CARBON BLACK FROM PYROLISED AGRICULTURE WASTE AS ELECTROMAGNETCI SHIELDING MATERIAL

By

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DECLARATION

I hereby declare that the work in this report is my own except for quotations and summaries which have been duly acknowledged.

Signature :

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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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SYNOPSIS

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 pyrolisis 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 cm^{-1}, probably due to water absorption during test and the metal-oxygen vibrations at 577 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

SINOPSIS

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 Diffratometry* (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 Diffratometry* (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 cm^{-1}, mungkin disebabkan oleh penyerapan air semasa ujian dan getaran logam oksigen pada 577 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

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~	-	approximately
%	-	percent
λ	-	wavelength
μ	-	micron (10^{-6})
η	-	coulombic efficiency
2θ	-	Bragg angle
^{o}C	-	degree celcius
Å	-	angstrom (10^{-10})
$m^2\ g^{-1}$	-	meter per gram
$cm^3\ g^{-1}$	-	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

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, $Y_3Fe_5O_{12}$ (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 Y_2O_3 and Fe_2O_3 above 900 ºC 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 Diffractometry (XRD) and Field Emission Scanning Electron Microscopy (FESEM).

1.4 STATEMENT OF CONTRIBUTION

The scope of this study was be initiated with a sawdust sample preparation which is fabrication of carbon residue using pyrolysis techniques. The pyrolysis waste carbon residue is mixed with metals which are iron garnet and rare earth metal.

The waste residue (sawdust) is collecting at sawmill factory. Then the sawdust will be prepared by grinding and sieving with the size of 0.5 mm into fine and uniform particles. The sawdust then undergoes pyrolysis technique to get the waste carbon powder. The technique were using nitrogen gas at temperature 600 ℃ for 3 hours to produce the activated carbon. Then it will be mixed with Yttrium Iron Garnet (YIG) to enhance capability of microwave absorber.

The YIG solution was synthesized by dissolving and mixing the required amount of the iron (II,III) oxide and yttrium (III) oxide. The samples were dissolve in absolute ethanol solvent and will be stirred on the magnetic stirrer. After the sample dissolve, the sample will undergoes preheat treatment using box furnace at temperature 900 ℃ for 3 hours to remove solvent. Then, the pyrolysed waste carbon will mixed with YIG with different ratio. The absolute ethanol used as a bonding agent of the sample. After that, the sample will undergoes heat treatment at temperature 900 ℃ for 3 hours. Finally the sample will be characterization by using Field Scanning Electron Microscopy (FESEM), X-Ray Diffratometry (XRD) and Fourier Transform Infrared Spectroscopy (FTIR).

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This parts discusses how the experiment are carried out by other people. The experiments that have been reviewed discuss about the method had been run along the experiment such as the agriculture waste materials are used to produce carbon black using their method and the organic substances as a microwave absorber. This chapter also shows the result of their experiment. The review are important so that the experiment can be run by using the following method indeed.

2.2 CARBON BLACK

There are several exploration has been done in several years to produce carbon black from agricultural waste materials. The study by Matos et al., (2011) about carbon black was prepared and characterization from sawdust of Algarroba wood. The sawdust was synthesis using physical activation and pyrolysis technique under CO_2 and N_2 to obtained activated carbon (AC). The raw material (sawdust) was collecting from Algarroba (Hymenaea Courbaril) wood. To get the means size of particles 350 μm, the sawdust is grind and sieved earlier. All samples was undegoes thermally treated for 0, 1 or 2 hours at the higher temperature was 900 °C from 200 °C. Then characterization was completed by adsorption–desorption N_2 isotherms, Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectronic spectroscopy (XPS), and scanning electron microscopy (SEM). The maximum volume surface area was gained by Brunauer

Emmet–Teller (BET) at 800 °C, both under CO_2 and N_2 atmospheres to the lessening higher temperatures activation. FTIR and XPS prescribe that the essential is the functional groups on surface of carbons will change because of enactment temperatures. It can be assumed that mean pore size and functionalization on the carbon dark surface can be effectively controlled and that part permits to consider in waste biomass as a potential hotspot for the combination of carbon materials with different and potential current uses.

Another examination to produce carbon black had been appeared in the investigation by (Wang et al., 2011) study about rice husk was synthesized carbon black using pyrolysis, carbonization and hydrolysis. The rice husk was getting from a rice mill Changchun, China. Then carefully splashed the sample was with distilled water to eliminate additional soil and dust. Then the sample was dried at temperature 110 °C for 24 hours. Then the sample was undergoes pyrolysis technique were using nitrogen gas at temperatures 400 °C up to 800 °C for 1 hour. The temperature rate increment for 20 °C /min. 72 wt. % sulfuric acid at temperature 50 °C for 10 minutes for hydrolysis conditions, was accomplished 52.72 % of hydrolysis proportion. The carbon will undergoes further pyrolyzed, after carbonization of the hydrolysis solution by water bath. The carbon content will increase and surface functional groups decreased due to the temperature of pyrolysis was improved from 400 °C up to 800 °C. In view on Brunauer–Emmett–Teller (BET) results, the pore size and specific surface region of activated carbon was increased from 389 to 1034 m^2/g and 0.258 to 0.487 cm^3/g, individually. X-ray diffraction pattern (XRD) and Raman spectroscopy analyses of samples pyrolyzed at temperature 400 °C up to 800 °C demonstrated a localized graphitic structure. It can conclude that the process created in this research could likewise relevant to the synthesis of activated carbon from other types of biomass.

Activated carbon was prepared from rubber wood sawdust was exploration by (Srinivasakannan and Zailani Abu Bakar, 2004). In this research was used of two-stage activation process. Which is first stage actuation process is semi-carbonization organize up to 200 °C, then took after by an enactment arrange at a picked temperature, phosphoric acid was utilized as the initiating specialist to combination the initiated carbon from carbonaceous precursors. Examinations are led in a lab scale suppress heater under static

conditions in a self-created environment covering process parameters, for instance, impregnation extent, temperature and carbonization time. In this study the antecedent material with impregnation specialist is shown straightaway to semi-carbonization and temperature of enactment not in the least after the specific temperature development. In this study the procedure parameters are enhanced by iodine number and product yield. In view on a BET, that at 200 °C for 15 minutes of semi-carbonization will produce of 1496 m^2/g surface region with 35 % of product yield and activation agent recovery of 90 % of activation agent recovery. Then followed by temperature of activation at 500 °C for 45 minutes with impregnation proportion a product of 1.5 yielded with iodine number 1096.

Carbon black produced from jackfruit peel waste by using phosphoric acid (H_3PO_4) as solution activation agent had been invested by (Prahas et al., 2008). Their studies demonstrate that the activated carbon of jackfruit peel was developed by collecting jackfruit waste variety *nangka kunir* from a local fruit store at Malang, East Java. The sample was washed and cleaned for few times with distilled water to removeall the impurities by removing the carpel fibers. Then, the sample was grinded by using micro hammer mill. Chemical activation method using phosphoric acid was used to activate the raw material. This research studies about the impact of impregnation extent and temperature of enactment on pore structure and the subsequent carbon by its surface chemistry. The pore structure of the carbons was determined BET, XRD and SEM, and its surface chemistry was investigated by FTIR and Boehm titration method. The result shows that the activation temperature at 350 °C was produced non-porous carbons, while at activation temperature of 450 °C and 550 °C was produced the carbons with well-developed pores. It can be concluded that the increasing of activation temperature due to decreases of the amount of acidic functional groups, however the basic surface groups of the carbon will increase.

