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DEVELOPMENT OF EPOXY-OPEFB COMPOSITES FOR SHIELDING APPLICATIONS

AHMAD KHAMIS

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DEVELOPMENT OF EPOXY-OPEFB COMPOSITES FOR SHIELDING APPLICATIONS

By

AHMAD KHAMIS

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Faculty : Science

Microwave absorbers are widely used in applications to eliminate unwanted or stray radiated electro-magnetic signals which might interfere with a system's operation. These absorbers are basically made up of a polymer matrix reinforced with a filler material which might be one element or more. Ferrite composites are widely used in the development of microwave absorbers. However, ferrites are expensive, corrosive, heavy, and non-biodegradable.

This research focuses on using oil palm empty fruit bunch (OPEFB) fibres as a filler for microwave absorber with epoxy resin as the host matrix. OPEFB fibres have several advantages including biodegradability, low density, low cost, and better thermal properties. Also epoxy resin has various advantages such as good dimensional stability, high mechanical properties, ease of processing and curing, and moisture resistance. Epoxy-OPEFB composites were fabricated by varying the OPEFB percentages (0%, 5%, 10%, 15%, 20%, 25%, 30%, and 40%). OPEFB fillers of 300 µm size, epoxy resin and hardener were mixed and stirred to produce Epoxy-OPEFB composites using mini vortex mixer at 3000 rpm for 30 minutes at room temperature (25 °C). The total mass of each mixture was 12 g which then poured into two different flanges in order to use two different techniques of complex permittivity characterizations. The dielectric constant and loss factor are main parameters related to dielectric characteristics of a particular material, and directly associated to the absorbing characteristics. The complex permittivity, transmission coefficient |S21|, reflection coefficient |S11|, power loss, reflection loss, and total shielding effectiveness (SE) were studied in the frequency range (8-12) GHz. Dielectric constant (ε'), loss factor (ε''), reflection and transmission coefficients of the composites were measured using rectangular waveguide (RWG) connected to vector network analyser (VNA) in the frequency range (8-12) GHz. Also an open ended coaxial probe (OECP)

connected to a vector network analyser was utilized to measure the dielectric constant and loss factor of all composites at room temperature (25 °C). X-Ray diffraction was utilized to analyse the microstructure of the composites. The results show that the dielectric properties increased but $|S_{11}|$ and $|S_{21}|$ decreased by increasing OPEFB percentage in the composites. The complex permittivity was dependable on the OPEFB percentage of the composites, increasing OPEFB content in the composites caused an increment in the complex permittivity of Epoxy-OPEFB composites. Epoxy-OPEFB composites were found to have a very close dielectric constant and loss factor to ferrite-polymer composites. At 10 GHz, the dielectric constant and loss factor of the Epoxy-OPEFB composites were be between 2.8 to 3.35 and 0.14 to 0.29, respectively.

In addition, results of $|S_{11}|$ and $|S_{21}|$ were used to determine the power loss, reflection loss and total shielding effectiveness of Epoxy-OPEFB composites. OPEFB had a strong influence on the shielding effectiveness, power loss and reflection loss which increased by increasing OPEFB percentage in the composites. The total shielding effectiveness values were found to be between 15.4 dB and 18.9 dB at 10 GHz. The calculations of the S-Parameters coefficients of the samples were carried out using Finite Element Method (FEM) technique by COMSOL software. The measured and calculated results of $|S_{11}|$ and $|S_{21}|$ were also investigated.

The simulated and measured results of $|S_{11}|$ and $|S_{21}|$were in a good agreement. Finally, the electric field distribution was visualized using Comsol software, it was found that the amplitude of electric field was strongly reduced after passing through Epoxy-OPEFB composites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PEMBANGUNAN KOMPOSIT EPOXY-OPEFB UNTUK APLIKASI PERISAI ELEKTROMAGNETIK

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Penyerap gelombang mikro banyak digunakan dalam aplikasi untuk menghilangkan isyarat elektromagnet yang tidak diperlukan atau penyerakan pancaran yang berkemungkinan menganggu sistem operasi. Pada asasnya, penyerap ini diperbuat menggunakan polimer matriks yang dikuatkan dengan pengisi bahan yang terdiri daripada pelbagai elemen. Komposit besi oksida digunakan secara meluas di dalam pembinaan penyerap gelombang mikro. Walaubagaimanapun, besi oksida sangat mahal, mempunyai sifat menghakis, berat serta tidak biodegradasi.

