhu 773, 873, 973, dan 1073 K selama 4, 6, 8, 10 12 dan 14 jam untuk menghasilkan serbuk $Ca_3Co_4O_9$. Pelet $Ca_3Co_4O_9$ telah disediakan dengan menggunakan sebuah penekan hidraulik 5 tan, kemudiannya ia disinter di udara pada 1173K selama 20 jam. Sifat terbaik serbuk $Ca_3Co_4O_9$ kemudiannya didop dengan unsur Aluminium (Al). Jumlah unsur dopan diubah pada x = 0.1, 0.2, 0.3 and 0.4 ke atas serbuk $Ca_{3-x}Al_xCo_4O_9$. Angka merit, *ZT* telah diukur ke atasserbuk $Ca_3Co_4O_9$ dan dibandingkan. Nilai *ZT* tertinggi pada $Ca_3Co_4O_9$ yangtelahdidopdipilihuntukpengedopan-bersama dengan Nikel (Ni) yang diubah pada y = 0.05, 0.1,0.15 dan 0.2. Nilai *ZT* tertinggi yang diperoleh daripada serbuk dop-bersama Ni dipilih dan setelah itu keliangannya diubah dengan menambah kanji pada peratusan berat yang berbeza, iaitu 3%, 5% dan 7%. Sintesis dan rawatan $Ca_3Co_4O_9$ telah dianalisa dengan penganalisis permeteran graviti haba (TGA), mikroskop imbasan elektron yang dilengkapi dengan spektroskopi serakan tenaga (SEM-EDS), pembelauan sinar-X (XRD), spektroskopi fotoelektron sinar-X (XPS) dan mikroskop elektron transmisi (TEM). Perubahan dalam sifat termoelektrik telah diukur dari 300 hingga 700K. Proses kalsinasi yang menghasilkan parameter kekisi (b_1/b_2 = 1.613) dan saiz hablur (458 ˚A) yang terbaik telah diperhatikan pada 12 jam. ZT tertinggi untuk dop-Al adalah 0.139 untuk x = 0.1 pada 700K. Sementara *ZT* tertinggi untuk $Ca_{3-x}Al_xCo_{4-y}Ni_yO_9$ adalah 0.145 pada x = 0.1 dan y = 0.05. Kajian telah dilakukan terhadap peningkatan struktur keliangan dengan dop-bersama Al-Ni. Angka merit *ZT* yang tertinggi tercapai adalah 0.161 pada 700K, untuk sampel Al-Ni dop-bersama pada 5wt%. Keputusan menunjukkan bahawa unsur pengedopan mempunyai kesan yang signifikan terhadap sifat mikrostruktur dan morfologi polihablur $Ca_3Co_4O_9$ seperti pengurangan saiz butir sehingga 1.76 µm untuk dop-bersama yang terbaik. Perubahan ini telah meningkatkan sifat termoelektrik secara keseluruhannya.

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LIST OF ABBREVIATIONS

DOS	-	Density of states
EDS	-	Energy Dispersive X-Ray Spectroscopy.
FIB-SEM	-	Focused Ion Beam – Scanning Electron Microscope
TE	-	Thermoelectric
TEC	-	Thermoelectric Cooling
TEG	-	Thermoelectric Generators
TEM	-	Transmission Electron Microscopy
TGA	-	Thermogravimetry Analysis
TMF	-	Thermoelectromotive Force
VP-SEM	-	Variable Pressure-Scanning Electron Microscopy
XPS	-	X-Ray Photoelectron Spectroscopy
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

$\emptyset$ - Coefficient of performance

A_{am} - Integrated areas of amorphous phases

A_{cr} - Integrated areas of the crystalline phases

c_p - Specific heat capacity

e - Electric charge

E_f - Fermi energy

E_g - Band gap energy

h - Planck’s constant

K_B - Boltzmann constant

k_e - Electronic thermal conductivity

k_l - Lattice thermal conductivity

m^* - Effective carrier mass

m_w - Mass flow rate of the water

Q - Heat transfer rate

Q_C - Cooling rate

Q_H - Thermal power supplied

S - Seebeck

T - Mean temperature

T_c - Cold-side temperature

T_h - Hot-side temperature

W - Power

Xcr - Crystallinity

ZT - Figure of merit

η - Energy conversion efficiency

κ - Thermal conductivity

μ - Mobility

ρ - Electrical resistivity

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter describes the concepts of thermoelectric materials with background development. It is followed by the problem statement that highlighting the research gaps and then the objectives and scopes of the study. Finally, it describes the significance of the research and thesis organization.

