

STRUCTURAL PROPERTIES OF YTTRIUM  
IRON GARNET DOPED CARBON BLACK  
PREPARED VIA SOL-GEL TECHNIQUE

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Thesis submitted in fulfillment of the requirements  
for the award of the degree of  
Bachelor of Applied Science (Honor) Material Technology

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## **SUPERVISORS' DECLARATION**

I hereby declare that I have checked the thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Applied Science (Honor) Material Technology.

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## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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*In dreams or achieve the success of many sacrifices we have to do. My humble effort I dedicate to my sweet and loving father and mother, I could never have done this without you faith, support, and constant encouragement. Thank you for teaching me to believe in y self, in God, and in my dreams. To all my friend and my partner, without whom none of my success would be possible, and along with all hard working and respected lecturers.*

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## LIST OF SYMBOLS

$\sim$	-	approximately
%	-	percent
$\lambda$	-	wavelength
$\mu$	-	micron ( $10^{-6}$ )
$\eta$	-	coulombic efficiency
$2\theta$	-	Bragg angle
$^{\circ}\text{C}$	-	degree celcius
$\text{\AA}$	-	angstrom ( $10^{-10}$ )
$\text{m}^2 \text{ g}^{-1}$	-	meter per gram
$\text{cm}^3 \text{ g}^{-1}$	-	volume per gram
g	-	grams
h	-	hour
$t$	-	time

## LIST OF ABBREVIATIONS

BET	-	Brunauer, Emmet-Teller
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
SEM	-	Scanning Electron Microscope
XPS	-	X-ray Photoelectronic Spectroscopy
XRD	-	X-ray Diffraction
EMI	-	Electromagnetic Interference
PU	-	Polyurathane
MWCNT	-	Multi-Walled Carbon Nanotubes

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## ABSTRACT

There are several issues arise when using inorganic materials, for example, the procedure to generate literal substrate is complex because of many materials needed. It is also very expensive to run this product. Furthermore, the inorganic material is not environmental friendly and can lead to pollution and ecosystem imbalance. To overcome this issue, this agricultural waste such as sawdust can be set to be an important item as important as crude palm oil. Agriculture waste (sawdust) was controlled to be a carbon black by pyrolysis methods and have a good medium to be used as an electromagnetic wave absorber given the nature of the carbon material in maintaining the electromagnetic waves. Adding some rare earth elements and iron can increase the ability of the material to absorb electromagnetic waves. The main objective of this research are to prepare the waste carbon black by using pyrolysis technique and study the physical and structural properties of Yttrium Iron Garnet doped waste carbon black by using X-ray Diffratometry (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscopy (FESEM). The methodology of this study started with the preparation of waste carbon black by using pyrolysis technique. The sample will be mixed with the yttrium iron garnet with the ethanol absolute solution as the bonding agent for the sample. The finish sample then will undergoes characterization. Element of the sample has been analysis in the characterization of X-ray Diffratometry (XRD). Each of the graph of the sample show the present of Yttrium and Ferrite. For Fourier Transform Infrared Spectroscopy (FTIR), showed all powders presented O–H bond stretching from 3100 to 3600  $\text{cm}^{-1}$ , probably due to water absorption during test and the metal-oxygen vibrations at 577  $\text{cm}^{-1}$  which are due to the lattice vibrational modes of the YIG unit cell. For FESEM, the surface and diameter of the particle are strongly depend on their molecular weight.