Penyelidikan ini mengfokuskan kepada penggunaan fiber kelapa sawit (OPEFB) sebagai pengisi untuk menyerap gelombang mikro dengan menggunakan resin epoksi sebagai matrik utama. OPEFB fiber mempunyai beberapa kelebihan termasuk mampu biodegradasi, berketumpatan rendah, murah dan mempunyai sifat terma yang lebih baik. Selain itu, resin epoksi mempunyai pelbagai kelebihan seperti mempunyai stabiliti dimensi yang baik, sifat mekanikal yang baik, mudah untuk diproses, dan dibaiki serta tahan kelembapan. Komposit epoksi-OPEFB direkabentuk dengan peratusan OPEFB yang berbeza (0%, 5%, 10%, 15%, 20%, 25%, 30% dan 40%). Pengisi OPEFB bersaiz 300 µm, resin epoksi, dan pengeras dicampur dan digaul untuk menghasilkan komposit epoksi-OPEFB dengan menggunakan penggaul vortex mini berkelajuan 3000 rpm selama 30 minit dalam suhu bilik (25 °C). Kesemua campuran yang berjisim 12 g itu kemudiannya dituang ke dalam dua bebibir yang berbeza supaya dua teknik pencirian ketelusan dielektrik kompleks boleh digunakan. Pemalar dielektrik dan faktor kehilangan adalah parameter utama yang berkaitan dengan ciri-ciri dielektrik sesuatu bahan, dan secara langsung dikaitkan dengan ciri-ciri penyerap. Ketelusan dielektrik kompleks, pekali transmisi S_{21}, pekali pantulan S_{11}, kehilangan tenaga, kehilangan pantulan dan keberkesanan perisai (SE) telah dikaji pada frekuensi 8 hingga 12 GHz. Pemalar dielektrik, faktor kehilangan, pantulan dan pekali transmisi komposit diukur menggunakan pandu gelombang segi empat tepat (RWG) yang disambungkan dengan penganalisa rangkaian vector (VNA) pada frekuensi 8 hingga 12 GHz. Selain itu, prob sepaksi hujung terbuka (OECP) yang bersambung dengan penganalisi rangkaian vector telah digunakan untuk mengira pemalar dielektrik dan faktor kehilangan untuk setiap komposit pada suhu bilik (25 °C). Pembiasan sinar-X digunakan untuk menganalisa struktur mikro komposit. Keputusan menunjukkan sifat dielektrik bertambah tetapi S_{11} dan S_{21} berkurang berbanding pertambahan peratusan OPEFB dalam komposit. Ketelusan dielektrik kompleks bergantung kepada peratusan OPEFB komposit, pertambahan kandungan OPEFB dalam komposit yang menyebabkan peningkatan ketelusan dielektrik kompleks komposit Epoksi-OPEFB. Komposit Epoksi-OPEFB didapati mempunyai pemalar dielektrik dan faktor kehilangan yang sangat dekat kepada komposit ferit-polimer. Pada 10 GHz, pemalar dielektrik dan faktor kehilangan komposit Epoksi-OPEFB dijumpai dalam lingkungan 2.8 ke 3.35 dan 0.14 ke 0.2. Tambahan lagi, keputusan S_{11} dan S_{12} digunakan untuk menentukan kehilangan tenaga, kehilangan pantulan dan keberkesanan perisai komposit Epoksi-OPEFB. OPEFB mempunyai pengaruh yang tinggi terhadap keberkesanan perisai, kehilangan kuasa dan kehilangan pantulan yang meningkat dengan peningkatan peratusan OPEFB dalam komposit. Nilai keberkesanan perisai keseluruhan dijumpai dalam lingkungan 15.4 dB dan 18.9 dB pada 10 GHz. Pengiraan sampel pekali Parameter-S telah dilakukan menggunakan kaedah unsur terhingga (FEM) menggunakan perisian COMSOL. Pengiraan S_{11} dan S_{21} juga diperiksa. Keputusan simulasi serta pengiraan adalah bersepakat. Akhir sekali, taburan medan elektrik komposit Epoksi-OPEFB telah dilakukan dengan menggunakan perisian COMSOL bagi menyiasat kesan peratusan OPEFB dalam medan elekrik komposit.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS AND SYMBOLS