1.2 Background of Research

In the field of science and engineering, the thermoelectric devices are widely used for thermoelectric power generation (TEG) and thermoelectric cooling (TEC). The thermoelectric power generators produce electrical energy from the waste heat, thus provide an encouraging solution to the waste heat recovery, and self-powered systems [1-3]. Such devices can also perform Peltier cooling by the reverse process, i.e., generate a temperature gradient when current is applied [4]. These devices have several advantages compared to other energy technologies because they are compact and reliable without moving parts, long life, silent in operation and environmental friendly [5]. Since the inception of the Seebeck effect [6], intensive efforts have been made to get diverse applications of the thermoelectric devices in the technology of cooling and power generation [7].

The mechanism of thermoelectric effect is based on the diffusive transport of the electrons or holes (called charge carriers) in the materials that is driven by an induced electrical potential difference because of the temperature drop between the hot and cold junction. The efficiency of a thermoelectric device depends upon the materials' electrical resistivity (ρ), thermal conductivity (κ) and Seebeck coefficient

(*S*). These three parameters are related through a dimensionless quantity called the figure of merit (*ZT*).

The thermoelectric effect was not fully developed until the second half of the twentieth century despite its discovery in the nineteenth century [8, 9]. Over the years, the thermoelectric power generation and cooling efficiency became feasible [10-12]. However, the low energy conversion efficiency and high capital cost of the thermoelectric technologies compared to their conventional counterparts are the main limitations for the wider applications which need to be resolved. In the Seebeck effect, due to an applied temperature gradient across a material, the more energetic charge carriers are diffused to a lower potential until an equilibrium electric field is established to impede the further flow of the carriers [13]. In fact, this potential difference is used to power an external load. Generally, the Seebeck coefficient is very low for metals (only a few μV/K) but high for semiconductors (typically a few 100 μV/K). The opposite polarity of the thermo-electro-motive force (TMF) generated by the n-type and p-type semiconductors is responsible for doubling the voltage when combined. In practice, a semiconductor element (uncouple) is connected in series to assemble the thermoelectric modules wherein the direct conversion of heat to electricity is called thermoelectric conversion or thermoelectric power generation. To achieve thermoelectric modules with high efficiency, smart materials with large *ZT* remain demanding. Recent researches revealed that various nanostructured (pure or doped) materials can be the potential candidates for making these efficient thermoelectric devices.

In the past, various alloys including SiGe and PbTeSe have served as the thermoelectric materials for automotive applications. Lately, some of the oxide-based materials have emerged as potential candidates for the waste heat conversion due to their promising thermoelectric traits as well as the excellent chemical and thermal stability at high temperatures, oxidation resistance, less toxicity and low cost [14-16]. Nevertheless, owing to their low carrier mobility these oxides were never considered earlier as prospective TE candidates until the high-performance $Na_xCo_2O_4$ appeared [17]. Nowadays, the p-type cobalt-based oxides such as the $Ca_3Co_4O_9$ and $NaCo_2O_4$, are utilized in the TE modulus [18]. Two specific compounds in the low-dimensional structures of the Ca-Co-O cobaltite family (for example $Ca_3Co_2O_6$ and $Ca_3Co_4O_9$) became promising because of their distinct thermoelectric characteristics. In fact, misfit layered $Ca_3Co_4O_9$ material in the single crystal phase reveals excellent thermoelectric performance with ZT ~ 0.3 at 700K [19]. Considering such unique thermoelectric traits of the calcium-cobalt-oxides, the present study intends to further improve their thermoelectric properties by Al/Ni doping in the nanostructured phases.