Carbon dark is absolutely comprehended as permeable material, with an expansive particular surface area, which helps in the adsorption of both gasses and solutes from fluid solution. One more examination to produce carbon black had been appeared in the research by (Adinata et al., 2007). Their study is about the activated carbon was produced from palm shell by using potassium carbonate K_2CO_3 as activating

agent. First, palm shell was collected from Malaysia oil palm shell (MOPS). Then the sample was grinded to obtain a particle size portion of 1-2 mm. Then, the sample was dried and sieved. Subsequently, a saturated solution of potassium carbonate was prepared by dissolved in distilled water. The effect of carbonization temperatures of 600 °C until 1000 °C and impregnation proportion of the synthesized carbon black on the pore change and yield was determined. In this study can be conclude that in all cases, the yield decreased due to increasing the carbonization temperature and impregnation proportion, however the adsorption of CO2 was increased, dynamically. At temperature 800 °C and 1170 m^2/g with activation duration for two hour and impregnation proportion of 1.0 is the maximum value for specific surface region of activated carbon. The summary of carbon production was tabulated in Table 2.1.

Table 2.1
Summary of Carbon Black Production

SUMMARY	REMARK	REF
Sawdust from wood Algarroba was synthesis to become activated carbon as a function of temperature under CO_2 and N_2 flow. It prescribe that waste biomass is a potential hotspot for the preparation of carbon materials with potential uses.	-Raw material is sawdust -Thermally pickled by 0, 1 or 2 hours at the final temperature 200 °C until 900 °C -use FTIR and XPS for examine the carbon black	(Matos et al., 2011)
The rice husk was developed using pyrolysis technique, carbonization and hydrolysis. As the temperature increase from 400°C – 800°C, the content of carbon also increase. It is shows that this process could likewise use to prepare activated carbon from other types of raw material.	-use XRD and Raman spectroscopyto examine carbon black -temperature at 400 °C to 800 °C -raw material use is rice husk	(Wang et al., 2011)

Activated carbon was prepared from elastic wood sawdust utilizing a two-organize actuation prepare with utilized phosphoric acid as the initiating agent. Examinations are led in a lab scale muffle furnace under static conditions in a self-created air covering process parameters.	-Raw material use is rubber wood sawdust -Prepare carbon black by using phosphoric acid	(Srinivasakannan and Zailani Abu Bakar, 2004)
Carbon black produced from jackfruit peel waste using phosphoric acid (H_3PO_4) as solutian activation agent. It can be concluded that the increasing of activation temperature due to decreases of the quantity of acidic functional groups, however the basic surface groups of the carbon will change.	-Raw material use is jackfruit variety *nangka kunir*. -chemical activating agent is phosphoric acid.	(Prahas et al., 2008)
Carbon black from palm shell was prepared using potassium carbonate (K_2CO_3) as activating agent. This experiment was study on development and yield of pore. It shows that the mesopore volume will increase due	-raw material is palm shell -using potassium carbonate (K_2CO_3) as activating agent.	(Adinata et al., 2007)

2.3 MICROWAVE ABSORBER

Nanocomposites have been used widely in producing of microwave absorber. In the investigation of Island-like nickel/carbon nanocomposites as conceivable microwave absorbers, (Meng et al., 2015) has studied about the nanocomposite was possibly as a light microwave absorber. In this study, nickel-carbon (Ni/C) nanocomposite was arranged just as light weight microwave absorber potential by calcination of nickel nitrate-polyacrylamide combination of the stream of ammonia. Electromagnetic properties of as-prepared Ni/C nanocomposite were explored regarding their arrangement and microstructure. It demonstrate that the as-synthesized nanocomposites Ni/C is the morphology of the island-like and consist of scattered consistently Ni nanoparticles and carbon permeable medium. Electromagnetic properties of nanocomposites Ni/C is highly dependent on the temperature of calcination and Ni/C nanocomposite shows the electromagnetic properties are great obtained at 600 °C. This is credited to the porous structure of extraordinary and craved electromagnetic impedance coordinating of the carbon medium and in addition the uniform scattering of Ni nanoparticles in the carbon medium.

Other study of light weight microwave absorber have been conducted by (Liu et al., 2015) where reported that the center/shell-organized Mg/C nanocapsules with Mg nanoparticles as the center and anion-like carbon as the shell was prepared using the arc-discharge technique. The ideal return loss estimation of 17.02 dB was obtained at 11.2 GHz with coordinating thickness of 3.0 mm. Hypothetical recreation for the absorption of microwave transmission line using a hypothetical prudent agree well with the test results. The transmission-line hypothesis likewise can be utilized to compute the return loss of non-magnetic nanocapsules.

2.4 MICROWAVE ABSORBER MADE OF SAWDUST

Sawdust is one of the agriculture waste that have great physical and structural properties for led the microwave absorber. The investigation by (Shaaban et al., 2015) about synthesized carbon black from rubber wood and its function as a filler for

polyurethane matrix composite. Activated carbon has been prepared from wood sawdust of elastic with using $ZnCl_2$ as agents of activation at 500 °C for one hour with $ZnCl_2$ and ratio of the mass of wood sawdust of elastic is 1.0 - 2.0. In determining their transparency and complex properties of microwave absorption that used in electromagnetic interference (EMI) shielding, loaded activated carbon has been provided with a polyurethane (PU) composite by chemical foaming techniques used at different amount of the activated carbon. EMI shielding is within the scope of valuable productivity of about 3 dB in a wide frequency range 1 to 2.5 GHz. This composite is more appropriate for microwave absorption and is a potential possibility for EMI shielding uses, as compared with other conventional materials for example polyethylene and polyester which loaded with metal additives.

Another examination utilizing sawdust of fiber wood had been appeared in the research by (Se et al., 2011). Their study is about microwave absorbing material from rubber wood sawdust. This study determine the impregnation of activated carbon and polyurethane that will produce microwave absorber at 1.8 GHz. Through a chemical activation process using $ZnCl_2$ solution as precursor agents the carbon black was prepared from wood sawdust. The sample was heated at temperature of 500 °C for an hour. It was reported that the activation of the chemical at proportion 2 : 1 create a high-purity activated carbon with a carbon content of 79 percent. The BET analysis, for impregnation ratio 1.5 : 1 reported that the elevated estimation of 1301 m^2/g, have been used to preparing microwave absorber which have return loss of 10 dB at 1.8 GHz.