OPEFB	Oil palm empty fruit bunch
Fe_2O_3	Iron Oxide
OECP	Open Ended Coaxial Probe
RWG	Rectangular waveguide
c	Velocity of light
ε*	Complex permittivity
ε'	Dielectric constant
ε''	Loss factor
EM	Electromagnetic
EMI	Electromagnetic interference shielding
σ	Electrical conductivity
ε	Permittivity
μ	Permeability
SE	Shielding effectiveness
dB	Decibels
FEM	Finite Element Method
XRD	X-ray diffraction
T/R	Reflection/Transmission
S_{11}	Input reflection coefficient of port one
S_{12}	Transmission coefficient port one
S_{22}	Input reflection coefficient of port two
S_{21}	Transmission coefficient port two

TE	Transverse Electric
TM	Transverse Magnetic
TEM	Transverse electromagnetic modes
MoM	Method of Moment
f	Frequency
VNA	Vector Network Analyzer
MW	Microwave
RF	Radio Frequency

CHAPTER 1

INTRODUCTION

1.1 Overview of Electromagnetic Shielding Materials

The extensive growth of telecommunications and electronic equipment has caused a new type of pollution known as electromagnetic interference (EMI). Electromagnetic interference has become a drastically significant problem, disturbing the functioning of electronic devices as well as causing harmful consequences to the health of mankind. Therefore, shielding is needed for protecting the electronic devices related to strategic systems, such as nuclear reactors, aircrafts, control systems, transformers, and communication systems, from inevitable severe electromagnetic radiations. An EMI shield is required to attenuate the electromagnetic radiations either by absorption or reflection, and it could be utilize to reduce the reflection of electromagnetic waves from metallic surfaces, such as, tanks, ships, electronic equipment, and aircrafts (Verma et al., 2015). A variety of materials have been used and developed in the fabrication of shields. Shielding structures could be realized by utilizing absorbing and/or conductive materials such as metallic conductors, polymeric composites non-metallic with and metallic conducting inclusions. These shielding materials must own particular requisites such as lightness, good process ability, low cost, and good mechanical properties. These shields can be achieved with composites which comprise non-conductive polymer matrix reinforced with conductive materials such as cooper, aluminium, stainless-steel or nickel-coated carbon. Consequently, EMI shielding efficiency will increase as the conductivity of the filler increases (Morari et al., 2011). While these fillers enhance the shielding characteristics of the resultant composites, they are disadvantaged by low corrosion resistance, physical rigidity and large density of the metallic phase (Qin et al., 2010). Moreover, metal sheet (such as nickel film, copper film, and iron and cobalt particles) which is a common shielding, also has the aforementioned disadvantages in addition to the possibility of the secondary electromagnetic pollution from reflection. Therefore, it is important to develop new composites which have good EMI shielding characteristics and easy to be fabricated, as well as more portable and economical (Xia et al., 2015). The relative permeability and relative permittivity are the main parameters related to the magnetic and dielectric characteristics of a particular material, and directly associated to their absorbing characteristics (Folgueras et al., 2010). Epoxy resins have been intensively used as a matrix for absorbing composites. Epoxy resins have excellent thermal and dimensional stabilities, high mechanical properties, easy processing and low cost (Gu et al., 2016).