Over the decades, several techniques have been developed to synthesize $Ca_3Co_4O_9$ powder including the thermal hydro-decomposition [20], pechini [21], polymer solution synthesis [22], and solid-state reaction [23-27]. However, these methods operate at high reaction temperatures and need long time, producing materials with low chemical homogeneity [28]. Previous studies showed that the sol–gel method can produce high-quality $Ca_3Co_4O_9$ powder with fine particles sizes and uniform distribution, leading to improve magnetic, electrical, and optical characteristics desirable for real applications [29-33]. Thus, it is worth to use the combined sol-gel and auto-combustion technique to produce high quality p-type $Ca_3Co_4O_9$ powder with enhanced attributes useful for the thermoelectric applications.

Dedicated attempts have been made to improve the thermoelectric properties of $Ca_3Co_4O_9$ material including the electrical resistivity, thermal conductivity and Seebeck coefficient. Earlier, the polycrystalline $Ca_3Co_4O_9$ samples were partially substituted by other atoms to improve their overall attributes. Theses substitution was added as single element doping such as Sr [34], Pr [35], Ga [36], Ba [25], B [37], Bi [38-40], Cd [41], Ce [42], Cr [43], Er [44, 45], Ho [44], Fe [2], Gd [46], Na [25, 47], Pd [48], Sm [49], Tb [23], Y [50] and Yb [51-53]. Several other studies present a detailed account of the effect of simultaneous substitution of two different elements as co-doping [42, 54, 55]. Despite the improvement in the electrical resistivity and Seebeck coefficient of $Ca_3Co_4O_9$, for practical applications various other properties that remain poor still need enhancement. In addition, the porous $Ca_3Co_4O_9$ samples were shown to have an excellent thermoelectric response [56]. Looking at the significant benefits of the newly emerged cleaner, cheaper, more compact and better electricity technologies compared to the existing ones (for example gasoline cells and lithium batteries) it is necessary to develop nanostructured $Ca_3Co_4O_9$ materials based on thermoelectric power generator systems to solve the waste heat problems efficiently.

1.3 Problem Statement

Several techniques have been developed to synthesize $Ca_3Co_4O_9$ powder such as polymer solution synthesis [22], and solid-state reaction [23-27], while the sol-gel is considered as the most straightforward technique. Generally, the sol-gel synthesis technique uses citric acid and polyethylene glycol to polymerize the solution that produces carbonaceous xerogel and finally needs to be crushed to become powder [3, 32, 41, 42, 57]. Most of the polymerizing agents used in the ceramic powder preparation are acid-based that require special care for handling. In the last 6 years, it was reported the starch can be used effectively as a fuel to produce $Ca_3Co_4O_9$ powder using the sol-gel combustion method [58]. Starch is easy to prepare and environmental friendly. However, they did not evaluate the purity of $Ca_3Co_4O_9$ powder prior to bulk pressing and sintering. The investigated thermoelectric properties were only limited to electrical resistivity and magnetic properties. It is also noticed that the purity of $Ca_3Co_4O_9$ powder is seldom reported in the past literatures regardless of their properties methods, i.e. sol-gel [41, 59], sol-gel combustion method [58] and solid-state [25, 48, 56, 60]. The preparation of bulk samples from these powders at different purity and crystallinity may be the reason causing large variations of ZT value reported in the literature. As a result, no standard reference can be made for comparison since the purity of $Ca_3Co_4O_9$ powder is unknown.