2.5 RARE EARTH METAL AS MICROWAVE ABSORBERS

In a most recent couple of decades, there is some learn about the rare earth metal, for example, lanthanum and yttrium were utilized as microwave absorber. The study in electromagnetic characteristics and absorbing properties of multi-walled carbon nanotubes filled with Er_2O_3 nanoparticles as microwave absorbers by Lan Zhang et al. (2008) display the Multi-walled carbon nanotubes (MWCNTs) loaded with Er_2O_3 were integrated by wet chemical method. Their electromagnetic traits and microwave absorbing properties were investigated in the frequency scope of 2–18

GHz. The unpredictable permittivity and electric misfortune digression of Er_2O_3 -filled MWCNTs show a decreasing example while the perplexing porousness and attractive misfortune digression are greater than those of the unfilled MWCNTs. The Er_2O_3 nanoparticles encapsulated in the cavities direct the electromagnetic parameters of MWCNTs, and as needs be impact the microwave engrossing properties. The changed MWCNTs have broader absorbing bandwidth and larger reflectivity than those of unfilled MWCNTs. With the increment of thickness, the peak value of reflectivity movements to lower frequencies and numerous retaining tops show up. The after effect of estimation shows that Er_2O_3-filled MWCNTs have potential applications in slender thickness and light-weight microwave absorbers.

CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

This chapter started with sample preparation of waste material (sawdust). The waste residue (sawdust) is collecting at sawmill factory. Then the sawdust will be prepared by grinding and sieving with the size of 0.5 mm into fine and uniform particles. The sawdust then undergoes pyrolysis technique to get the waste carbon powder. The technique were using nitrogen gas at temperature 600 ℃ for 3 hours to produce the activated carbon. Then it will be mixed with Yttrium Iron Garnet (YIG) to enhance the capability of microwave absorber.

The YIG solution was synthesized by dissolving and mixing the required amount of the iron (II,III) oxide and yttrium (III) oxide. The samples were dissolve in absolute ethanol solvent and will be stirred on the magnetic stirrer. After the sample dissolve, the sample will undergoes preheat treatment using box furnace at temperature 900 ℃ for 3 hours to remove solvent. Then, the pyrolysed waste carbon will mixed with YIG with different ratio. The absolute ethanol used as a bonding agent of the sample. After that, the sample will undergoes heat treatment at temperature 900 ℃ for 3 hours. Finally the sample will be characterization by using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffratometry (XRD) and Field Scanning Electron Microscopy (FESEM). The methodology of this study can be summarized as shown in Figure 3.1.

3.2 RESEARCH METHODOLOGY

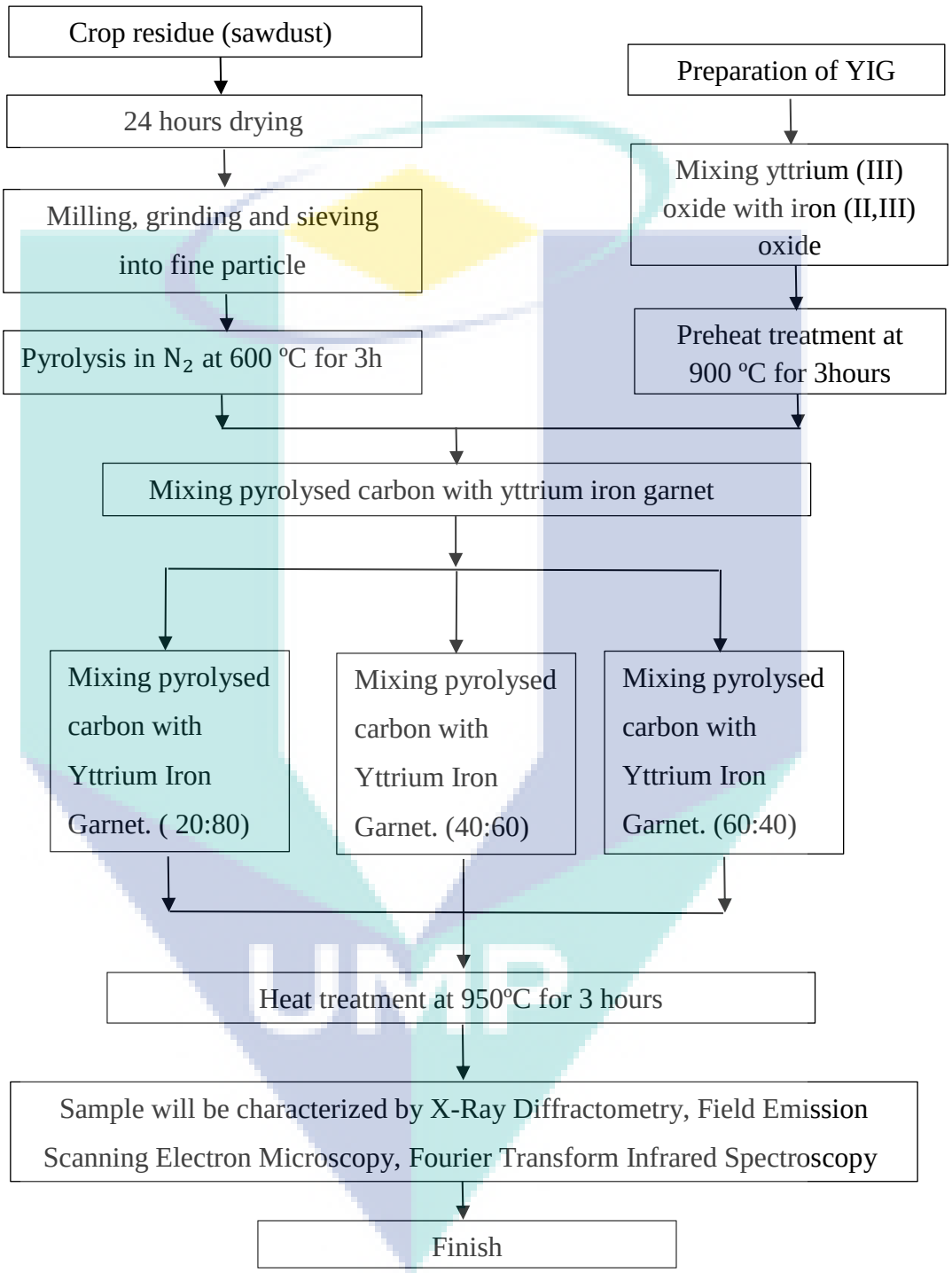

Figure 3.1. Procedure of synthesized the Ytrrium & Iron oxide doped carbon black

3.3 CHEMICAL AND SOLVENT

The chemical and solvent used in this research are a listed below:

1. Iron (II,III) Oxide Fe_3O_4 from Bendosen Laboratory Chemical with Mw=231.53 g/mol, 99.99%
2. Ytrrium (III) Oxide Y_2O_3 from Aldrich Chemical with Mw=225.81 g/mol, 99.99%
3. Ethanol absolute (liquid) from HmbG Chemical with Mw=46.07 g/mol, 99.7%

3.4 EQUIPMENT

All the eqiupment utilized as a part of this research was standard laboratory and are listed below:

1. Laboratory magnetic stirrer for mixing the carbon, Iron (II,III) Oxide, Yttrium (III) Oxide by ethanol absolute.
2. Ball milling from Retsch brand with model PM 100 for milling the powder with Zirconia ball.
3. Fourier Transform Infrared Spectroscopy with model Perkin Elmer Spectrum 100 for approve the execution particulars of these materials to block or transmit IR radiation.
4. X-Ray Diffraction from Ragaku brand with model MiniFlex II for synthesizing pure yttrium iron garnet and mixing pyrolysed carbon.
5. Field Emission Scanning Electron Microscope from JEOL JAPAN brands with model of EQPCL 170 JSM-7800F to analyzed surface morphology and diameter of particle. (10x to 300,000x of magnifications)
6. Tube Furnace from Germany Nabertherm brand model of B 180. (Temperature maximum 1200 ºC)

3.5 SAMPLE PREPARATION

The preparation of sample started with the collection of sawdust sample from saw mill factory. The sample will be dried in oven for 24 hours. This is to ensure the sample is fully dried and completely removed the solvent from sawdust. In order to remove foreign substances and to get the uniform size, sawdust then will be sieved to get the size of 0.5mm.

3.5.1 Ball Milling Technique

In order to get the nanosize particles, sawdust sample will undergoes milling process. 16 g of sample were placed in the ball mill with 3.24 kg in full weight and undergoes ball milling technique within 1 hour with the constant speed of 100 rpm. After the milling process done, the samples were dried within 24 hour to remove the solvent from sample. RETSCH brands Planetary Ball Mills as shown in Figure 3.2 are utilized wherever the most elevated degree of fineness is required. Aside from the established mixing and size lessening forms, the mills likewise meet all the specialized reqiurements for colloidal crushing and have the vitality input fundamental for mechanical alloying forms. The greatly high diffusive strengths of the Planetary Ball Mills result in high pulverization energy and therefore short crushing times.

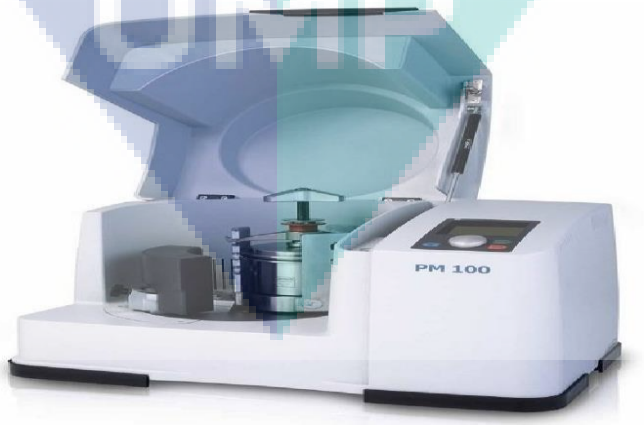

Figure 3.2. RETSCH Brand Ball Milling Machine Model PM100

3.5.2 Fabrication of waste carbon

The pyrolysis technique has been used in fabrication of the waste carbon. Pyrolysis is the procedure where the carboneous source materials will be burnt, decomposed and changed over to carbonized material in the absent of air . At that point, the procedure was proceeded by initiation step which will change the surface area of the carbonized material. In this work, nitrogen gas has been used into the tube furnace as show in Figure 3.3. The sample were placed in tube furnace with nitrogen gas flow for 3 hours with constant temperature of 600 °C.

Figure 3.3. Nabertherm Brands Tube Furnace Model B 180

3.5.3 Fabrication of Yttrium Iron Garnet

Yttrium (III) oxide, Iron (II,III) oxide and ethanol were used for the synthesis of Yttrium Iron Garnet (YIG). YIG was synthesized by using sol-gel technique. The YIG precursor solution was synthesized by dissolving and mixing the necessary amount of the metal oxide in stoichiometric ratio of Y:Fe = 3:5 in ethanol solution. The sample were dissolved by the absolute ethanol solvent in the beaker and placed on the magnetic stirrer. The solutions were stir at 100 °C for 6 hours until it completely dissolve and dried. Then the dried samples were undergoes preheat treatment using the box furnace at 900 °C for

3 hours to remove the solvent completely. After the solvent completely removed, the samples were placed into the pestle and mortar and crushed it into the nanosized.

3.5.4 Fabrication of waste carbon doped Yttrium Iron Garnet

Three sample of pyrolysed crop residue waste carbon were prepared with different ratio of YIG. Absolute ethanol utilized successfully to dissolve metallic powder. The three type of sample have been summarized as shown in Table 3.1.

Table 3.1
Weight ratio of the samples

Sample	Compound	Ratio
1	Pure YIG	
2	YIG + Carbon black	80:20
3	YIG + Carbon black	60:40
4	YIG + Carbon black	40:60

The following sample then were dissolved by absolute ethanol solvent in the beaker and placed on the magnetic stirrer as shown in Figure 3.4. The solutions were stir at 100 °C for 5 hours until it completely dissolve and dried. The dried samples then were placed inside the furnace for heat treatment at 950 °C for 3 hours to remove the solvent completely. After the solvent completely removed, the samples were placed into the pestle and mortar and crushed it into the nanosize particle for the final step characterization of sample.

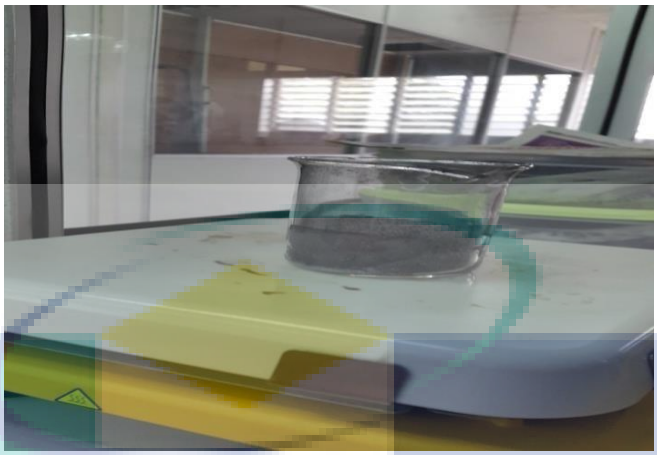

Figure 3.4. Sample dissolved on the magnetic stirrer

3.6 MATERIAL CHARACTERIZATIONS

The characterization of sample has been carried out to study the structural properties of the samples. The entire samples were characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffactometry (XRD) and Field Emission Scanning Electron Microscopy (FESEM). The infrared spectrum of emission for the chemical content can be investigated by Fourier Transform Infrared Spectroscopy (FTIR). The FTIR instrument as illustrate in Figure 3.5. The composition for the chemical content can be investigated from X-ray diffraction (XRD) experiment's result. The XRD instrument as illustrate in Figure 3.6 usually detects the presence of defect within a crystal. This uniformity can be investigated by Field Emission Scanning Electron Microscope (FESEM) as illustrate in Figure 3.7 since it will able to view the composite micro structure. FESEM is a tool to observe invisible worlds of microspace and nanospace and reveals level of detail and complexity inaccessible by light microscope.

