UNIVERSITI PUTRA MALAYSIA

EFFECT OF DIFFERENT FILLERS ON OIL ABSORPTION AND FLEXURAL PROPERTIES OF FILLER-BASED POLYETHYLENE COMPOSITE

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180117

PROJECT REPORT SUBMITTED IN PARTIALLY FULFILLMENT OF
THE REQUIREMENT FOR THE
BACHELOR OF ENGINEERING (PROCESS AND FOOD)

DEPARTMENT OF PROCESS AND FOOD ENGINEERING
FACULTY OF ENGINEERING
UNIVERSITI PUTRA MALAYSIA
2018

ACKNOWLEDGEMENT

First of all, I owe my utmost gratefulness to my project supervisor, Dr. Mohd Salahuddin Mohd Basri. His guidance and advice have contributed to great part of the research. He had also granted me an unlimited supply of material and assistance which eases the specimen manufacturing work greatly. He is a qualified and excellent supervisor in research associated composite material. His teaching would inspire students in the study of material science. For that I would like to thank him gratefully, may his research in futuristic material finds fruitful conclusions.

I also like to thank the examiners, Dr. Mohd Nazli Naim and Dr. Mohd Zuhair Mohd Nor. I really appreciate your advice and suggestion during the presentation of project proposal. I found them very helpful, especially in the manufacturing of material.

Also, thank you to all lab supervisors and technicians from the Department of Food and Process Engineering, Mechanical and Manufacturing Engineering, Chemical Engineering, Institute of Tropical Forestry and Forest Products and Institute of Bioscience UPM. I was an amateur in the field of material fabrication and testing. Without your help, my research would have met a dead end.

Finally I would like to dedicate this thesis to my parents, Mr. Noor Arzahan and Mrs. Maya, my siblings and my best friend, Ahmad Dasuki. May all of you live long and prosper.

ABSTRACT

Fried food products are generally known to its undesirable oil absorbed during frying process. In industry, fried food is being packed into plastic before its oil can fully being tossed. Conventional plastic in the market, made from polyethylene, is not able to absorb excess oil from the fried food due to its nature to resist water and oil. As a result, oil will be accumulated inside the plastic and will affect the freshness and shelf life of the product. This research aims in producing a plastic material, made of polyethylene, which able to absorb oil from fried food (when the food is packed inside the plastic) and at the same time, this plastic material can retain its shape and mechanical strength. Rice husk (RH) and rice husk ash (RHA) were used as fillers while polyethylene (PE) acted as the binder matrix. Five levels of filler loading (10, 15, 20, 25, and 30 wt.%) were incorporated into the PE to produced PE-RH and PE-RHA composites. Two tests were conducted which were oil absorption and flexural test. Based on the results from both tests, addition of fillers at any loading percentages showed significant improvement on the oil absorption and flexural properties of the composites as compared to control sample which is PE. PE-RH and PE-RHA with 25 percent of fillers was found to be the best oil absorbent. For flexural properties, PE-RH with 20 percent of RH and PE-RHA with 30 percent of RHA was found to possess the highest flexural strength. In overall, based on the concept scoring matrix, PE-RH composite with 20 percent of RH and PE-RHA composite with 25 percent of RHA were the best sample which possessed the combination of good oil absorption and flexural properties. This research proved that PE-RH and PE-RHA composites have great potential to be a good oil absorbent.

ABSTRAK

Produk makanan goreng umumnya diketahui menyerap minyak yang berlebihan semasa proses penggorengan. Dalam industri, makanan goreng ini dibungkus ke dalam plastik sebelum minyaknya dapat dibuang sepenuhnya. Plastik konvensional di pasaran, yang terbuat dari polietilena, tidak dapat menyerap minyak yang berlebihan dari makanan goreng disebabkan sifatnya yang tidak menyerap air dan minyak. Hal ini menyebabkan minyak akan terkumpul di dalam plastik dan akan menjejaskan kesegaran dan jangka hayat produk. Penyelidikan ini bertujuan untuk menghasilkan bahan plastik, diperbuat daripada polietilena, yang dapat menyerap minyak dari makanan goreng (apabila makanan dibungkus di dalam plastik) dan pada masa yang sama, bahan plastik ini dapat mengekalkan bentuk dan kekuatan mekaniknya. Sekam padi (RH) dan abu sekam padi (RHA) digunakan sebagai pengisi manakala polietilena (PE) bertindak sebagai matriks pengikat. Lima tahap pengisi (10, 15, 20, 25, dan 30 wt.%) telah dicampur bersama PE untuk menghasilkan komposit PE-RH dan PE-RHA. Dua ujian dijalankan iaitu ujian penyerapan minyak dan ujian lenturan. Berdasarkan hasil dari kedua-dua ujian ini, penambahan pengisi keatas mana-mana peratusan menunjukkan peningkatan yang ketara terhadap kadar penyerapan minyak dan sifat lenturan komposit dibandingkan dengan sampel malar iaitu PE. PE-RH dan PE-RHA dengan 25 peratus pengisi didapati sebagai penyerap minyak terbaik. Bagi sifat lenturan, PE-RH dengan 20 peratus RH dan PE-RHA dengan 30 peratus RHA didapati memiliki kekuatan lenturan tertinggi. Secara keseluruhan, berdasarkan matriks pemarkahan konsep, PE-RH komposit dengan 20 peratus RH dan PE-RHA komposit dengan 25 peratus RHA adalah sampel terbaik yang mempunyai gabungan penyerapan minyak yang baik dan sifat lentur. Kajian ini membuktikan bahawa komposit PE-RH dan PE-RHA mempunyai potensi besar untuk menjadi penyerap minyak yang baik.

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LIST OF SYMBOLS AND ABBREVIATIONS

%wt	Weight percentage
°C	Degree celcius
g	gram
OAC	Oil absorption capacity
ORC	Oil retention capacity
PE	Polyethylene
RH	Rice husk
RHA	Rice husk ash
SEM	Scanning electron microscope
µ	micron

CHAPTER 1

INTRODUCTION

1.1 Overview

Rice (*Oryza sativa* L.) is one of the major crops and a primary source of food for billions of people around the world. A statistics from Food and Agriculture Organization of the United Nations showed that, between 2010 and 2013, the average annual global production of rice was 725 million metric tons (FAO & FOODS, 2004). Asia region alone producing over 90 percent of the total global rice production.

Rice husk, a by-product of rice processing is one of the major agricultural waste. Rice husk is separated from rice grain during the rice milling process. It is reported that, for every ton of rice produced, about 0.23 tons of rice husk is formed (Lim *et al.* ,2012). Rice milling is one of the most important industry in many countries such as China, India, Indonesia, Malaysia, and Bangladesh (Aprianti *et al.*, 2015).

In the paddy plants of Malaysia, with a land area of approximately 680,000 hectares, a total of 840,000 tons of rice husk is produced every year (Isa *et al.*, 2011). These rice husks can be processed into activated carbons which are used as adsorbents in water purification or in the treatment of industrial wastewater. It would help to reduce the cost of waste disposal and provide a potential cheap alternative to existing commercial carbons (Chuah *et al.*, 2005).

Rice husk is a cellulose-based fibrous material with a wide range of composition. Due to its high availability, low bulk density, toughness, and unique composition, a variety of applications have been proposed by many researchers. Rice husk has the potential to be utilized as an insulating material, in the production of organic chemicals (Das *et al.*, 2014), panel boards, activated carbon, and supplementary cementing material (Aprianti *et al.*, 2015).

Potential of rice husk as the source of power generation and its financial viabilities have also been studied and have produced encouraging results (Sookkumnerd *et al.,* 2001). Even though some of this husk is converted into the mentioned end products, however, the industrial applications of this biomass are still limited with little economic value. Therefore, it is very important to find pathways to fully utilize rice husk to identify its potential applications and to develop economically feasible processes for these applications in a commercial scale.

1.2 Problem statement

Fried food products are generally known to its undesirable oil absorbed during frying process. Fried food products require longer time to toss the oils before it can be served to consumer. In industry, fried food is being packed into plastic before its oil can being fully tossed. Since time is very important in any production, fully tossed the oil from fried food will require longer time. Conventional plastic in the market, made from polyethylene, is not able to absorb excess oil from the fried food due to its nature to resist water and oil. As a result, oil will be accumulated inside the plastic and will affect the freshness and shelf life of the product. Although using paper can be an alternative to absorb oil, utilization of this absorbent material will add to the overall production cost. Therefore, this research aims in producing a plastic material made of polyethylene which able to absorb oil from fried food (when the food is packed inside the plastic) and at the same time, this plastic material can retain its shape and mechanical strength. Besides, problems regarding the utilization of rice husk, which is one of the biggest agricultural waste in the world, may be reduced.

1.3 Research Objectives

The main objective of this research is to study the oil absorption and flexural properties of rice husk-based polyethylene (PE-RH) and rice husk ash-based polyethylene (PE-RHA) composites.

Along with the main objective, there are several specific objectives including:

1) To determine the effect of different loading of RH and RHA on the oil absorption and flexural properties of PE-RH and PE-RHA composite.
2) To characterize the microstructure characteristics of PE-RH and PE-RHA composite and their potential as oil absorbent.

CHAPTER 2

LITERATURE REVIEW

2.1 Rice husk (RH) and rice husk ash (RHA)

Rice husk is a by-product from rice mill that was used as an energy source in many industries such as biomass power plant and rice mill. Burning rice husk will generates rice husk ash (RHA) which contain high amount of silica and can be an economically valuable raw material for production of natural silica (Siriluk and Yuttapong, 2005).

The incorporation of rice husk as cellulose-based fibrous material into polymer matrices increase its characteristics, such as biodegradability, light weight, toughness, and resistance to weathering. The final products produced are more economically competitive. Table 2.1 shows the components and physical properties of rice husk (Ludueña *et al.,* 2011).

Table 2.1 Components and physical properties of rice husk

Property	Rice Husk
Components (%)	
Cellulose	25-35
Hemicellulose	18-21
Lignin	26-31
SiO_2 (silica)	15-17
Solubles	2-5
Moisture content	5-10
Physical properties	
Particle size (μm)	26.64
Surface area (m^2/g)	0.92
Density (g/cm^3)	1.00

Ismail *et al.* (2003) studied on rice husk burnt in open air which yields two types of ashes that can be served as fillers in plastics materials, namely, white rice husk ash (WRHA) and black rice husk ash (BRHA). The upper layer of the RHA mound is exposed to open burning in air and yields BRHA as carbonized layer. The inner layer of the mound being subjected to a higher temperature profile yields WRHA that consists predominantly of silica resulted from the oxidation of the carbonized ash. Table 2.2 shows the chemical and physical properties of typical RHA fillers.

Table 2.2 Chemical and physical properties of typical RHA fillers
(G. Pritchard, 1998)

Property	WRHA	BRHA
Chemical composition (%)		
CaO	0.360	0.120
MgO	0.160	0.078
Fe_2O_3	0.041	0.022
K_2O	0.690	0.950
Na_2O	0.034	0.018
Al_2O_3	0.025	0.023
P_2O_5	0.570	0.270
SiO_2 (silica)	92.200	53.880
Loss of ignition (LOI)	1.620	44.480
Physical properties		
Particle size (μm)	6.60	19.50
Surface area (m^2/g)	1.40	26.80
Density (g/cm^3)	2.20	1.80

It can be observed that WRHA contained approximately 92 percent of silica, while BRHA contained 54 percent of silica and 44 percent of carbon compound. Utilization of rice husks as possible sorbent materials for oil spills has been discussed in several studies. Kumagai and Matsuo (2013) studied the use of carbonized rice husk on the absorption of oil. After pretreatment with steam and refining, the husk was pyrolyzed in a vacuum at 500 Pa between 300°C and 800°C. Maximum uptake capacity of 6.7 g heavy oil/g as-prepared husk was obtained in which indicates their usefulness as an adsorbent for oil spill cleanup. Rather than their porosity, the residual fluid components in the carbonized rice husks are closely related to oil adsorption capacity.