The TE performances of oxide materials can be improved by doping elements [61]. Some researchers doped $Ca_3Co_4O_9$ material either in Ca site like La [62] or in Co site such as Zr [60] as a single doping. Co-doping also has been used to increase ZT, such as Na at Ca site and W at Co site [63]. ZT improvement is a challenging task due to linked of thermoelectric properties to each other [61]. Therefore, the doped element should be selected accurately. In the case of Ca-site, many researchers reported their studies using ions oxidation state 3+, such as Sm^{3+} [64], including rare earth elements like Ho [27]. Despite many studies of doping 3+ ions on Ca-site for $Ca_3Co_4O_9$, there is none reported using Al^{3+}. Furthermore, aluminum has been reported in doping ZnO to reduce density [1] and particle size [65, 66]. The use of aluminum was able to improve thermoelectric properties [67, 68]. The ionic radius of Ca^{2+} and Al^{3+} are 1 Å [63, 69] and 0.53˚A [70], respectively. The difference of radius causes lattice deformation [71], which may have the potential for decreasing both carrier concentration and thermal conductivity [72] in $Ca_3Co_4O_9$. On the other hand, Ni co-doped with Lu [57] and Ce [42] element into $Ca_3Co_4O_9$ material was reported able to improve the *ZT* value after doped at Co site. However, the Al doping in Ca-site, and co-doped Al- Ni (Ni at Co site) to $Ca_3Co_4O_9$ samples have not been reported in the literature. Therefore, their doping effects on the thermoelectric properties performance are still unknown. It was acknowledged that the thermoelectric efficiency of the materials can also be enhanced by selecting suitable dopant elements and by controlling their porosity [73]. The increase of porosity reducing thermal conductivity [74] which subsequently increases ZT value. Starch has been successfully used to produce pores in bulk samples as porous alumina [75] and 3Y–ZrO_2 [76]. The porous $Ca_3Co_4O_9$ samples were synthesized using wood as a porous forming agent [56] which showed ultralow thermal conductivity and high *ZT*. The advantage of using starch instead of wood powder, it offers a low burning temperature (~673K [77] versus ~773K for wood [56]), easily available and cheap. However, no published report presents the performance of starch as a porous forming agent to produce pores in $Ca_3Co_4O_9$ bulk samples.

The relationship of phonon scattering models to their respective material systems is complex and fraught with controversy. Interface and grain-boundaries have been reported to facilitate the scattering effect in the case of mid- and long-wavelength photons, while short-wavelength phonons can be scattered by atomic flaws (e.g. extrinsic dopants) [78]. Scale hierarchical structures were employed in another study to observe that the scattering of short-wavelength phonons occurred at point defects, including substitution atoms, the scattering of mid-wavelength phonons occurred at grain boundaries, displacements and lamellar/multilayer [79]. The electron and phonon path effecting by macroporous structure also modulated to understand the decreasing thermal conductivity after porosity increases [80]. However, there is no literature reported a combined model to represent the effect of grain boundaries, purity and impurity from one set of experiment using the same TE material.

The sol-gel auto-combustion technique was adopted in this study for synthesizing a high purity $Ca_3Co_4O_9$ powder. This technique is much simpler to conduct as compared to polymer solution synthesis and solid-state reaction [64].

1.4 Objectives of the Research

Based on the abovementioned problem statement, the following objectives were set:

i. To evaluate the effect of temperature and heating time on the phase transformation and purity of synthesized $Ca_3Co_4O_9$.
ii. To compare the thermoelectric properties performance of pure, Al-doped, Al-Ni co-doped and porous Al-Ni co-doped of $Ca_3Co_4O_9$ samples.
iii. To propose a revised model for the phonon scattering mechanism in $Ca_3Co_4O_9$ that considering porosity, grain boundary and impurity.

1.5 Research Scopes

The scopes of the research were as follows:

i. Commercially available raw nitrite materials $Co(NO_3)_2.6H_2O$ and $Ca(NO_3)_2.4H_2O$) were used for synthesizing p-type $Ca_3Co_4O_9$ thermoelectric material via the sol-gel auto-combustion method.

ii. Starch $[(C_6H_{10}O_5)_n]$ was used in preparing the slurry for thermoelectric materials and served as a pore-forming agent to produce porous $Ca_3Co_4O_9$ bulk material.

iii. $Ca_3Co_4O_9$ thermoelectric material was doped and co-doped with Al- and Al-Ni elements, respectively. The atomic number of Al doping element was varied in the range between 0.1 to 0.4. While Ni element was varied in the range between 0.05 to 0.2.

iv. The temperature range for evaluating thermoelectric properties of the bulk $Ca_3Co_4O_9$ samples was limited from 300K (27 ˚C) to 700K (423 ˚C). This temperature suits with the temperature range in the automobile exhaust system.