3.6.1 Structural Characterization

In this section, the structural properties of sample will be characterizaed by using Fourier Transform Infrared Spectroscopy, X-ray Diffactometry and Field Emission Scanning Electron Microscopy.

3.6.1.1 Fourier Transform Infrared Spectroscopy (FTIR)

The Spectrum 100 Fourier Transform Infrared Spectroscopy (FTIR) gives enhanced ordinate precision to the estimation of optical filters and high-refractive-index materials. Manufacturers of optical filters and strength coatings can utilize the Spectrum 100 to approve the performance of these materials to block or transmit IR radiation.

Figure 3.5. Perkin Elmer Spectrum 100 Fourier Transform Infrared Spectroscopy Machine

3.6.1.2 X-Ray Diffractometer (XRD)

The Rigaku Miniflex X-Ray Diffractometer is a Desktop Powder Diffractometer fit for measuring powder diffraction plots from 3 to 80 degrees in two-theta filtering range. It can be utilized for phase identification, qualitative and quantitative analysis and quality control of crude materials and products. The sorts of tests can be multi-stage microcrystals of powdered materials, metal and ceramic plates. It is furnished with a 6-test holder for most outrageous computerization of test estimations. The X-ray of the Cu Kα radiation separated by a Ni filter has a wavelength of 1.54 Å. The instrument is furnished with the latest form of PDXL, Rigaku's full-work powder diffraction investigation bundle. The latest variant of PDXL offers basic new usefulness; including a key parameter technique (FP) for more precise peak calculation and phase identification utilizing the Crystallography Open Database (COD).

Figure 3.6. RIGAKU Brand X-Ray Diffractometer Machine Model Miniflex II

3.6.1.3 Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy (FESEM) gives geographical and natural information at magnifications of 10x to 300,000x, with virtually boundless depth of field. Differentiated to tradition filtering electron microscopy (SEM), field emmision SEM (FESEM) produces clearer, less electrostatically mutilated pictures with spatial resolution down to 1/2 nanometers – 3 to 6 times better.

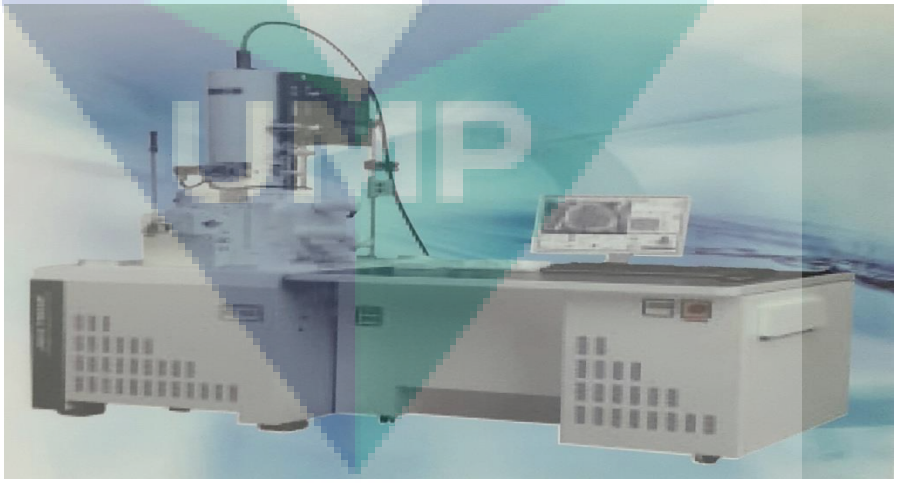

Figure 3.7. JEOL JAPAN brands with model of EQPCL 170 JSM-7800F Field Emission Scanning Electron Microscopy Machine

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CHAPTER 4

RESULT AND DISCUSSION

4.1 STRUCTURAL CHARACTERIZATION ANALYSIS

In this section, the structural properties of sample which is FTIR, XRD and FESEM have been anylyzed.

4.1.1 Fourier Transform Infrared Spectroscopy Analysis

The typical wastes carbon were used with the different ratio of YIG as shown in Figure 4.1

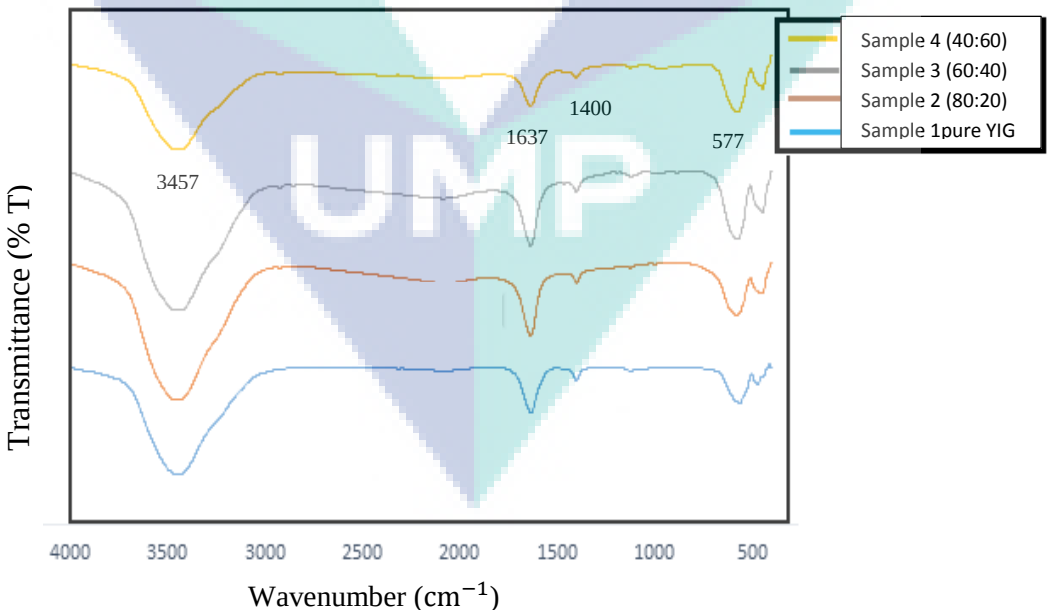

Figure 4.1. Infrared spectroscopy of pure YIG and YIG doped carbon black powders synthesized at 900 °C for 3 hours

FTIR spectrum for the powders are shown in Figure 4.1. The presence of different bonds in the all specimen powders can be taken note. All powders introduced O–H bond extending from 3100 to 3600 cm^{-1}, most likely because of water absorption during test. Wavenumbers extending from 825 to 930 cm^{-1} represent axial strain of C–O bonds in carbonates and carboxylates, respectively, demonstrating the presence of these compound in the samples. Wavenumbers extending from 577 cm^{-1} are assigned to extending mode of YIG tetrahedra, normal for metal-oxygen bonds in ceramic. There was, a vibration band noticed that is related to the distortion of O–H bonds close to 1000 and 1222 cm^{-1}. This is attributed to water adsorbed at the powder surface when the sample was in contact with atmosphere. The spectrum displays two bands at 575 and 445 cm^{-1} alloted to the extending method of YIG tetrahedral. The increasing carbon black, these bands are slightly broadened and shifted towards higher wavenumbers. Figure 4.1 demonstrates all peaks at 3457, 1637, and 577 cm^{-1}, relating to the extending and bending vibrations of O–H, C=O and metal-oxygen vibrations, respectively. The FTIR spectrum indicates very weak carbonate vibrations. The metal-oxygen vibrations at 577 cm^{-1} which are because of the lattice vibrational modes of the YIG unit cell.