In a study conducted by Kenes *et al.* (2012) , the rice husk is first pretreated via thermal treatment under carbon dioxide at 200 ml/min flow between 300°C and 800°C for one hour. It was observed that the density of the sorbate had a direct effect on the sorption capacity. The lowest density hydrocarbon (gasoline q = 0.734 g/ cm3) showed a sorption capacity of 4.5 g/g whereas the highest density hydrocarbon (heavy crude petroleum q = 0.937 g/cm3) showed a sorption capacity of 15 g/g.

Nwankwere *et al.* (2010) acetylated rice husks using N-bromosuccinimide (1 percent NBS) as a catalyst. Acetylation for one hour and 3.5 hours increased the crude oil sorption from 1.9 g/g to 8.2 g/g and 10.3 g/g, respectively.

Chuayjuljit *et al.* (2003) successfully obtained silica (SiO_2) from RHA and explored the possibility of using the silica as antiblocking agent in LDPE film. Properties of RHA silica were compared with commercial silica, as shown in Table 2.3. The suitable amount of silica used as an antiblocking agent in LDPE film was also investigated. The results indicated that silica prepared from RHA showed less porosity than that of commercial silica, resulting in a lower specific surface area. They found that the addition of RHA silica to LDPE film modifies the film blocking behavior. However, the blocking is reduced not as much as in the case of commercial silica. It was found that, as the amount of silica increased, certain mechanical properties including tensile strength, elongation at break, and tear strength were decreased markedly. This is due probably to lower bulk density and larger average particle size of silica.

Table 2.3 Properties of RHA silica and commercial silica

Properties	RHA Silica	Commercial Silica
Average particle size (micron)	4.27	6.61
Specific surface area ($m^2 g^{-1}$)	131.25 ± 2.34	511.97± 4.97
Bulk density ($g cm^{-3}$)	0.667	0.319
pH	5.7	6-8
Oil absorption($g g^{-1}$)	1.97	2
Silica content (% SiO_2)	99.6	99.0

2.2 Polyethylene (PE)

Polyethylene is a thermoplastic polymer with variable crystalline structure and an extremely large range of applications depending on their particular type. It is one of the most widely produced plastics in the world which marked tens of millions of tons are produced worldwide each year. Strong (2000) defined polyethylene (PE) as a thermoplastic that contains chains of monomer called ethylene. Polyethylene is commonly used in plastic bags, plastic milk cartons, and water bottles. There are three main types of PE which are high density polyethylene (HDPE), low density polyethylene (LDPE), and linear low-density polyethylene (LLDPE). These types of PE differ in the shapes of the PE molecules arise from the changes that exist during the polymerization reaction. Different temperatures, pressures and catalysts during the polymerization reaction may affect the branching of PE molecules. Branching is the formation of side chains off the basic polymer backbone. With a significant amount of branching on a PE molecule, the structure will become longer and has uninformed chains. The application of all types of PE are dependent on their properties. High density polyethylene (HDPE) is a strong, high density, moderately stiff plastic with a highly crystalline structure. Bleach and detergent bottles are usually made of HDPE because the bottles need to be thin yet strong in order to hold its shape. Food container, milk jugs and folding tables are made of HDPE as well. Meanwhile, low density polyethylene (LDPE) is a very flexible material with unique flow properties that makes it particularly suitable to plastic film applications such as shopping bags. Linear low-density polyethylene (LLDPE) is very similar to LDPE with the added advantage that the properties of LLDPE can be altered by adjusting the formula constituents and that the overall production process for LLDPE is typically less energy intensive than LDPE. The LDPE has high ductility but low tensile strength which is evident in the real world by its tendency to stretch when strained. As compared to LDPE and HDPE, the LLDPE is more suitable in producing plastic wrap and stretch wrap.

2.3 Oil absorption properties

Aboul-Gheit *et al.* (2006) conducted a study on absorption ability of waste plastic for spilled oil from seawater. The first objective of the study was to find a useful application of the waste plastics, namely polyethylene (PE) and polypropylene (PP). The second objective was to find solution of the oil spill problem in aquatic locations. They used polyethylene and polypropylene waste powders and sheets to absorb the oil from water, via simple contacting for few minutes. The effect of contacting the plastics powders and sheets to a low dose of γ-irradiation (3 Mrad) on the activation of the current polymers was investigated. Differential scanning calorimetric (DSC) and scanning electron microscopic (SEM) examinations of the oil sorbing polymers were performed to throw light on the behaviors of these polymers. The oil absorption test was conducted by mixing 1 ml of crude oil with 50 ml of fresh water in a batch contactor and the mixture was stirred for 2 minutes. Then, it was left for 20 minutes. Different mass of waste plastic powder or sheet were added to the oil-water mixture and agitated for 10 minutes. The water was then completely separated from the oil containing the waste plastic. The absorption efficiency is defined by the percent ratio of the weight of oil absorbed by the polymer and weight of initial oil in water.

Polyethylene powder and sheets as well as polypropylene powder showed higher absorption capability of heavier crude oil than light crude oil. On the other hand, polypropylene sheets absorb more light crude oil than heavier crude oil.

In a previous study conducted by Deng *et al.* (2010), water uptake of woven fabric-based self-reinforced polypropylene (SRPP) composite laminates based on co-extruded tapes were analyzed. The mechanical properties of this composites were observed and compared with commercial glass mat reinforced PP (GMT) and natural fibre mat reinforced PP (NMT). Although all-PP composites described in this paper showed significant water uptake through the presence of voids between tapes, the amount of water uptake and their mechanical properties are not significantly affected by water absorbed. Conversely, the NMT used in this research demonstrated significant mechanical degradation with increasing water uptake. In this study, oil absorption was determined by measuring the change in buoyant force. According to the Archimedes principle, the buoyant force of a solid is equal to the weight of the volume of fluid it displaces when submerged. Fluid absorbed by the solid lowers the buoyant force by reducing the volume of fluid displaced. Hence as the buoyant force decreases, the apparent weight of the solid in the oil increases. This variation of weight of the immersed solid is equal to the weight of the oil absorbed. The weight of oil absorbed can be practically determined by using a standard density measuring apparatus attached to a microbalance. The oil absorbed in weight percent is derived in Equation 2.1 where $m_{oil(t=0)}$ is the apparent weight of the specimen when first immersed in oil, $m_{oil(t)}$ is the apparent weight of the specimen in oil after time, t, and m_{air} is the weight of the specimen in air.

$$Oil\ absorbed\ \left(\%\frac{w}{w}\right) at\ time\ (t) = \frac{\mathrm{moil(t)} - \mathrm{moil(t{=}0)}}{mair} \qquad \text{(Eq 2.1)}$$

2.4 Composites

Fávaro *et al.* (2010) conducted a study on high-density polyethylene (HDPE) composites containing different concentrations of rice husk. HDPE and rice husk were chemically modified to improve their compatibility in composite preparation. Rice husk was treated with sodium hydroxide (NaOH) solution and was acetylated. The chemically modified fibers were characterized by fourier-transform infrared spectroscopy (FTIR) and carbon-13 nuclear magnetic resonance spectroscopy (13C NMR). The composites were prepared by extrusion of modified and unmodified materials containing 5 and 10 percent fibers. The morphology of the obtained materials was analyzed using scanning electron microscope (SEM). The chemical modification of the fiber surface was found to improve its adhesion with matrix. Results from flexural and impact tests demonstrated that HDPE-RH composites improved mechanical performance comparatively to the pure polymer matrix. On the contrary, no benefit is observed in the tensile strength over the pure PE.

Yang *et al.* (2004) studied the possibility in using lignocellulosic materials as reinforcing fillers in the thermoplastic polymer composite. Polypropylene were used as the matrix and rice husk flour as the reinforcing filler to prepare a particle-reinforced composite. In determining the physical, mechanical and morphological properties of the composite based on the filler loading in respect to the thermoplastic polymer, four levels of filler loading (10, 20, 30 and 40 percent) were prepared. Six levels of test temperature (-30,0,20,50,80 and 110°C) and five levels of crosshead speed (2, 10, 100, 500 and 1500 mm/min) were designed for the tensile test. From the results, it showed that increased in loading resulted in reduced the tensile strengths and improved tensile modulus. Notched and unnotched Izod impact strengths were lowered by the addition of rice husk flour. The composite became brittle at higher crosshead speed and showed plastic deformation with increasing test temperature.

2.5 Flexural properties of PE-composites

Premalal *et al.* (2002) conducted a study on unmodified and ground talc and rice husk (RHP) fillers in which were compounded with polypropylene (PP) separately to obtain composites with 0 to 60 percent php (per 100 part of polymer) of filler at 15 percent intervals. The mechanical properties of the composites with reference to filler type and filler loading were investigated. In terms of mechanical properties, young's modulus and flexural modulus increased, whereas yield strength and elongation at break decreased with the increase in filler loading for both types of composite. Of these PP composites, the RHP composites exhibited lower yield strength, young's modulus, flexural modulus, and higher elongation at break compared to that of talc composites. Scanning electron microscopy (SEM) was used to examine the structure of the fractured surface and to justify the variation of the measured mechanical properties.

Sachudhanandan *et al.* (2015) used rice husk as the major reinforcement and aramid as an additional fiber in their study to improve the mechanical property of polymer composite. The base material used was vinyl ester and been prepared by hand layup process according to ASTM standards. Test specimens were prepared based on different weight fractions of rice husk. At the optimum point of tensile test, a small percentage (3, 5 and 7 percent) of aramid are added, tests were conducted and the improvement in mechanical properties (tensile strength and flexural strength) of the hybrid composite material was observed. From the results, it showed that there was significant increase in the tensile strength and flexural strength of the composite. With the addition of rice husk to the vinyl ester, the tensile strength increased by 11.65

percent at 10 percent fiber loading and decrease by 8.34 percent at 15 percent of rice husk loading. Further addition of rice husk will only decrease the strength of the material. The flexural properties of rice husk composite increased by 8.90 percent and 12.80 percent at 5 percent coir loading and 10 percent rice husk loading, respectively. Meanwhile, the flexural strength increased further by 22.80 percent at 5 percent aramid.

I Wayan *et al.* (2014) reported on mechanical properties of rice husks fiber polyester composites. Composites were produced with unsaturated polyester resin as matrix and rice husks as fiber. Composites were made by hand layup techniques with different fiber weight fraction of 20, 30, 40, and 50 percent. Tensile test and flexural test specimens were prepared according to the ASTM D3039 and ASTM D790M, respectively. The results showed an increase in the tensile and flexural strength of the composites when the fiber weight fraction increased.

Atuanya *et al.* (2013) discussed on the effect of rice husk filler loading (10, 20, 25, 30, and 35 percent) on the mechanical properties of recycled low-density polyethylene (RPE) mixed with 20 percent weight fraction of virgin polyethylene (MPE) composites. The waste polyethylene was blended with virgin polyethylene and the composites of RPE and MPE were moulded with the addition of rice husk filler using injection moulding machine at 150MPa of pressure and temperature of 160°C. The composites were cut into specified dimensions and mechanical properties were conducted. Tensile strength increased up to 10 percent weight fraction of rice husk filler in the composites and before it decreased with an addition of more than 10 percent filler loading. Tensile modulus, flexural strength and modulus, and Brinell hardness increases with increased filler loading, but impact strength decreases with increased in filler loading. Rice husk filler loading had significant effect ($p < 0.05$) on the mechanical properties of MPE composite as compared to RPE composite which indicated that rice husk filler may be useful as reinforcement in polyethylene

2.6 Potential application of PE-composites

As one of the by-products of rice production, rice husk (RH) has high probability to cause environmental pollution as it is difficult to be disposed and therefore its proper utilization is necessary. Since the main composition of RH are carbon and silica, it could be used in adsorption processes for removal of toxic heavy metals from water and wastewaters (Uddin, 2018). Today, rice husk is widely available and easy to obtain in large amount. Because of its high specific surface area, it was proved to be a potential low-cost material for water treatment and building materials applications. This economically valuable agriculture waste product is a great source of silica and potentially used in wide range of applications.