v. The porosity of the Al-Ni co-doped $Ca_3Co_4O_9$ bulk samples was varied using starch at 3%, 5% and 7% wt.

vi. Various analytical equipments were used to characterize the developed thermoelectric materials were characterized using various analytical equipment. These include thermogravimetry analyzer (TGA), particle size analysis, variable pressure scanning electron microscope (VP-SEM), focused ion beam – scanning electron microscope (FIB-SEM), transmission electron microscope (TEM), equipped with energy-dispersive x-ray spectroscopy (EDS), x-ray photoelectron spectroscopy (XPS), and x-ray diffraction (XRD).

vii. A five-tons uniaxial cold-pressing technique was used to prepare bulk size of sample i.e., 15×15×5 mm.

viii. Investigated thermoelectric properties were only limited to figure of merit (*ZT*), Seebeck, electrical resistivity and thermal conductivity.

1.6 Significance of the Research

Ceramic materials are known as non-toxic materials that can be synthesized and modified to become as thermoelectric materials. In this work, starch was used during synthesis as well as in preparing porous bulk $Ca_3Co_4O_9$. It is not only economical, easy to process but also more environmental friendly compare to many other synthesis methods which are toxic and posing safety issues during handily. The enhanced TE performance of $Ca_3Co_4O_9$ is expected to increase the efficiency of waste heat conversion to useful energy which will directly benefit the society in many ways. The fundamental knowledge that generated from this study will provide valuable information on the control of the thermoelectric traits, morphology, and structure of the $Ca_3Co_4O_9$ by adjusting the external parameters.

1.7 Thesis Organization

This thesis consists of five chapters. In the first chapter, the basics of thermoelectricity are introduced in the background and research problem statement explained with objectives as well as the scopes of the research. Chapter two overviews past research and recent developments that devoted to state of the art on thermoelectric oxides, focusing mainly on $Ca_3Co_4O_9$. The experimental procedure, materials as well as analytical equipment used in this research are explained in Chapter 3. Chapter four presents the experimental and analysis of data between treatments. Statistical techniques are used to confirm the variations in the data. Finally, chapter five outlines the conclusions and recommendations of the research work.

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LIST OF PUBLICATIONS

1- Mohammed M. A., I. Sudin., Uday M. B., *Effects of calcination temperature and time on the of* $Ca_3Co_4O_9$ *purity when synthesized using starch-assisted sol-gel combustion method*. Journal of Advanced Ceramics, 2020. 9: p. 162-172 (***ISI, Q1***)

2- Mohammed M. A., I. Sudin., Uday M. B., *Effects of calcination temperature and time on the of* $Ca_3Co_4O_9$ *purity when synthesized using starch-assisted sol-gel combustion method*. Journal of Advanced Ceramics. **Accepted**, (***ISI, Q2***)

3- Mohammed M. A., I. Sudin., Alias Mohd Noor, Srithar Rajoo, Uday M. B., Noor H. Obayes, Muhammad Firdaus Omar, *Investigation on microstructure and electrical properties of Bi doping* $Ca_3Co_4O_9$ *nanoparticles synthesized by sol-gel process*. International Journal of Engineering & Technology, 2018. 7 (2.29) (29): p. 31-33. (***Scopus***)

4- Mohammed M. A., I. Sudin., Alias Mohd Noor, Srithar Rajoo, Uday M. B., Noor H. Obayes, Muhammad Firdaus Omar, *A review of thermoelectric ZnO nanostructured ceramics for energy recovery*. International Journal of Engineering and Technology, 2018. 7, No 2.29 (29): p. 27-30. (***Scopus***)

5- Mohammed M. A., I. Sudin., Alias Mohd Noor, Srithar Rajoo, Uday M. B. 2017. *A review of thermoelectric p-type* $Ca_3Co_4O_9$ *nanostructured ceramics for exhaust energy recovery*. 2nd International Symposium on Engine Boosting and Energy Recovery 2017 (UTM Centre for Low Carbon Transport).