1.2 Absorbing Composites

The polymeric matrix materials with proper and suitable filler, better filler/matrix interaction together with new and advanced methods or approaches are able to develop polymeric composites. This shows a great prospective applications in buildings and constructions, automotive, packaging and aerospace industries. The biodegradability property of the natural fibers is considered as the most important and interesting aspects of their usage in polymeric materials. In addition, polymers are three kinds if classified with regard to reticulation degree; thermosetting polymers, thermoplastic polymers, and rubbers. Thermosets or thermosetting polymers are insoluble in solvents, heavily cross linked and also infusible. Rubbers are slightly cross linked, which prevents the chains from sliding when stretched and the last kind is thermoplastic polymers which have little or no reticulation, are melt easily, and often solvent soluble. In the composite industries most important and applicable thermosetting materials are phenolic resin such as epoxy resin which will be used in this research (Saba et al., 2014). In addition, the natural fiber reinforced polymer composites have become recently highly valuable materials. In this kind of materials, natural fibres are utilized as reinforcing material for polymer based matrices (Al-Oqla and Sapuan, 2014b). Polymer composites reinforced with natural fibres are increasingly utilized in aircraft and dielectric applications (Ahmad et al., 2018). Moreover, the crucial applications of microwave absorbing composites are as follows: military application for antiradar camouflage, to decrease the radar cross section of objects; antenna techniques and improving the antenna parameters; protection of humans and other biological objects from the dangerous effect of the electromagnetic waves; improving the electromagnetic compatibility between different electronic devices, and reducing undesirable reflections from devices and objects (Al-Sehemi et al., 2017).

1.3 Interactions of Materials with Microwaves

The material ability to interact with electromagnetic energy is associated to the complex permittivity (susceptibility or dielectric properties) of the material. This property, in any isotropic, homogenous, and linear dielectric material is measured by a frequency depending absolute complex permittivity. The dielectric constant is a measure for the stored amount of energy from an external electric field in the material while the loss factor accounts for the loss energy dissipative mechanisms in the material. Consequently, a material with a high loss factor is easily heated by microwave (Barba and d’Amore, 2012).

In addition, microwaves have frequencies in the range between 300MHz and 300GHz, which corresponding to wavelengths range from 1m to 1mm. Within this range of the electromagnetic spectrum there are frequencies that are used for radar, cellular phones, and television satellite communications (Gao, 2010).

However, the material characterization is important for the correct selection of a substance for scientific, medical and industrial applications. The dielectric characteristics over a wide temperature range are essential to assess their suitability to utilize in telecommunication, lenses, dielectric resonators, dielectric waveguides, and microwave integrated circuits (MICs).

The EM spectrum consists of different types of EM signals. Microwave behaves likewise to the light wave that travels in straight lines refract, reflect, scatter, interfere and diffract in same physical length. But they vary in the behaviour because of the difference in wavelength (Bahr, 1982).

Microwaves usually never change or heat in any-way the material because of the absolutely low energy which emitted as it would require approximately 1000 emitters to make any measurable effect (Yahaya et al., 2015). The signals can easily penetrate inside dielectric material (electrically insulating). The penetration depth is usually dictated by the loss factor of the material (the ability to absorb energy of microwave), the operation frequency and the transmitted or reflected signal can then be associated to the dielectric properties of the material (Zoughi and Ganchev, 1995).

Several ideas have been applied to adapt the aforementioned phenomena to the microwave applications. There are two critical applications which deal with the utilizing of microwave properties are radar absorbing materials and EMI shielding. The utilizing of microwave technology might be found in different fields such as military, radio, communications, weather monitoring, soil settlement system, environmental remote sensing and forecasting, medical system and astronomy. Today's success in microwave technology can be attributed to decades of tireless efforts, careful research and hard work done by Carl Friedrich, Andre-Marie Ampere, Oliver Heaviside, Michael Faraday, James Clerk Maxwell and Heinrich Hertz (Pozar, 2009).

The interest of this research is in the interaction of microwaves with the materials. It includes absorption parameters of materials, reflection and transmission. These effects are used in various test setups in order to allow quantitative measurements in the materials.