**Keyword:** Waste Carbon Black, Pyrolysis, Yttrium Iron Garnet

## ABSTRAK

Terdapat beberapa isu timbul apabila menggunakan bahan-bahan bukan organik, sebagai contoh, prosuder untuk menjana substrat literal adalah kompleks kerana banyak bahan yang diperlukan. Ia juga sangat mahal untuk menjalankan produk ini. Tambahan pula, bahan bukan organik tidak mesra alam sekitar dan boleh membawa kepada pencemaran dan keseimbangan ekosistem. Untuk mengatasi isu ini, sisa pertanian ini seperti habuk kayu boleh ditetapkan untuk menjadi bahan penting sama penting dengan minyak sawit mentah. Sisa pertanian (habuk papan) telah dikawal untuk menjadi karbon hitam dengan kaedah pirolisis dan mempunyai medium yang baik untuk digunakan sebagai penyerap gelombang elektromagnet memandangkan sifat bahan karbon dalam mengekalkan gelombang elektromagnet. Menambah beberapa elemen nadir bumi dan ferit boleh meningkatkan keupayaan bahan untuk menyerap gelombang elektromagnet. Objektif utama kajian ini adalah untuk menyediakan sisa karbon hitam dengan menggunakan teknik pirolisis dan mengkaji sifat struktur *Yttrium Iron Garnet* didopkan sisa karbon hitam dengan menggunakan *X-ray Diffractometry* (XRD), *Fourier Transform Infrared Spectroscopy* (FTIR) dan *Field Emission Scanning Electron Microscopy* (FESEM). Metodologi kajian ini bermula dengan penyediaan sisa karbon hitam dengan menggunakan teknik pirolisis. Sampel seterusnya di campur dengan *Yttrium Iron Garnet* dengan larutan etanol sebagai agen ikatan bagi sampel. Akhir sekali, sampel akan melalui proses pecirian. Elemen daripada sampel telah di analisis pencirian *X-ray Diffractometry* (XRD). Setiap graf daripada sampel menunjukkan kewujudan Yttrium dan ferit. Untuk *Fourier Transform Infrared Spectroscopy* (FTIR), semua serbuk menunjukkan kewujudan regangan ikatan O-H dari 3100 sehingga 3600  $\text{cm}^{-1}$ , mungkin disebabkan oleh penyerapan air semasa ujian dan getaran logam oksigen pada 577  $\text{cm}^{-1}$  yang disebabkan oleh kekisi getaran mod unit sel YIG. Untuk FESEM, permukaan dan diameter zarah sangat bergantung kepada berat molekul mereka.

**Kata-kata:** Sisa karbon hitam, Pirolisis, Yttrium Iron Garnet

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 BACKGROUND OF THE RESEARCH**

In recent years, there are an extensive study have been carried out on the natural waste and organic substances. The following studies conducted based on their massive criteria such as low-cost, simple fabrication method, high specific properties biodegradable on nature and environmental friendly. There are many example of the organic substances included a waste paper, palm oil, rice husk and sawdust. A few researches had been conducted by using agriculture waste and had obtained such a valuable result. Some research carried out in the past used wood ash waste as a replacement for cement in concrete mixed (Halima et al., 2013).

Positive physical properties is some of the advantages utilizing sawdust as a woody biomass in fertilizer assembling for example, high porosity, high water retention, moderate water drainage. Because of their properties, the sawdust had been experienced pyrolysis to end up a carbon black. Different materials are utilized to create carbon black and probably the most usually utilize are agriculture waste, for example, coconut shell, pistachio shell, sawdust, and tropical wood.

Carbon black is one of the most important micro porous adsorbents because it adsorption capacity that is so large, the attraction for several organic dissolved and the capability to custom-tailored the specific application (Ismadji et al., 2005). The term "carbon black" refers to a group of industrial products that involve heat, furnace, channel, and acetylene black. They are fundamentally comprised of basic carbon as round particles close colloidal size, converge into particles and agglomerates and totals got by halfway burning or thermal decomposition of hydrocarbons (Carbon Black: Science and



Technology, Second Edition). Activated carbon is likewise utilized as a part of electromagnetic impedance protecting applications, for the most part as conductive fillers in composite materials, because of its electrical conductivity, synthetic resistance and small thickness. Permeation happens at a basic carbon black stacking, called permeation focus, where the initial three-dimensional ceaseless carbon black system is worked all through the polymer grid. The permeation grouping of such blends relies on upon the carbon black structure, the nature of the polymer attributes and the handling techniques and preparing environments. Overall, the conductivity is higher for polymer composites can be obtained by using dark carbon smaller size molecules (surface area is greater), the thickness of the low molecular (porosity higher molecular), higher structure (aggregation better) and volatility low (less chemisorbed oxygen groups) (Dai et al., 2007). Carbon black is generally utilized as filters in plastics, elastomers and paints to change the mechanical properties, optical materials in which it is spread and to determine their application in specific market segments.