The unique properties such as high surface area, large adsorption capacity, and fast adsorption kinetics makes activated carbons as a valuable material for different industrial applications (Babaso and Sharanagouda, 2017). Production of activated carbon from rice husk can be achieved through activation with chemical or physical means (Della *et al.*, 2002; Rahman *et al.*, 2005; Tongpoothorn *et al.*, 2011; Van and Thi, 2014; Alvarez *et al.*, 2015; Mehta and Ugwekar; 2015). In addition, the nano porous activated carbon with large specific area of 2523.4 m^2 g^{-1} was successfully obtained from RH (Xu *et al.,* 2014).

High silica content of RH makes it insoluble in water, having good chemical stability, and high structural strength (Lee *et al.,* 1994). It plays an important role in water purification as well as in waste water treatment. Sorbent made by RH effectively used as removal agent for six heavy metals including iron, manganese, zinc, copper, cadmium, and lead (Munaf, 2010; Daifullah *et al.*, 2003; Yalcın and Sevinc, 2000). Moreover, RH is used as adsorbent to remove various pollutants, phenols, dyes, pesticides, inorganic anions, organic compounds, and heavy metals (Chauh *et al.,* 2005; Gupata *et al.,* 2006; Lata and Samadder, 2014).

RHA can be effective as an oil spill absorbent, used in waterproofing chemicals, flame retardants, and as a carrier for pesticides and insecticides (Kumar *et al.*, 2012). Besides, it was used to keep stored potatoes free from the potato tuber moth (Phthorimaea operculelle) for up to 5 months of storage (Das and Rahman, 1997).

CHAPTER 3

METHODOLOGY

3.1 Sample preparation

Preparation of rice husk-based polyethylene (PE-RH) and rice husk ash-based polyethylene (PE-RHA) composites were conducted in the *Laboratory of Biocomposite Technology (Makmal Teknologi Komposit), Institute Of Tropical Forestry And Forest Products, Universiti Putra Malaysia.*

3.1.1 Materials

Fillers used for the study are rice husk and rice husk ash while low density polyethylene was used as the binder.

Rice husk (RH) and rice husk ash (RHA) were obtained from *Maerotech Sdn. Bhd.* RH is brown in colour and slightly heavier and thicker compared to RHA. The mean particle size measured by particle size analyser is 52.30 μm for RH and 16.51 μm for RHA. The average density for RH is 0.57 g/cm^3 and 0.59 g/cm^3 for RHA.

Low density polyethylene (LDPE) was obtained from *Mylab Scientific Sdn. Bhd.* It is an odorless, chemically stable white particle that is generally used in plastic bags. The melting point and density are 116°C and 0.925g/mL, respectively.

3.2 Experimental design

Samples with different composition are shown in Table 3.1 and Table 3.2. The composition of the samples was calculated based on weight ratio.

Table 3.1 Sample composition of PE-RH composite

WEIGHT (%wt)	
PE	RH
100	0
90	10
85	15
80	20
75	25
70	30

Table 3.2 Sample composition of PE-RHA composite

WEIGHT (%wt)	
PE	RHA
100	0
90	10
85	15
80	20
75	25
70	30

3.3 Sample preparation

The composite was prepared by weighing the materials based on the calculation made beforehand. The sample weight in the mixer were determined based on the formula in Equation 3.1, where

m= mass of the sample material,

V= mixer volume (55 cm^3 for roller blades),

ρ= density of material, and K= constant 0.8.

$$m = V(cm^3) \times \rho \left(\frac{g}{cm^3}\right) \times K \qquad \text{(Equation 3.1)}$$

The actual mass of the fillers and binder was increased by 5 percent to account for wastage during manufacturing process. RH or RHA and LDP was mixed using Brabender Internal Mixer. The mixer is heated up at 130°C for a period of 5 minutes at preheating stage. The RH or RHA and LDP were poured into the mixer and allowed to be stirred at a speed of 50 rpm for 10 minutes. Once the mixer stopped, the front plate was opened to collect the RHA- or RH-based polyethylene composite. Similar steps were repeated for all sample compositions listed in Table 3.1 and Table 3.2. The composite were pressed using hot plate presser to obtain a thin sheet composite. The thickness of all samples was 1±0.05mm.

3.4 Microstructural analysis

Scanning Electron Microscope (SEM) was carried out in the *Institute of Bioscience, Universiti Putra Malaysia*. The equipment used to obtain the microstructure images of the samples was the Hitachi S-3400N variable scanning electron microscope.

3.5 Flexural test

Flexural test was conducted in the *Laboratory of Material Strength (Makmal Kekuatan Bahan), Faculty of Engineering, Universiti Putra Malaysia.* The equipment used to perform the flexural test is the Instron 5900 Series Universal Testing Instrument with a load cell of ±5kN. The tests were carried out according to the ASTM. First, the specimen was placed horizontally on the center of crosshead. The crosshead was then adjusted manually to barely touched the specimen to prepare for the machine preloading sequence. After that, the flexural test began with the preset crosshead speed of 1mm/min. The test was carried on until there is sudden drop in the load cell, which indicates that the specimen has failed under flexure.

3.6 Brabender internal mixer

The composites were prepared by mixing the fillers (RH and RHA) with PE binder in a Brabender internal mixer. The mixing process consisted of distributive mixing where the fillers were spread over different positions within the chamber and dispersive mixing where the application of high shear rates was required to overcome the fillers agglomeration. Different compositions were prepared with different weight percent ranging from 10,15,20,25 to 30 percent of fillers. After mixing, the material was compression molded at a temperature of 130°C to form thin sheet composite.

3.7 Oil absorption test

The oil absorption capacity was determined by first weighing the sample before immersed it into oil. Once immersed in the oil, the samples were stored at room temperature. The samples were weighed again on day 2,4,6,8 and 10 to determine the amount of oil absorbed. The oil absorption capacity was calculated using Equation 3.2 where m_{final} is sheet weight after dipping in the oil and $m_{initial}$ is initial sheet weight.

$$OAC\ \left({}^{g}/_{g}\right) = \frac{m_{final} - m_{initial}}{m_{initial}} \times 100 \qquad \text{(Equation 3.2)}$$

After oil absorption capacity were measured, the samples were wrapped using aluminum foil, placed under steel weight (1 kg) for 1 to 2 minutes and were then weighed again. The oil retention capacity was calculated using Equation 3.3 where m_{final} is sheet weight after dipping in the oil and $m_{initial}$ is sheet weight after pressing.

$$ORC\ (\%) = \frac{m_{final} - m_{initial}}{m_{final}} \times 100 \qquad \text{(Equation 3.3)}$$

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Surface morphology

Sample of rice husk (RH), rice husk ash (RHA), and polyethylene (PE) are observed under scanning electron microscope (SEM) to study their surface morphology.

4.1.1 Fillers

Figure 4.1 shows SEM images of RH and RHA. The image of RH shows rough surface along with some granules as compared to RHA, and the same result was obtained by Kunal, *et al.* (2015). The outer surface of RH is composed of dentate rectangular elements, which are mainly comprised of silica in which it is coated with thick cuticle and surface hairs. The middle part and inner epidermis contain small amount of silica (Brenzoak, 2003). Jauberthie *et al.* (2000) reported that the presence of amorphous silica is concentrated at the surfaces of the rice husk and not within the husk itself.

RHA were found to be solid in nature with amorphous forms as with cristobalite and trace crystalline quartz (Shinohara and Kohyama, 2004). Crystalline and amorphous forms of silica are obtained based on temperature range and duration of burning of the husk (Christopher *et al.*, 2017). The amorphous forms of silica are composed of silica tetrahedral arranged in a random three-dimensional network without regular lattice structures. The structure is open with holes in the network where electrical neutrality is not satisfied, and the specific surface area is also large due to disordered arrangement. This helps to increase the reactivity, since large area is available for reaction to take place (Ilochonwu *et al.*, 2016).

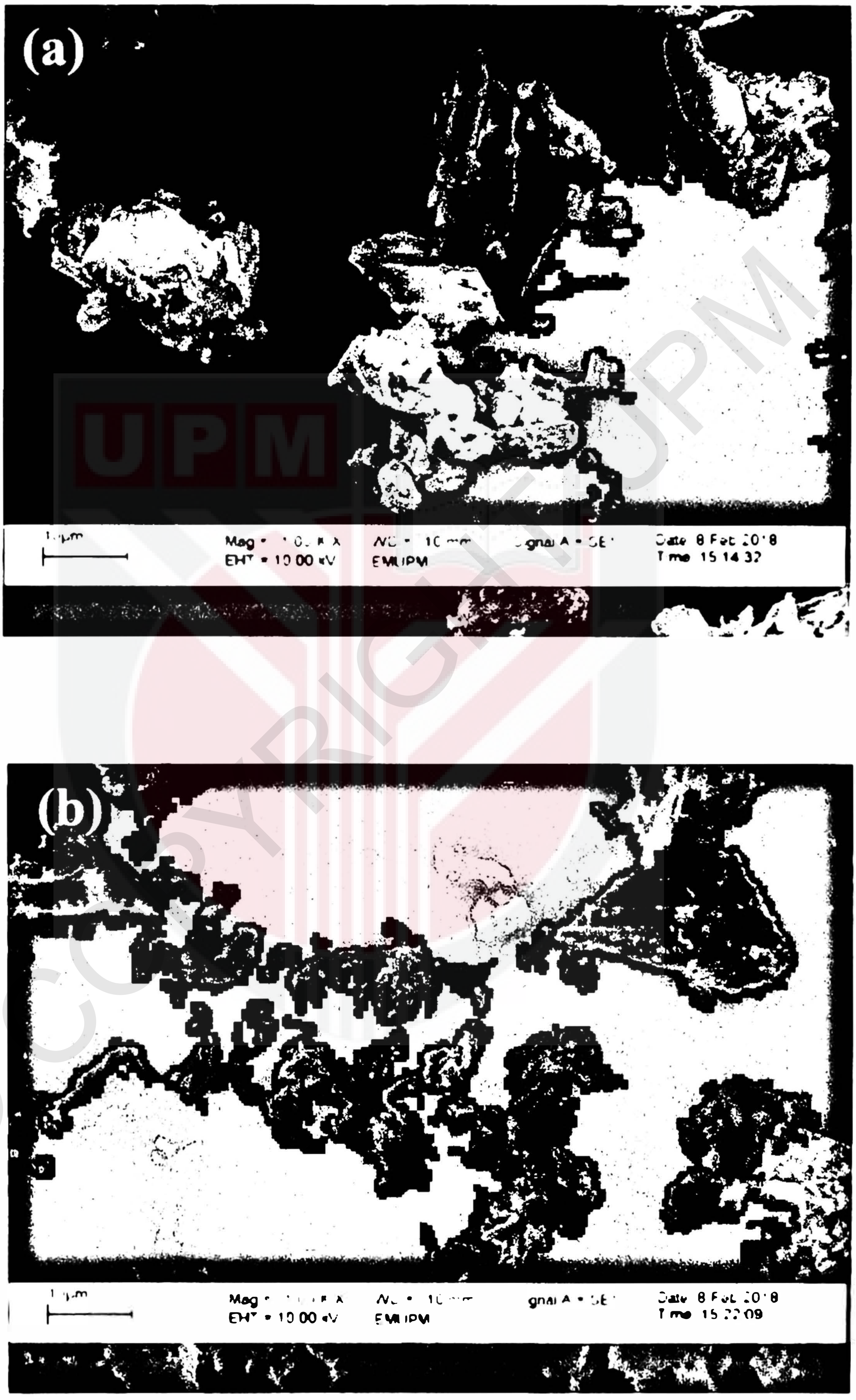


Figure 4.1 SEM micrographs of (a) rice husk (b) rice husk ash

4.1.2 Polyethylene

Morphological images of PE are shown in Figure 4.2. It can be seen that the surface of the film is smooth, which indicates a strong intermolecular bonding between fine particles of PE. Similar result was obtained by Wang and Wang (2017). According to Ozaltin *et al.* (2016), PE exhibits a homogenous, relatively smooth surface morphology with a minor uniform fiber-like feature stem. Ali *et al.* (2016) also found that low density polyethylene films were smooth and plane after curing. However, the surface roughness increased after degradation process.