1.4 OPEFB Background

Natural fibres with their so long history of serving humans are very essential in a wide range of applications, and they coexist and compete in the 21st century with artificial fibres, especially as far as sustainability, economy and quality of production are concerned. Natural fibres are an excellent raw materials for the green products. They are fully recyclable, biodegradable, relatively cheap to produce, and are characterized by low specific weight, thermal and acoustic parameters with favourable values (Kozłowski, 2012). Customarily, natural fibres such as hemp, flax or cotton are widely used in the textile industry for yarns and fabrics production. Nowadays, there is a growing interest in using natural fibres as reinforcement for polymer composites (Zafeiropoulos, 2011). Oil palm empty fruit bunch (OPEFB) fibre is one of the biomass which is used as a fuel in the oil palm mills itself to generate energy. Malaysia and South East Asian countries generate big amount of OPEFB fibre as waste around millions of tons per year. When OPEFB waste properly used, disposal problem will be solved and value added products can be created. OPEFB fibres have shown a great potential in utilize as a reinforcing with polymers to develop bio-composite materials. Over the past decades, OPEFB fibre has been studied for producing composite materials using various synthetic polymers for instance polyester, polypropylene, polyurethane, phenol formaldehyde and poly (vinyl chloride). In addition, OPEFB fibres are basically lingo-cellulosic fibres wherein the hemicellulose and cellulose are reinforced in the lignin matrix. Hemicellulose, lignin and cellulose which form major components of the natural fibres might differ depending on growth conditions, plant age and, weather effect, testing methods and soil conditions. The properties of the OPEFB such as flexural strengths, tensile strength, and rigidity rely on the cellulose fibre alignment (Hassan et al., 2010).

1.5 Epoxy Resin Background

Epoxy resins have become very important raw materials in so many fields since the commercialization of the initial synthetic epoxy in the early twentieth century. It can be used as matrices of composites adhesives and insulating paints. Epoxy resin (ER) is normally chosen as the matrix resin for modification because ER is used in many cutting-edge and civil fields (Wang et al., 2015). Epoxy resins (ER) have been commonly used in surface coating, laminates, and painting because of its superior chemical and mechanical properties (Yu et al., 2015). Nowadays, glass fabric-reinforced epoxy resin composites are usually applied in insulation devices, printed circuit boards and different electrical tools (Jiang et al., 2015). Epoxy resins are usually used because of their good performance at high temperature, as well as at room temperature, and its low weight. Epoxy has excellent adhesion characteristics with substrate. The two basic components of epoxy resin are epichlorohydrin and bisphenol A (Bhatnagar, 2016).

The material which reacts with the epoxy to finally form the epoxy network is named the hardener or curing agent. The mixing content of the hardener varies from several percent to as high as fifty percent. A large number of hardeners is able to react with epoxy to finally form a cross-link structure. The curing agent, in most cases, reacts with the available hydroxyl or epoxy groups. There are two kinds of curing techniques for current commercial epoxies. The first one is single constituent epoxies with heat curing, and the second one is multiple constituent epoxies curing at room temperature or at high temperatures. Heat curing usually leads to a relatively greater glass transition temperature and a greater crosslinking density than curing at room temperature. However, due to their rigidity, their peeling strength and toughness will be lower than those cured ones at room temperature (Pacheco-Torgal et al., 2015).

1.6 Measurement Techniques

The interaction between the material and microwaves can be determined by Maxwell's equations and properties of the material. The relations include a range of properties such as reflection, impedance, propagation mode, refraction, and transmission. The permeability and permittivity are complex numbers, the imaginary parts are associated with losses. This complex and rich system of characteristics allows a wide range of measurement methods at microwave frequencies. Some techniques have been used in measuring the electromagnetic characteristics at microwave frequencies. Amongst these techniques are, open-ended coaxial probe technique, free space measurement technique, resonant method and the transmission and reflection line technique. Nevertheless, these techniques are classified into two main groups of measurement depending on sample location: free space measurement and waveguide or coaxial line methods.

The second group of methods have been explained in details by but they are not applicable in many practical cases. The disadvantage of these methods is the high difficulty to fit the sample into the waveguide flange or coaxial fitting with free-air gap. The details of these methods would be described in the subsequent chapters.

1.7 Problem Statement

Good absorbing materials are in high demand to solve electromagnetic interference problems in fields such as industrial and commercial electronics. Mu-metal (nickel-iron alloy) is considered one of the most shielding materials, iron (ferrite) is normally used to develop the absorbing composites. However, ferrites are expensive, corrosive, heavy and non-biodegradable. Usage of ferrites as a shielding material might easily cause galvanic corrosion which increased the non-linearity behavior and reduced its shielding effectiveness. In recent years, plastic materials reinforced with conductive materials are gaining high interests as alternatives to mu-metal product. Nevertheless, these conductive particles are commonly non-biodegradable materials.