4.1.2 X-Ray Diffraction Analysis

The XRD spectra of the typical waste carbon with the different ratio of YIG samples synthesized by sol-gel technique and heated at same temperature of 900 ˚C for 3 hours are shown in Figure 4.2

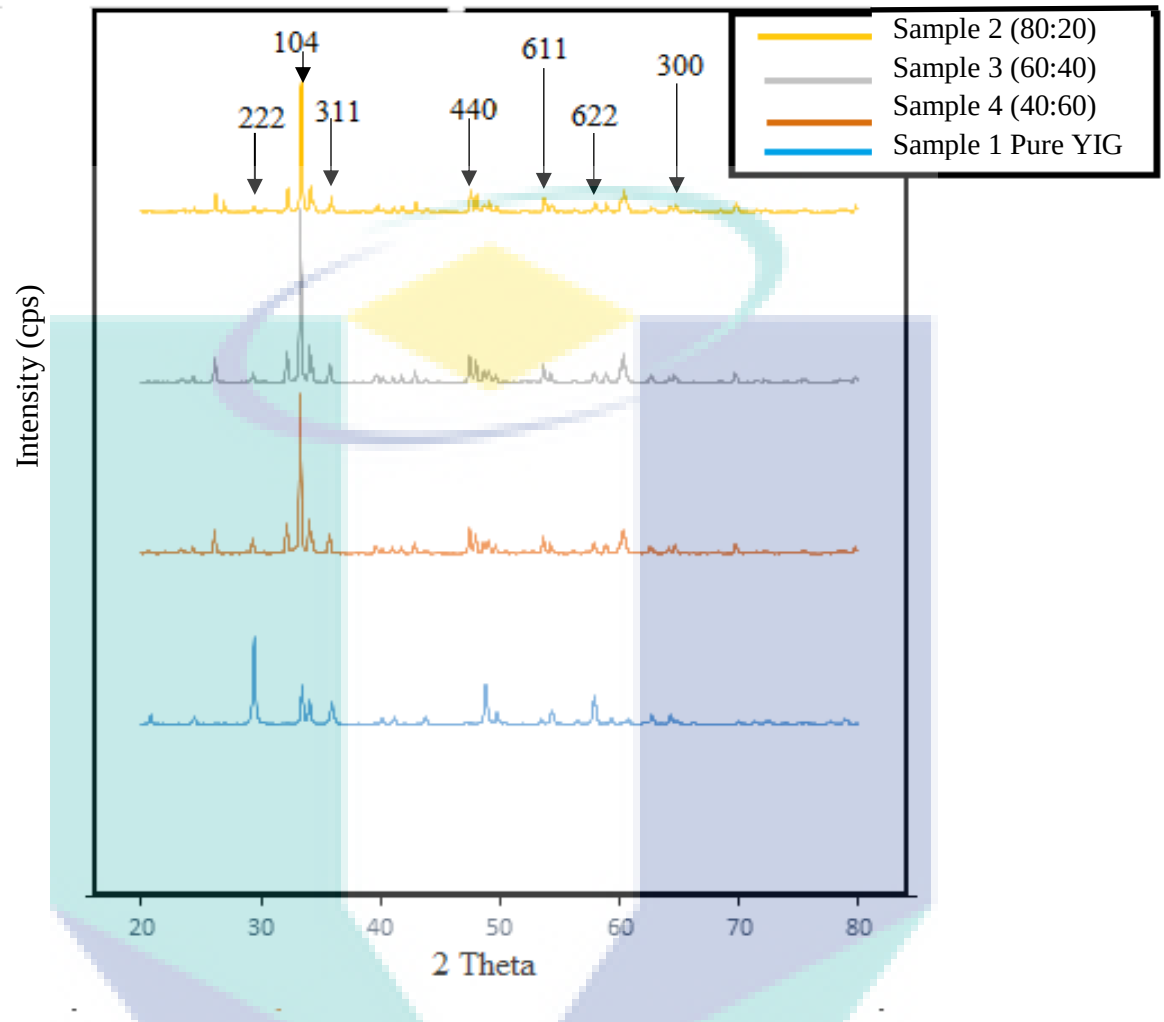

Figure 4.2. XRD spectra of powder obtained from the pure Yttrium Iron Garnet and Yttrium Iron Garnet doped carbon black with different ratio

The comparative study of the XRD profiles of all the four samples along that of pure yttrium iron garnet is shown in Figure 4.2. It can be clearly seen that all the samples shows two distinct and similar strong π band lied on prominent (311), (440) and (611). Each prominent contain graphitized for each sample. The presence of broadened peak in all samples suggests that the sample formed compose garnet crystal structure.

Small shift of diffraction lines of bragg angles with increasing the ratio of carbon black were observed. This is due to that the decreasing doping of carbon black on YIG, and by the different amount of carbon black the diffraction lines of bragg angles will small shift would be expected. Obviously that the 2θ position of each crystal plane

showed almost no differences with respect to the different amount of carbon black, suggesting that the crystal planes were not damaged by doping. A comparatively low intensity XRD profile of the samples indicates that all the nanomaterial including the ferrite and rare earth metal that has been prepared have very low fraction of disordered amorphous carbon while the presence of the high intensity π band in all of them shows that the samples have a high degree of graphitization or crystallinity.

The prominent of (104) shows very strong peak from sample 2 that obviously prove the presents of waste carbon in the Yttrium Iron Garnet and the π band lied on ~46 respectively and this generally occurs in graphitic carbon with microcrystalline structure. The distinctly peak from the pure YIG lie on ~36 and ~26 from the prominent (222) and (440). The broadening of the peak is an indication of delocalization of electronic states in an ensemble of close-packed nanocrystals. Broadening can also occur due to the strain in the crystal.

4.1.3 Field Emission Scanning Electron Microscopy Analysis

Surface morphology and the diameter of particle of the samples have studiedby FESEM characterization. The FESEM image showing the morphology of yttrium iron garnet mixed with the pyrolysed of carbon with different ratio as shown in Figure 4.3, 4.4 and Figure 4.5. The diameter of particle of each sample has been analyzed at x10000 magnification.