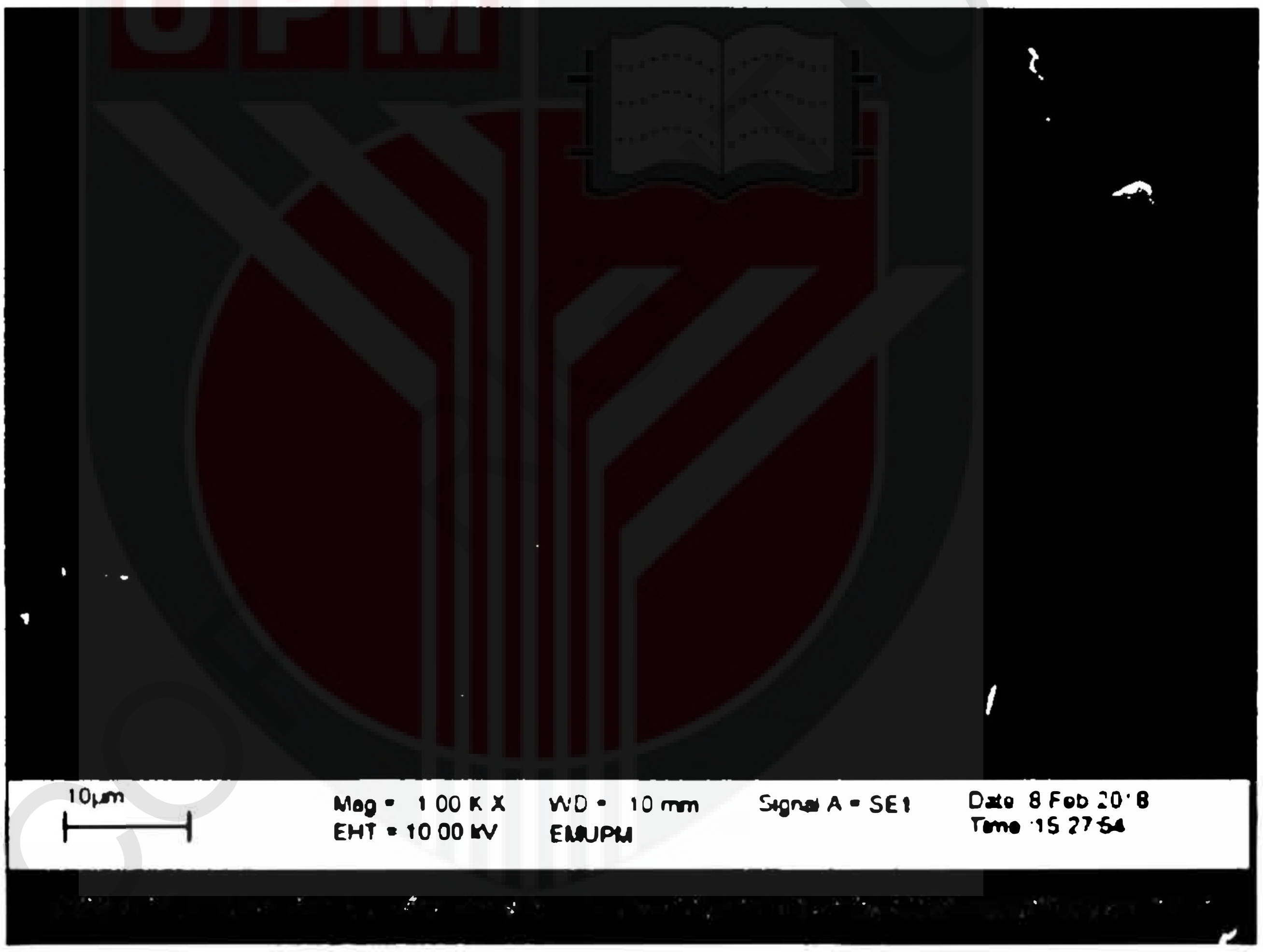
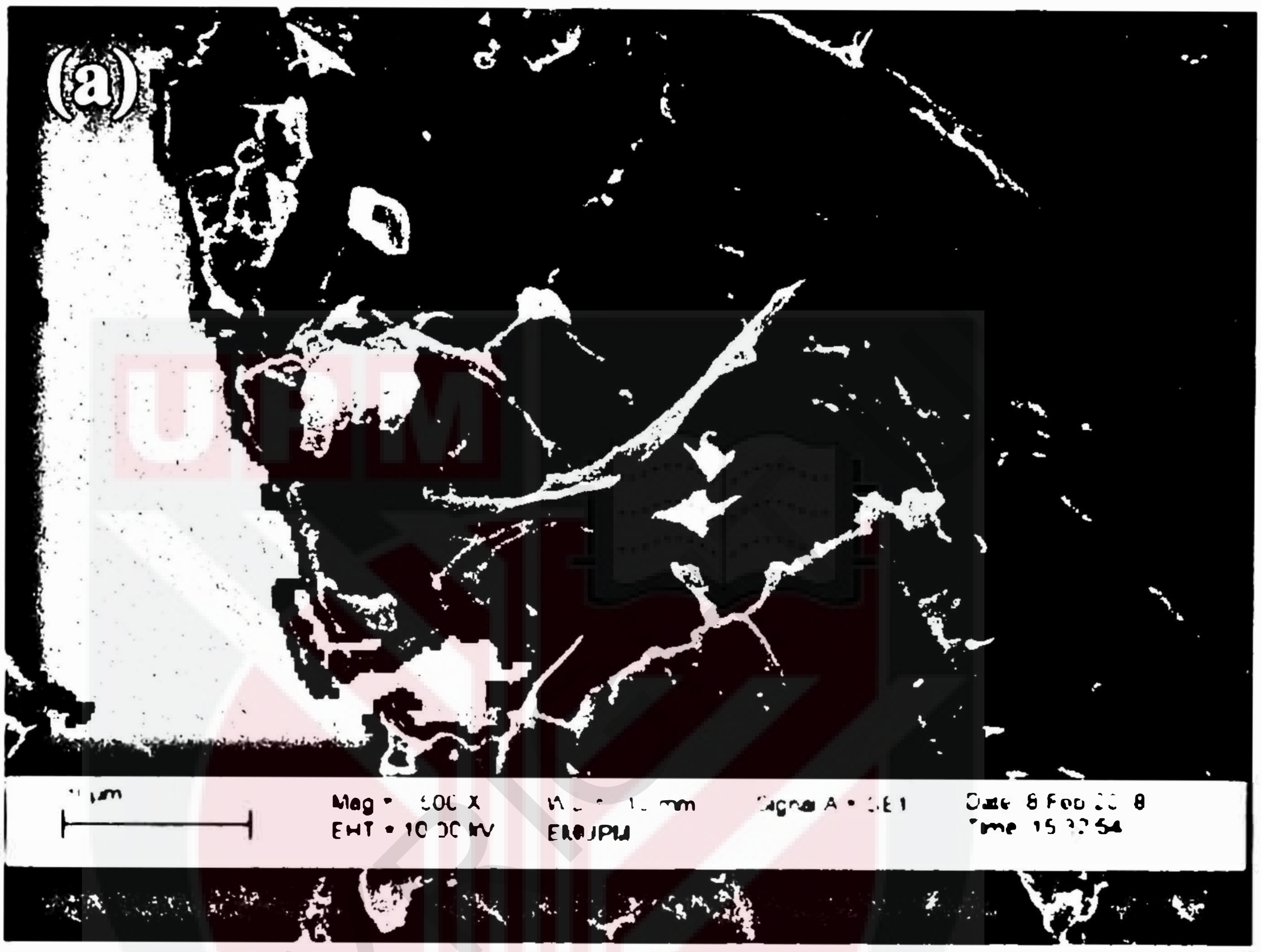
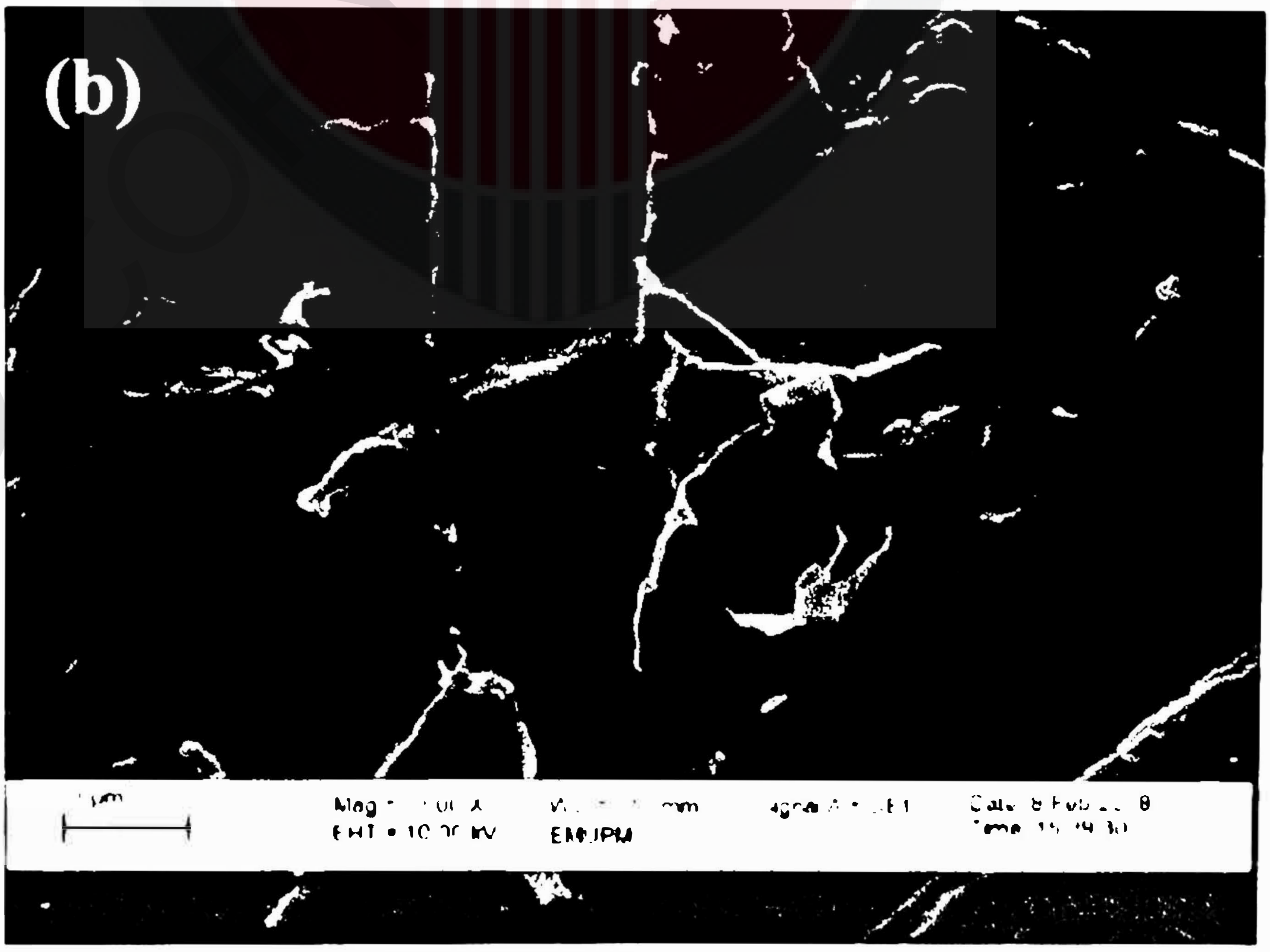


Figure 4.2 SEM micrographs of polyethylene film

4.2 Effects of RH and RHA loading on microstructure of PE-RH and PE-RHA composites

Polyethylene-rice husk (PE-RH) and polyethylene-rice husk ash (PE-RHA) composite with highest and lowest loading are analysed using SEM.