Yttrium iron garnet,  $\text{Y}_3\text{Fe}_5\text{O}_{12}$  (YIG) is beneficial for the assembly of isolators, circulators, and magneto-optical devices for its tremendous soft magnetic properties at optical frequencies and microwave frequencies. Yttrium iron garnet is widely utilized as a part of microwave devices which is typically prepared by heating a mixture of  $\text{Y}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  above 900 °C for several hours. The technique of synthesized strongly determines the magnetic and structural properties of YIG. There are numerous technique that used to prepare the YIG nanopowders such as organic precursor technique, sol–gel, microemulsion, mechanochemical, hydrothermal and co-precipitation technique.

## **1.2 PROBLEM STATEMENT**

There are some efforts and tests have been committed to produce microwave absorber. There are several issues arise when using inorganic materials, for example, the procedure to generate literal substrate is complex because of many materials needed. It is also very expensive to run this product. Furthermore, the inorganic material is not environmental friendly and can lead to pollution and ecosystem imbalance. The idea started when we realize that natural waste, for example, paper, sawdust and coconut oil

can conquer this sort of issue. Agricultural waste materials will be materials remain in agriculture field after the yield have been harvest. Part of it was reused in agriculture as fertilizer creation, while the vast sums remained unused and in some ways represents the transfer issue. This waste polluting the river through drains and damage to nature when it produces methane and carbon dioxide. To overcome this issue, this agricultural waste such as sawdust can be set to be an important item as important as crude palm oil. Agriculture waste (sawdust) was controlled to be a carbon black by pyrolysis methods and have a good medium to be used as an electromagnetic wave absorber given the nature of the carbon material in maintaining the electromagnetic waves. In the application microwave absorber, carbon has been used as resistance elements in transforming the microwaves into heat, hence promoting the reduction or attenuation in the reflected microwave. Adding some rare earth elements and iron can increase the ability of the material to absorb electromagnetic waves. The aim of this research are to determine the efficiency of agricultural waste materials to absorb electromagnetic waves. In addition, new product known as electromagnetic shielding materials made from agricultural waste materials, rare earth and iron compounds and their composites will be designed. Finally, to investigate and assess the viability of developing radiation materials for microwave frequencies protect.

### **1.3 OBJECTIVES OF RESEARCH**

Objectives of this research are:

1. To synthesis carbon black by using pyrolysis technique.
2. To synthesis Yttrium Iron Garnet by using sol-gel technique.
3. To examine structural properties of carbon black doped Yttrium Iron Garnet by using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffractrometry (XRD) and Field Emission Scanning Electron Microscopy (FESEM).