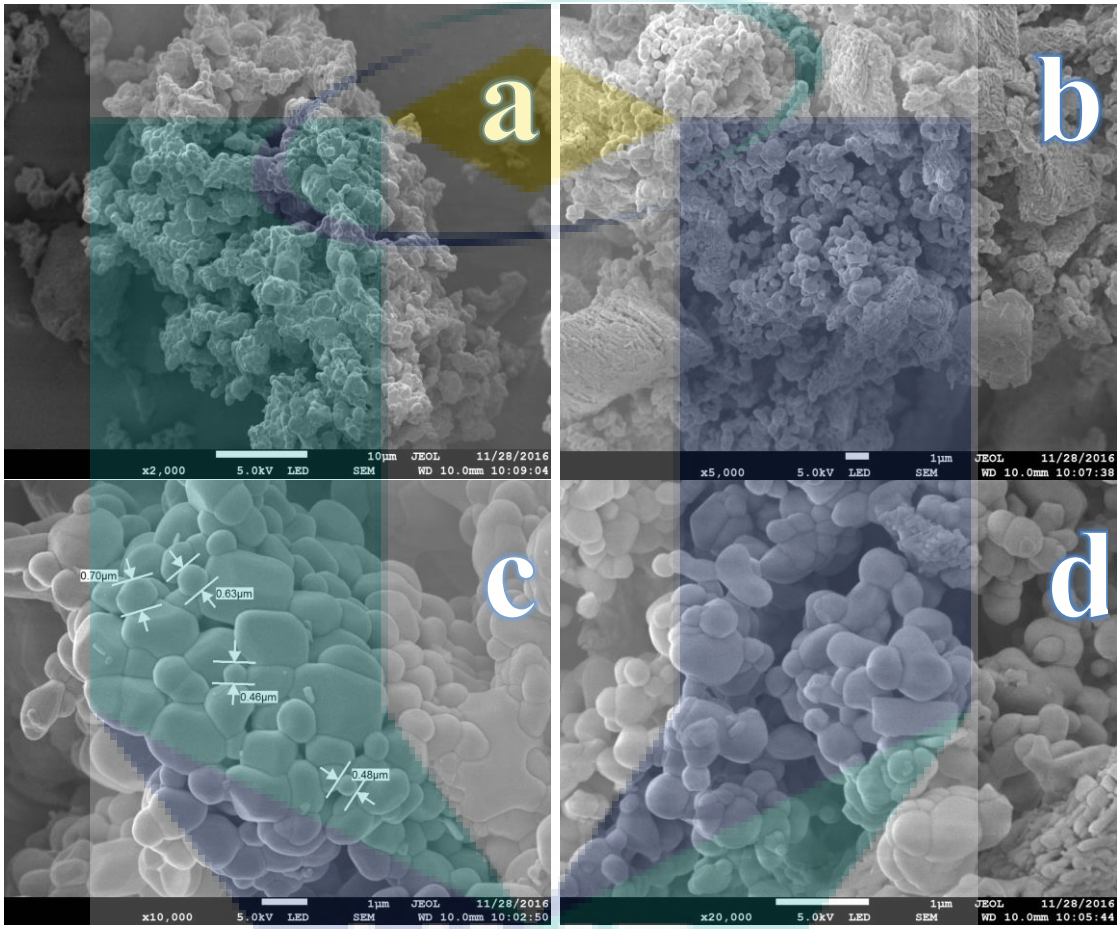

Figure 4.3. FESEM image of Yttrium Iron Garnet doped Carbon black with ratio (80:20) (a) x2000 magnification (b) x5000 magnification (c) x10000 magnification (d) x20000 magnification

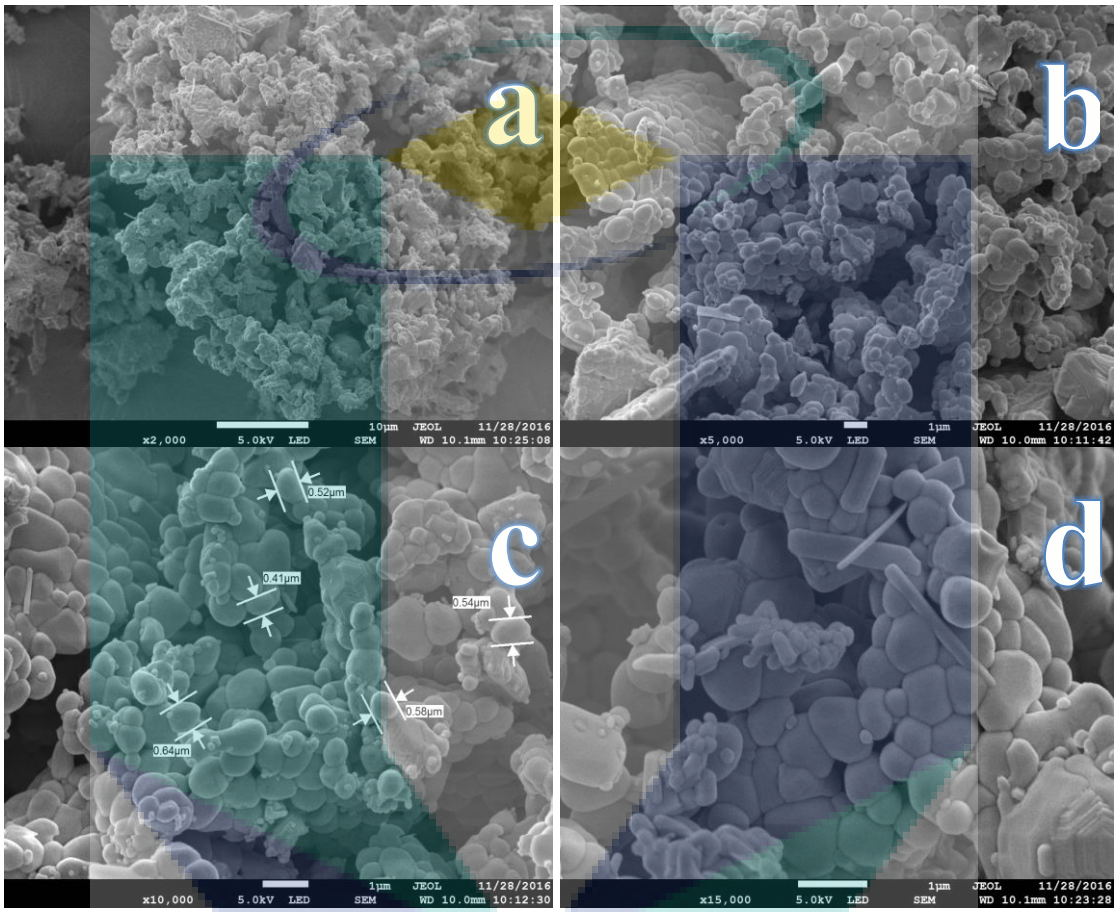

Figure 4.4. FESEM image of Yttrium Iron Garnet doped Carbon black with ratio (60:40) (a) x2000 magnification (b) x5000 magnification (c) x10000 magnification (d) x15000 magnification