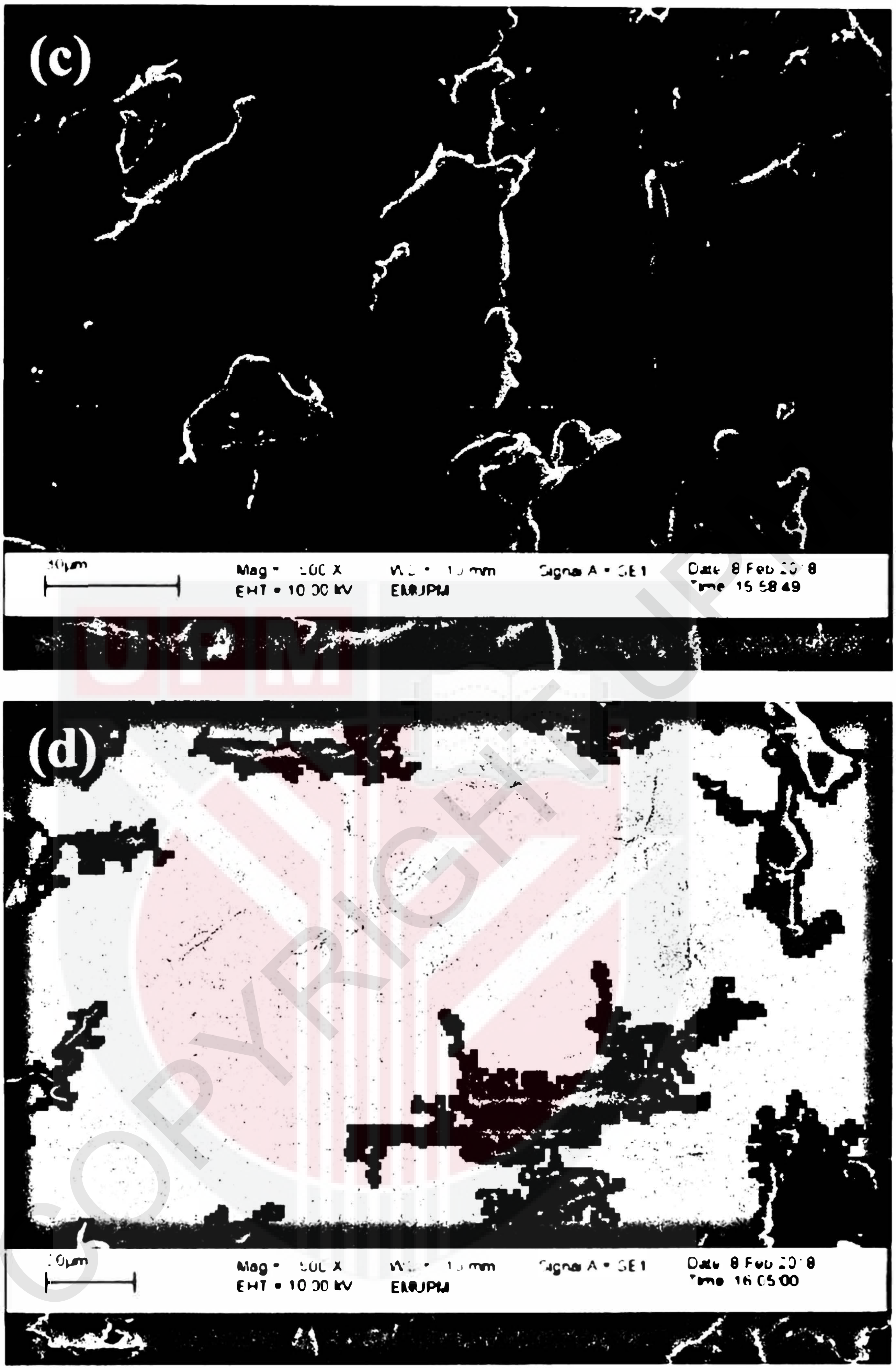


Figure 4.3 SEM micrographs of (a) PE-RH composite with highest loading (b) PE-RH composite with lowest loading (c) PE-RHA composite with highest loading (d) PE-RHA composite with lowest loading

Figure 4.3 (a) shows the surface of PE-RH composite with highest loading of RH which is 30 percent. It is clearly display that the fillers are dispersed randomly with some traces of rice husk can be seen accumulate in certain areas. This may possibly due to higher amount of RH and resulted in less homogenous composite mixture. The surface of PE-RH composite with lowest loading, which contained around 10 percent RH, is shown in Figure 4.3 (b). Due to lower amount of rice husk, the filler was found to mix well with the PE and the accumulation of the fillers is lower. As the filler loading increased, more pulled-out rice husk particles were observed (Hafizuddin, *et al.*, 2014). According to Daramola, *et al.* (2017), an increased in the content of fillers would lead to a larger agglomerates due to the formation of hydrogen bonds among the abundant hydroxyl groups and adsorbed water on their surface.

Figure 4.3 (c) and (d) show the surface of PE-RHA composite with highest loading of RHA (30 percent) and lowest loading of RHA (10 percent), respectively. Result shows that the RHA mixed well with PE compared to that of RH. Lower loading of RHA resulted in smoother PE-RHA composite compared to the higher loading of RHA. RHA able to decrease the volume of large pores and subsequently change the pores from continuous to discontinuous one. These changes make the structure appears more homogeneous and the matrix system become denser. The surface morphology of PE-RHA composite with higher RHA loading was found to be uneven. Although spaces between filler and matrix are smaller as compared to the lower loading of RHA, the interaction between both filler and matrix is weak and the adhesion bonding is poor (Tiwari *et al.*, 2015).

4.3 Effects of RH and RHA loading on oil absorption of PE-RH and PE-RHA composites

Oil absorption test provides information about the effects of different fillers loadings on the mass of oil uptake against time. Result for each composite showed different absorption rate as in Figure 4.4 with 0 percent of filler (100 percent PE) acts as a control.

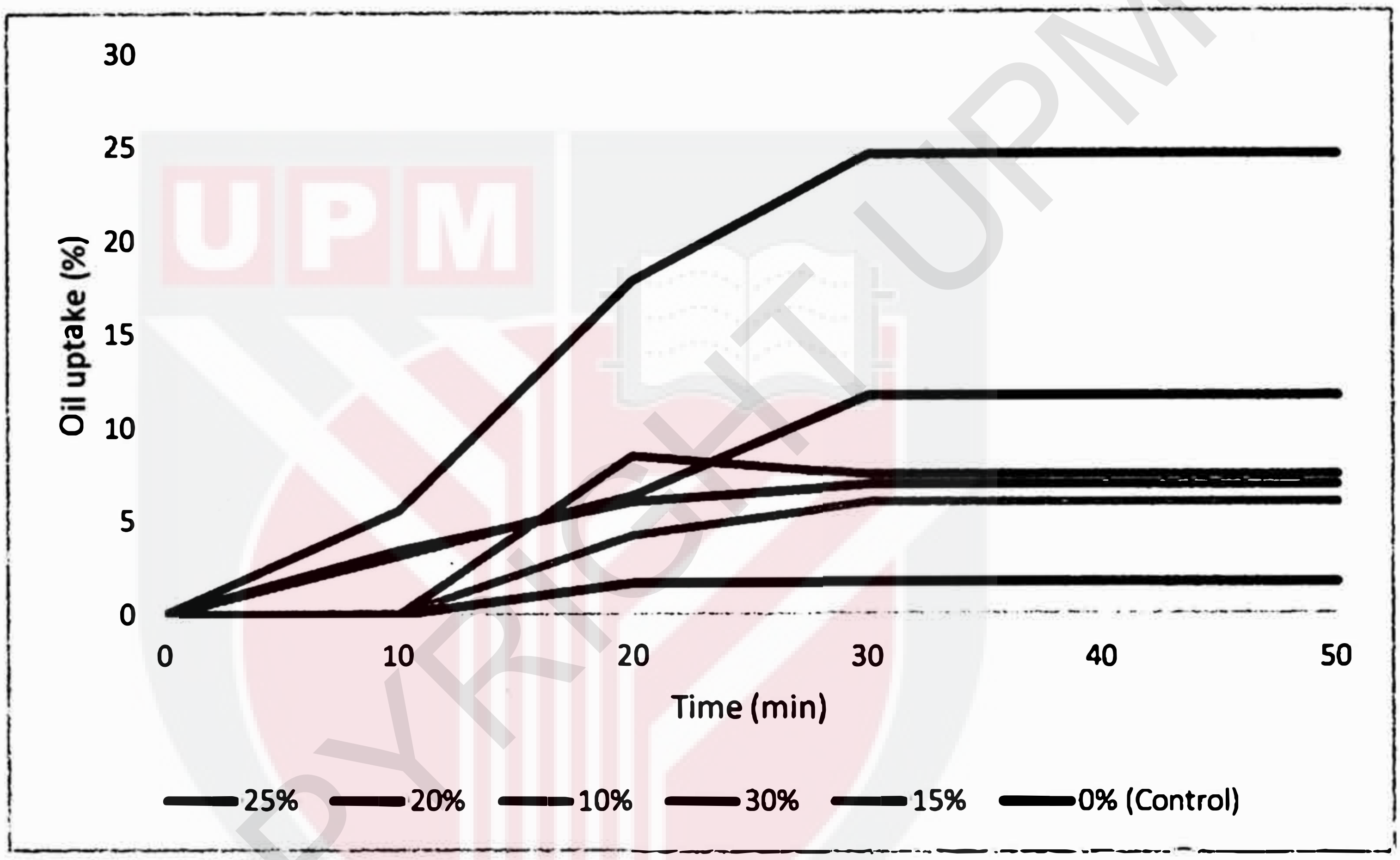
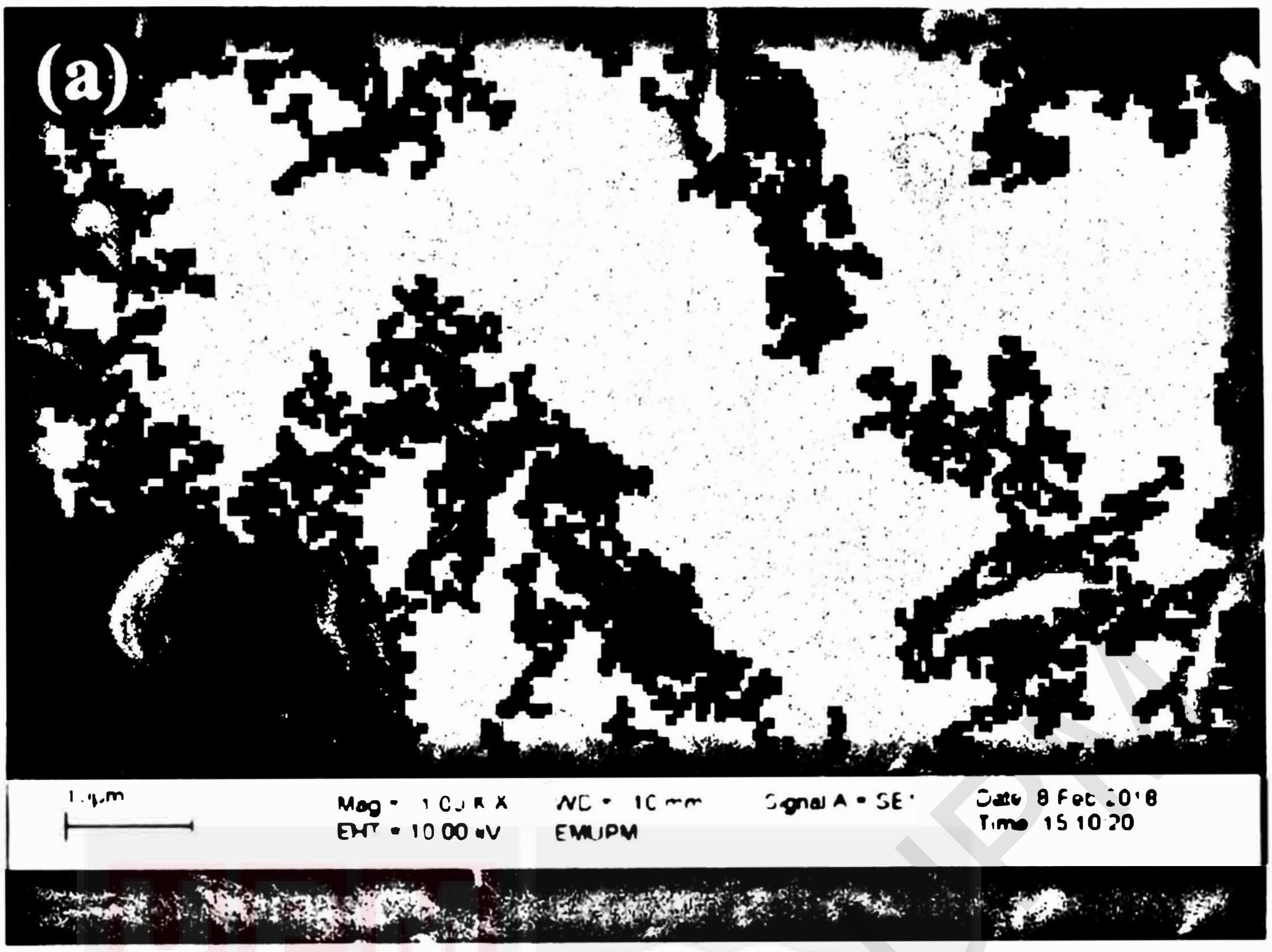