## REFERENCE

- Adinata, D., Wan Daud, W.M.A., Aroua, M.K., 2007. Preparation and characterization of activated carbon from palm shell by chemical activation with  $K_2CO_3$ . *Bioresource Technology* 98, 145–149.
- Akhtar, M.N., Bakar Sulong, A., Khan, M.A., Ahmad, M., Murtaza, G., Raza, M.R., Raza, R., Saleem, M., Kashif, M., 2016. Structural and magnetic properties of yttrium iron garnet (YIG) and yttrium aluminum iron garnet (YAIG) nanoferrites prepared by microemulsion method. *Journal of Magnetism and Magnetic Materials* 401, 425–431.
- Dai, K., Xu, X.-B., Li, Z.-M., 2007. Electrically conductive carbon black (CB) filled in situ microfibrillar poly(ethylene terephthalate) (PET)/polyethylene (PE) composite with a selective CB distribution. *Polymer* 48, 849–859.
- Ismadji, S., Sudaryanto, Y., Hartono, S.B., Setiawan, L.E.K., Ayucitra, A., 2005. Activated carbon from char obtained from vacuum pyrolysis of teak sawdust: pore structure development and characterization. *Bioresource Technology* 96, 1364–1369.
- Lin, Y., Liu, X., Ye, T., Yang, H., Wang, F., Liu, C., 2016. Synthesis and characterization of  $CoFe_2O_4/Y_3Fe_5O_{12}$  composites based on polyaniline. *J Mater Sci: Mater Electron* 27, 4833–4838.
- Matos, J., Nahas, C., Rojas, L., Rosales, M., 2011. Synthesis and characterization of activated carbon from sawdust of Algarroba wood. 1. Physical activation and pyrolysis. *Journal of Hazardous Materials* 196, 360–369.
- Meng, H., Zhao, X., Yu, L., Jia, Y., Liu, H., Lv, X., Gong, C., Zhou, J., 2015. Island-like nickel/carbon nanocomposites as potential microwave absorbers—Synthesis via in situ solid phase route and investigation of electromagnetic properties. *Journal of Alloys and Compounds* 644, 236–241.
- Nguyet, D.T.T., Duong, N.P., Satoh, T., Anh, L.N., Hien, T.D., 2012. Temperature-dependent magnetic properties of yttrium iron garnet nanoparticles prepared by citrate sol–gel. *Journal of Alloys and Compounds* 541, 18–22.
- Ortega, P.P.S., Ramirez, M.A., Foschini, C.R., Garcia, F.G., Cilense, M., Simões, A.Z., 2014. Synthesis, structure and magnetic properties of  $Y_3Fe_5-xAl_xO_{12}$  garnets prepared by the soft chemical method. *Processing and Application of Ceramics* 8, 211–218.
- Öztürk, Y., Erol, M., Çelik, E., Mermer, Ö., Kahraman, G., Avgın, I., 2013. Structural and magnetic properties of cerium-doped yttrium-iron garnet thin films prepared on different substrates using the sol-gel process. *ResearchGate* 47, 59–63.

- Prahas, D., Kartika, Y., Indraswati, N., Ismadji, S., 2008. Activated carbon from jackfruit peel waste by H<sub>3</sub>PO<sub>4</sub> chemical activation: Pore structure and surface chemistry characterization. *Chemical Engineering Journal* 140, 32–42.
- Rehspringer, J.-L., Bursik, J., Niznansky, D., Klarikova, A., 2000. Characterisation of bismuth-doped yttrium iron garnet layers prepared by sol–gel process. *Journal of Magnetism and Magnetic Materials* 211, 291–295.
- Se, S.M., Shaaban, A., Ibrahim, I.M., 2011. Microwave absorbing material using rubber wood sawdust, in: 2011 IEEE Symposium on Wireless Technology and Applications (ISWTA). Presented at the 2011 IEEE Symposium on Wireless Technology and Applications (ISWTA), pp. 192–197.
- Shaaban, A., Se, S.-M., Ibrahim, I.M., Ahsan, Q., 2015. Preparation of rubber wood sawdust-based activated carbon and its use as a filler of polyurethane matrix composites for microwave absorption. *New Carbon Materials* 30, 167–175.
- Shaiboub, R.E., Ibrahim, N.B., 2014. Characterization of Erbium Substituted Yttrium Iron Garnet Films Prepared by Sol-Gel Method. *Journal of Nanoscience* 2014, e158946.
- Srinivasakannan, C., Zailani Abu Bakar, M., 2004. Production of activated carbon from rubber wood sawdust. *Biomass and Bioenergy* 27, 89–96.
- Vaqueiro, P., Crosnier-Lopez, M.P., López-Quintela, M.A., 1996. Synthesis and Characterization of Yttrium Iron Garnet Nanoparticles. *Journal of Solid State Chemistry* 126, 161–168.
- Wang, L., Wang, X., Zou, B., Ma, X., Qu, Y., Rong, C., Li, Y., Su, Y., Wang, Z., 2011. Preparation of carbon black from rice husk by hydrolysis, carbonization and pyrolysis. *Bioresource Technology* 102, 8220–8224.
- Yarici, I., Erol, M., Celik, E., Ozturk, Y., 2016. Effect of pH and annealing temperature on the structural and magnetic properties of cerium-substituted yttrium iron garnet powders produced by the sol-gel method. *Materials Science-Poland* 34, 362–367.