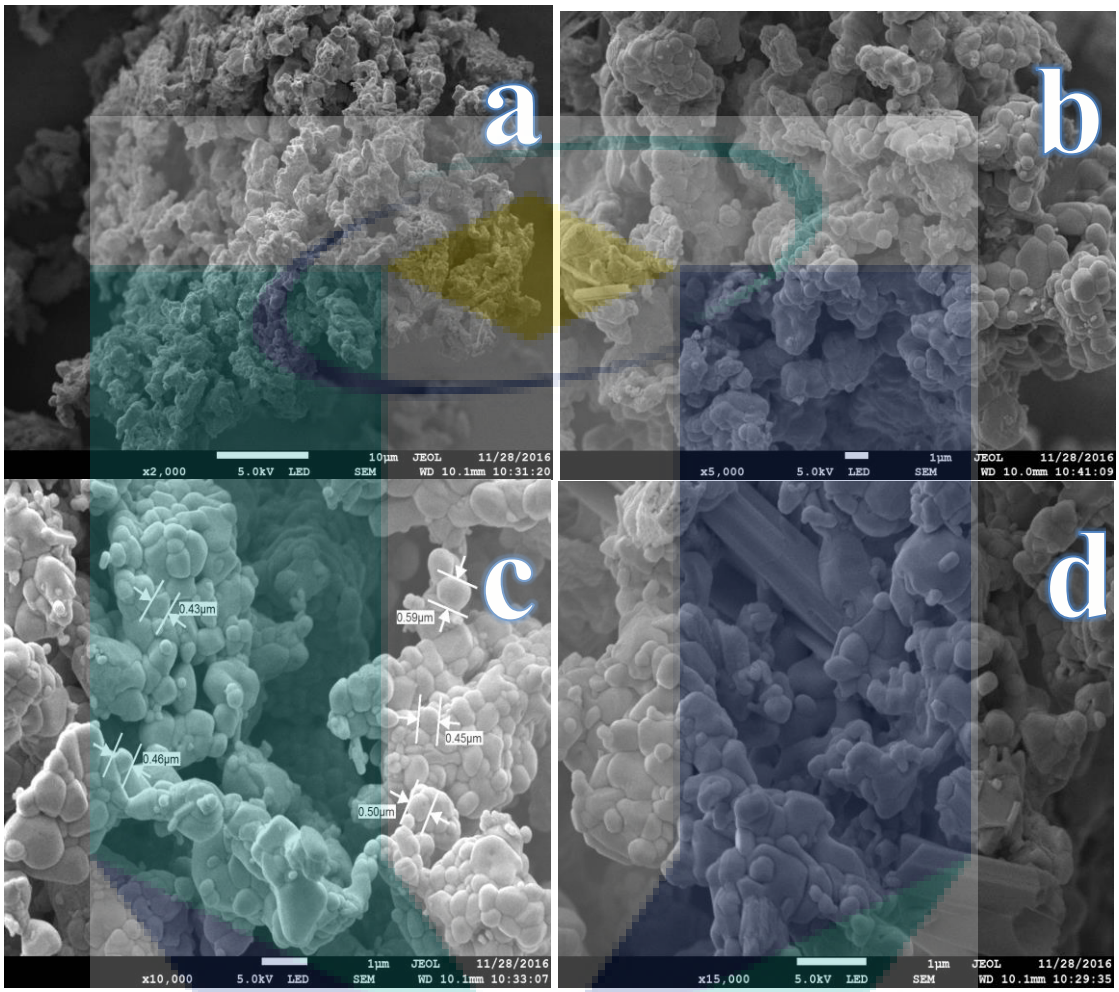

Figure 4.5. FESEM image of Yttrium Iron Garnet doped Carbon black with ratio (40:60) (a) x2000 magnification (b) x5000 magnification (c) x10000 magnification (d) x15000 magnification

Figure 4.3 are referring to the image of FESEM of YIG doped carbon black with ratio 80:20 that having range of diameter at x10000 magnification within ~0.46 µm – 0.70 µm. The image of FESEM of YIG doped carbon black with ratio 60:40 that having range of diameter of particle at x10000 magnification within ~0.41 µm – 0.64 µm as shown in Figure 4.4. While Figure 4.5 show the image of FESEM of YIG doped carbon black with ratio 40:60 that having range of diameter of particle at x10000 magnification within ~0.43 µm – 0.59 µm.

Table 4.1 are referring to the summary of the diameter of particl range and average diameter of particle of every sample. The FESEM results show that the average of the diameter of particle were decreasing with the increasing doping of carbon black. The increasing doping of carbon black, the molecular weight of sample will decreasing. The largest average diameter of particle are from sample 1 which are 0.56 µm with the molecular weight of Yttrium Iron Garnet ($Y_3Fe_5O_{12}$) are 590.35 g/mol. While in the sample 2, the average diameter of the particle was 0.54 µm with molevular weight of YIG are 442.76 g/mol. The average diameter of the particle from sample 3 was the smallest which is 0.47 µm with the higher amount doping of carbon black. Therefore, it can be concluced that the diameter of the particle strongly depend on the molecular weight of the compound.

Table 4.1
The summary of the diameter of the particle range and average diameter of every sample

Sample	Diameter range	Average diameter
Sample 1 : YIG + Carbon black (80:20)	~0.46 µm – 0.70 µm	0.56 µm
Sample 2 : YIG + Carbon black (60:40)	~0.41 µm – 0.64 µm	0.54 µm
Sample 3 : YIG + Carbon black (40:60)	~0.43 µm – 0.59 µm	0.47 µm

From table 4.1, clearly can seen that sample 1 which Yttrium Iron Garnet ($Y_3Fe_5O_{12}$) doped carbon black with ratio 80:20 was the largest diameter of the particle among others sample. This is due to the higher amount of YIG in the sample that increase the molecular weight then increase the diameter of the particle. The present of YIG also further enhance the capabilities of carbon compound.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUCION

The carbon black were successfully prepared by suing pyrolysis technique. $Y_3Fe_5O_{12}$ (YIG) was successfully obtain via sol-gel technique with the mixed amount of Yttrium (III) Oxide and Iron (II,III) Oxide. Both rare earth metal and ferrite enhance the capability of the compound. By using Fourier Transform Infrared Spectroscopy, showed all powders analysed presented O–H bond extending from 3100 to 3600 cm^{-1}, probably due to water absorption during test and the metal-oxygen vibrations at 577 cm^{-1} which are due to the lattice vibrational modes of the YIG unit cell. Carbon and graphite have been analysed in the characterization of X-ray Diffratometry analysis and the diameter of the particle have been analyse from Field Emission Electron Microscopy. The diameter of particle strongly depend on the molecular weight of particle. Sample 1 is the best samples among three sample for this analysis.

5.2 RECOMMENDATIONS

There are some recommendation to enhance the current finding in this study. Firstly, the solution on the magnetic stirrer should be dissolved in the longer period to make sure that yttrium and iron completely dissolve inside ethanol solution. Then, the YIG sample must be put inside the desiccator cabinet to make sure that YIG will not contaminated. Other than that, the sampl YIG should undergoes different of preheat treatment to study the effect of temperature.

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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 K2CO3. 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 CoFe2O4/Y3Fe5O12 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 Y3Fe5-xAlxO12 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., Avgýn, 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 H3PO4 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.