This research investigates the usage of oil palm empty fruit bunch fibers as an alternative material to conductive fillers such as ferrite for microwave absorbing applications with epoxy as the host matrix. OPEFB is classified as a waste of palm oil industry which is produced from the process of oil extraction mills. OPEFB has different advantages such as biodegradability, better insulating and thermal properties, low density and low cost. OPEFB provides good flexural strength, stiffness, tensile strength, and elongation at break. In addition, epoxy resin (ER) has been proposed as the host matrix for microwave absorber (Qin and Brosseau, 2012). Epoxy resins have unique characteristics such as low cost, dimensional stabilities and excellent thermal, high mechanical properties and easy processing (Guet al., 2016). Moreover, Epoxy resins are categorized as thermosetting polymers which are used as anticorrosive coating, paints, electronic devices, automotive industry, and adhesive in aerospace. The properties of epoxy resin are widely affected by their molecular structure, curing conditions, amount and type of used hardener (Radoman et al., 2014a). Therefore, epoxy resins are commonly used in preparing the natural-fiber reinforced composites, and in fabricating variety of industrial products because of their superior mechanical, electrical and thermal properties (Ricciardi et al., 2018; Saba et al., 2016a). Various composition of OPEFB filler will provide various absorbing properties of Epoxy-OPEFB composites. High percentage of OPEFB will give high shielding effectiveness value whilst low percentage will give low shielding effectiveness.

1.8 Research Objectives

The main objectives of this work are:

1. To fabricate Epoxy-OPEFB composites with varying percentages of OPEFB (0%, 5%, 10%, 15%, 20%, 25%, 30%, 40%) and characterize the microstructure properties using X-ray diffraction.
2. To determine the dielectric constant and loss factor values of Epoxy-OPEFB composites using the rectangular waveguide and open-ended coaxial probe techniques.
3. To examine the effect of OPEFB on the scattering parameters and total shielding effectiveness values of Epoxy-OPEFB composites using rectangular waveguide technique.
4. To analyze the electromagnetic field distribution in Epoxy-OPEFB composites and evaluate the accuracy of the calculation of S-parameters using finite element method.

1.9 The Study Scope

In this research, absorber composites would be prepared using epoxy resin and OPEFB to enhance the dielectric properties. The effects of OPEFB percentage on the dielectric properties of epoxy matrix would be characterised using rectangular waveguide and open ended coaxial probe techniques. The effects of OPEFB percentage on the scattering parameters of Epoxy-OPEFB composites would also be studied. Comsol software based on finite element method (FEM) is proposed to be used to numerically obtain S-parameters and to simulate the electromagnetic waves in Epoxy-OPEFB composites. The measured and simulated results of S-parameters would be compared. The microstructure properties of the composites would be characterized using X-ray diffraction technique.

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LIST OF PUBLICATIONS

Ahmad, A., Ab Aziz, S., Abbas, Z., Obaiys, S., Khamis, A., Hussain, I., Zaid, M. (2018). Preparation of a Chemically Reduced Graphene Oxide Reinforced Epoxy Resin Polymer as a Composite for Electromagnetic Interference Shielding and Microwave-Absorbing Applications. Polymers, 10(11), 1180.

Submitted Articles

Ahmad Mamoun Khamis, Zulkifly Abbas, Ahmad F. Ahmad, Raba'ah Syahidah Azis, Daw M. Abdalhadi, Ebenezer Ekow Mensah. Experimental and Computational Study on Epoxy Resin Reinforced with Micro-Sized OPEFB Using Rectangular Waveguide and Finite Element Method. IET Microwaves, Antennas & Propagation

Ahmad Mamoun Khamis, Zulkifly Abbas,Ahmad F. Ahmad, Daw M. Abdalhadi, Mohd Amiruddin Abd Rahman, Ebenezer Ekow Mensah. Oil Palm Empty Fruit Bunch Fiber Composites for Microwave Absorber Applications. Electronics Letters.

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