Figure 4.4 Effect of RH loading on oil absorption properties of PE-RH composite

Composites contained filler loading of 25, 20 and 10 percent appears to be the good, moderate and poor oil absorbent, respectively. It was observed that mass of oil uptake is increased with an increase in filler loading. However, further increase in filler loading, which is above 25 percent, does not give a significant difference to the rate of oil sorption capacity. This result is in agreement with Idris *et al.* (2014). He found that the efficiency of oil sorption capacity depends on the filler loading.

The effect of contact time on the sorption capacity of PE-RH composite were studied. Based on Figure 4.4, PE-RH composite with 25 percent filler showed the best oil absorption performance. The sorption capacity increases with the contact time within the first 10 minutes. High rate of oil uptake may be attributed by the presence of vacant voids on the sorbents surface. However, after 20 minutes, less vacant voids were available to be occupied due to the repulsive forces between the solute molecules on the solid and bulk phases. Therefore, the oil uptake achieve equilibrium (Razavi, *et al.*, 2015). Time for PE-RH composite to reach equilibrium is 30 minutes and the good absorbent able to absorb oil up to 25 percent. Similar result was reported by Kudaibergenov *et al.* (2013).

Figure 4.5 (a) shows SEM images of PE film after oil absorption test. There is no significant difference as compared to the sample before oil absorption test. It shows that the smooth plastic surface without any cellulose-based fibre is not a good absorbent. Figure 4.5 (b) to (d) show results for the PE-RH composite with different RH loadings.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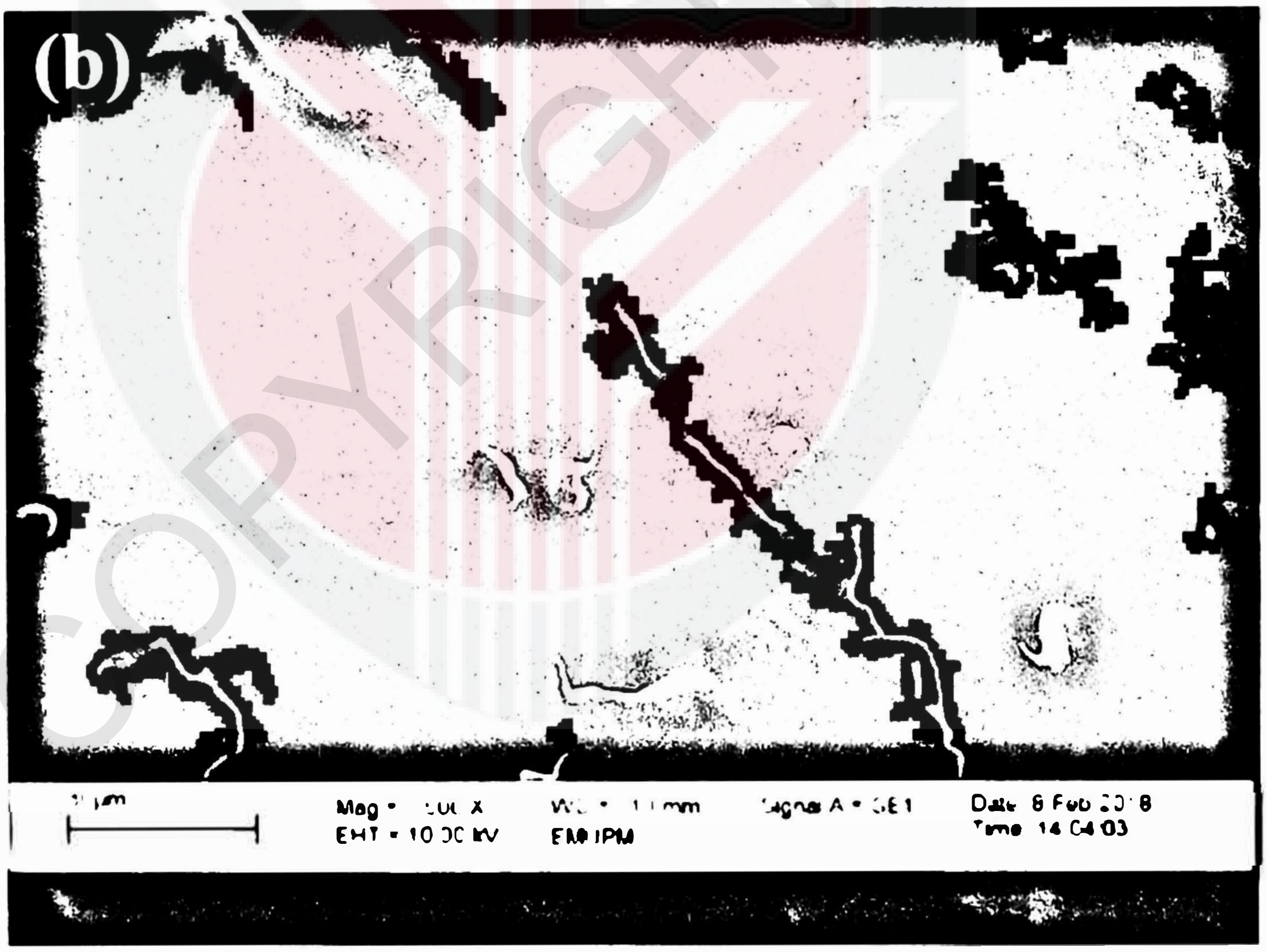
Time: 15:10:20


(b)
µm
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EHT = 10.00 kV
WD = mm
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Signal A = SE1
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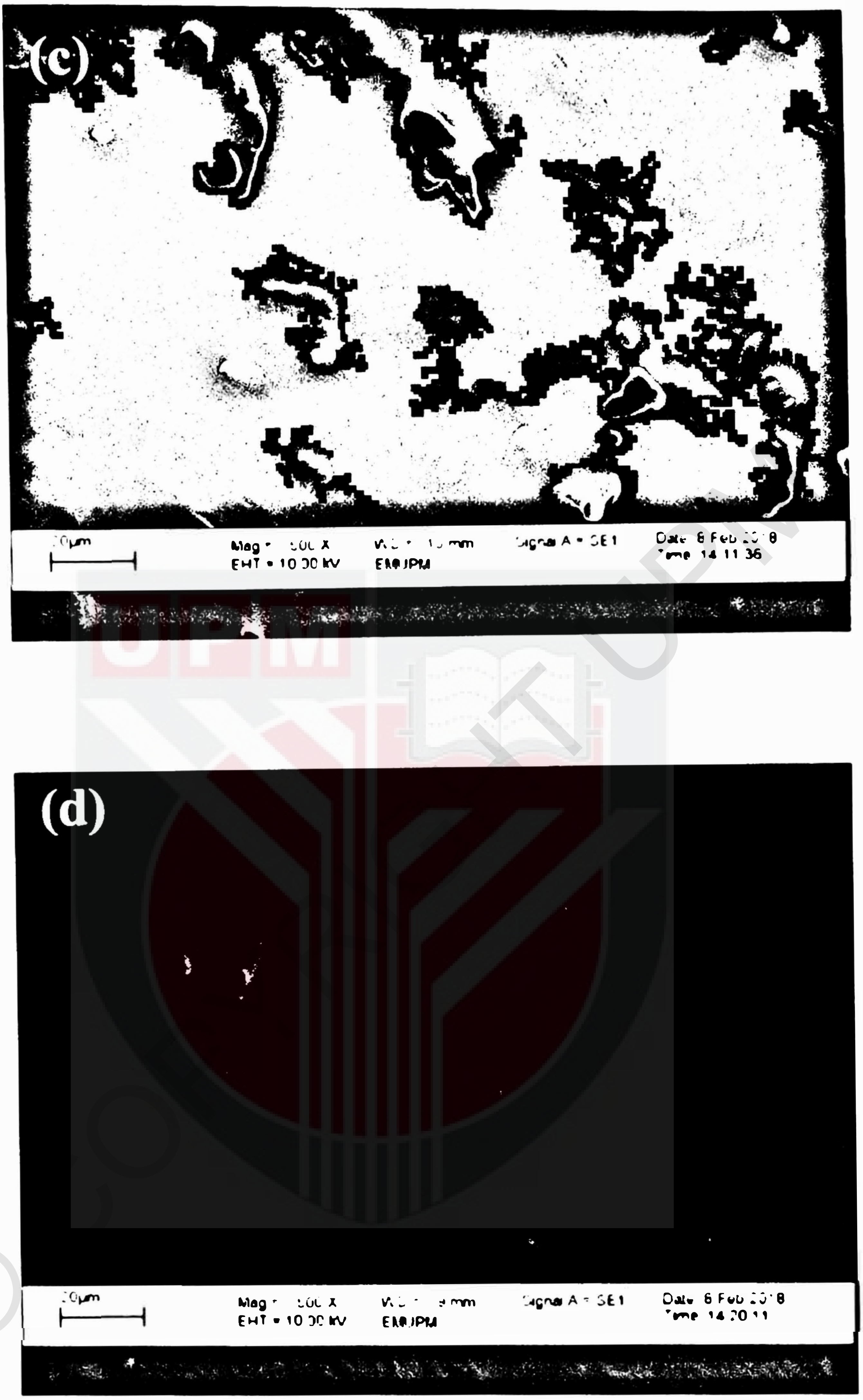

Figure 4.5 (a) Polyethylene film after oil absorption test (b) Good oil absorbance property PE-RH composite (c) Moderate oil absorbance property PE-RH composite (d) Poor oil absorbance property PE-RH composite

The SEM micrographs in Figure 4.5 revealed that PE-RH composite contained numerous pores which able to transport and hold oil. The pores vary in sizes and are distributed over the present silica craters. The spiky whitish materials are silica bodies, and underneath are perforations, which aid oil absorption (Idris *et al.,* 2014). Higher amount of spaces between rice husk and polyethylene may allow more oil to be absorbed into the composite. It indicates that higher filler content in the composite resulted in higher percentage of oil uptake.

Figure 4.6 shows the relationship between total oil uptake and time taken to reach equilibrium for five different composition of PE-RHA composites. PE-RHA composite with filler loading of 25 percent appears to be a good oil absorbent, filler loading of 20 percent and 10 percent was found to have a moderate and poor oil absorption capacity, respectively. From the result, it can be concluded that, PE-RH composite has better oil absorption capacity compared to PE-RHA composite.

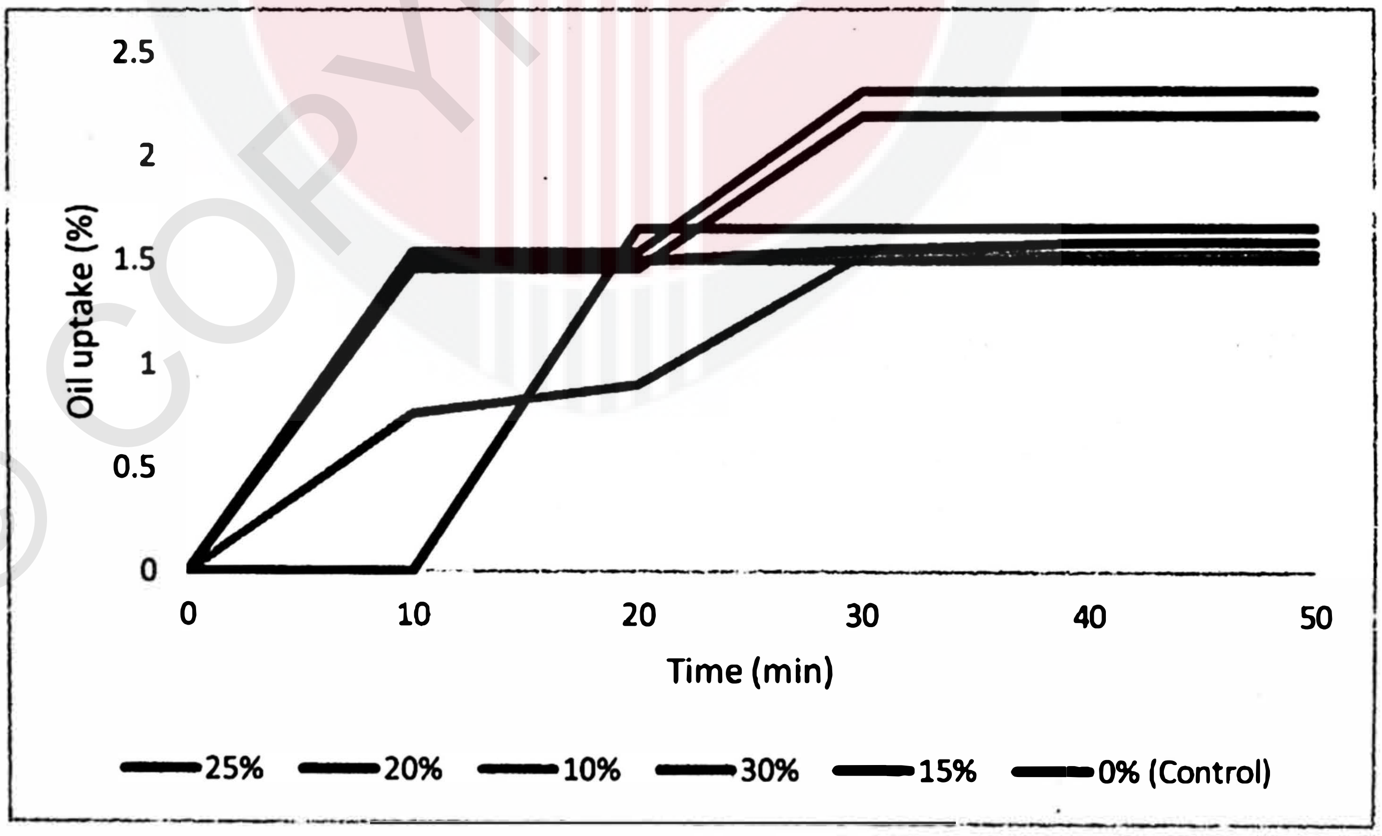

Figure 4.6 Effect of RHA loading on oil absorption properties of PE-RHA composite

Total mass of oil uptake increases due to the availability of more active areas on the surfaces of the composite which allow more oil absorption activity to take place. After attaining an optimum sorbent dose, the equilibrium between sorbate, which is the oil, and sorbent, which is the composite, at the operating conditions occurred (Razavi *et al.*, 2015).

A decrease in the particle size of RHA may probably be the reason of lower oil absorption capacity. Previous research found that the oil absorption was improved with an increase in the particle size of natural organic sorbents. Generally, grinding the sorbents caused damage to the particles and destroyed the pores, thus the sorbent was unable to hold more oil on the surface and the absorption rate was reduced (Razavi *et al.,* 2015).

Figure 4.7 (b) to (d) show the magnification images of SEM for PE-RHA composite with different filler loading. An increase in the oil sorption capacity was found to be influenced by a decrease in the particle size of the filler. This may probably due to an increase in surface area of the filler. On the other hand, with the increase in particle size of RHA, the oil sorption properties decrease. The reason for this behaviour might be due to the accumulation of small particles on each other, which resulted in plugging the pores and capillaries present between fibers and the polymer (Alaa El-Din, *et al.,* 2017).


(a)
1 μm
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EHT = 10.00 kV
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EMUPM
Signal A = SE1
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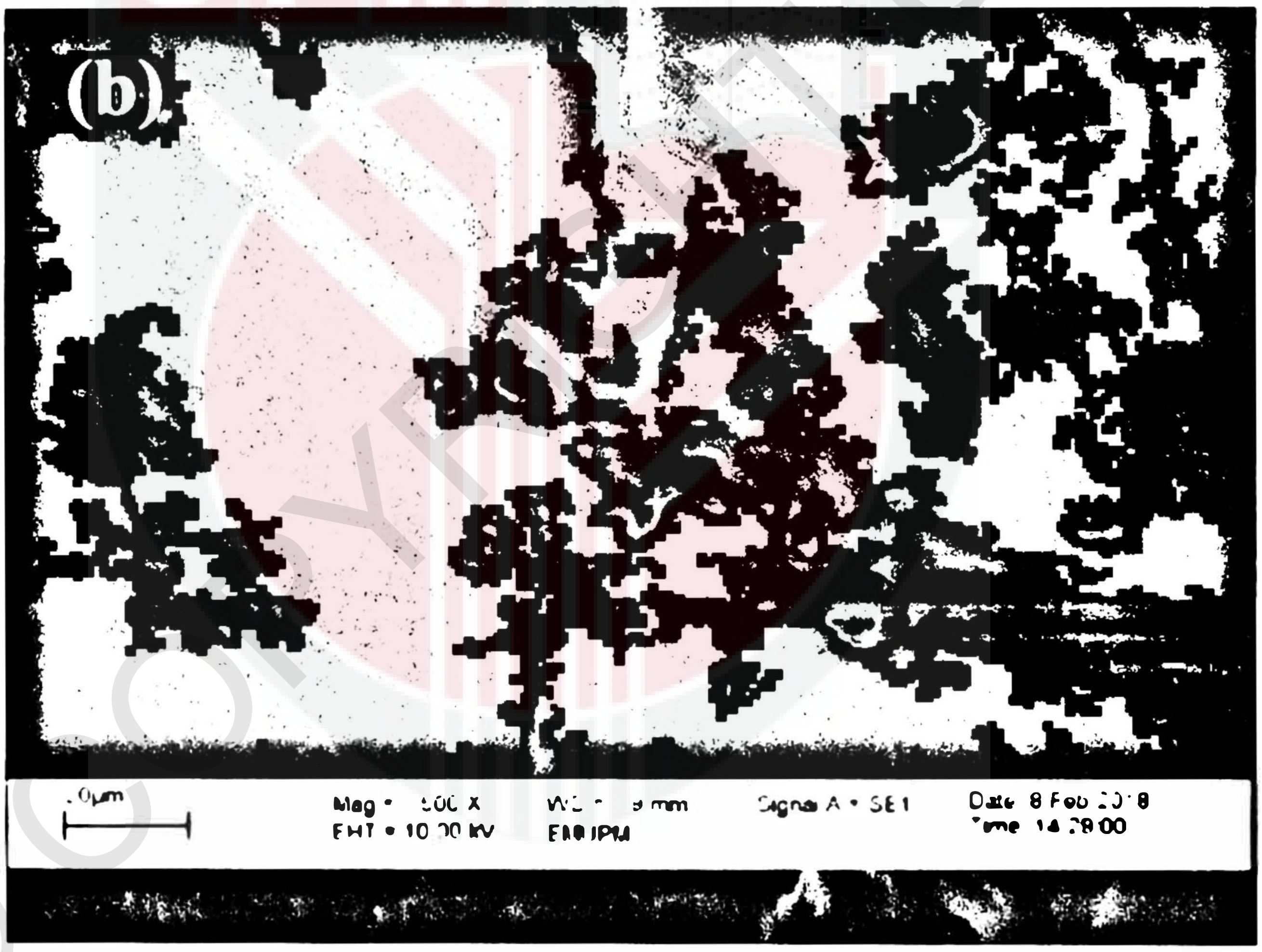
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Signal A = SE1
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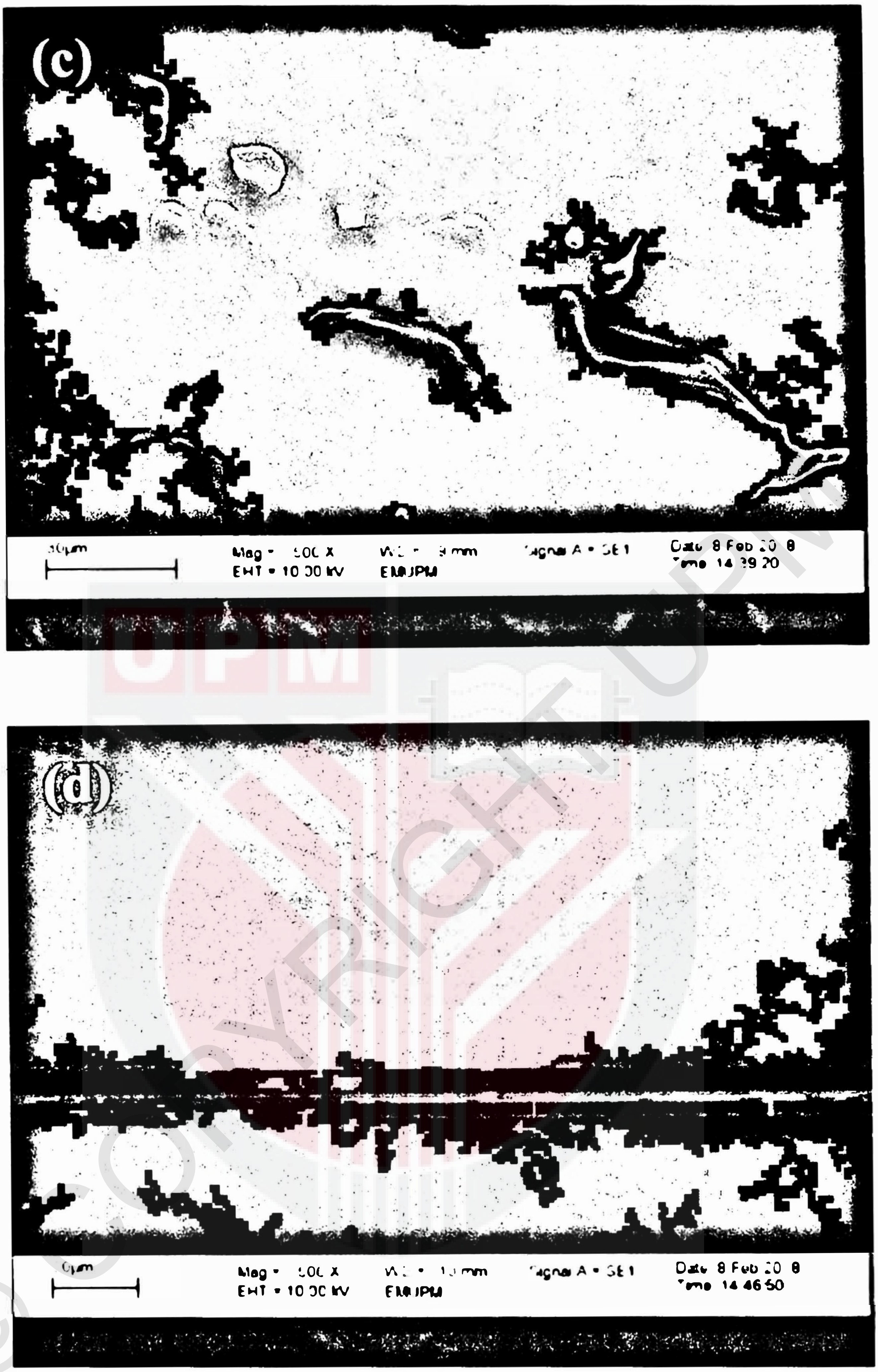


Figure 4.7 (a) polyethylene film after oil absorption test (b) good oil absorbance property PE-RHA composite (c) moderate oil absorbance property PE-RHA composite (d) poor oil absorbance property PE-RHA composite

4.4 Effects of RH and RHA loading on flexural properties of PE-RH and PE-RHA composite

Flexural test was conducted on PE-RH composites and PE-RHA composites. The results are presented in Figure 4.8 and 4.10. Figure 4.8 shows the effect of different fillers loading on flexural strength of PE-RH composites. It was observed that 20 percent of fillers loading has the highest flexural strength, which is around 73 percent higher than PE film without fillers (control). However, further increase in fillers loadings (25 percent and 30 percent) caused the flexural strength to significantly drop below the value of the PE film without fillers. Similar result was obtained by Atuanya *et al.* (2013) where the flexural strength increased steadily with an increased in the rice husk filler loading as well as flexural modulus. This indicate that the increase in filler loading will resulted in the increase in the stiffness of the RH-polyethylene composite. This was reported to be a common phenomenon and was discussed by many researchers (Hafizuddin *et al.*, 2014; Tong, *et al.*, 2014; Yang *et al.*, 2004).

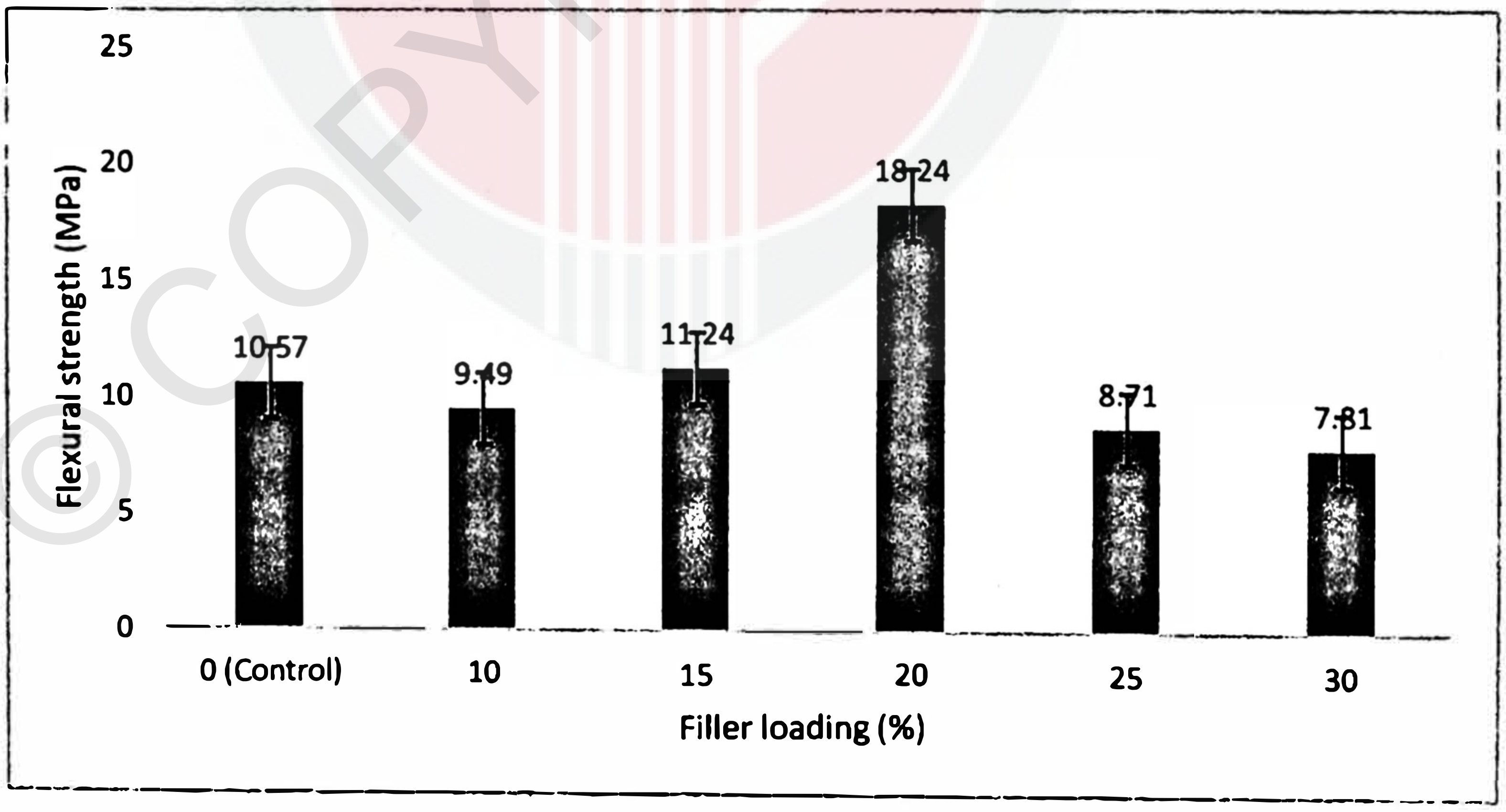
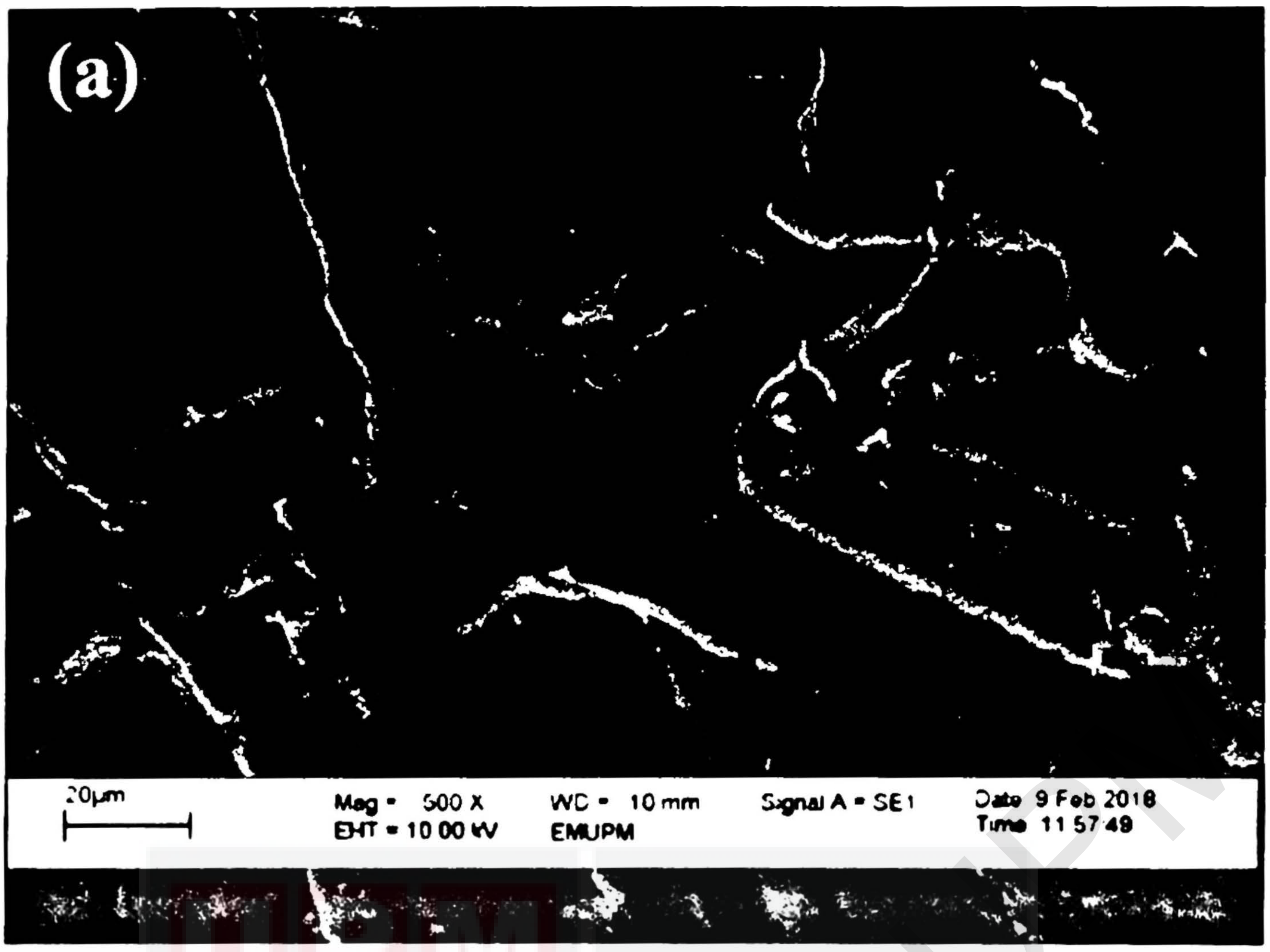


Figure 4.8 Flexural strength of PE-RH composite

According to Tong *et al.* (2014), further increase in the filler loading after the optimum loading was obtained is not highly recommended. In the study, flexural strength increased along with filler loading up to 35 percent, but the strength was decreased at 40 percent. The flexural modulus of RH reinforced composites increased with higher filler loading. Similar results with different reinforced materials were reported in previous researches by Rahman, *et al.* (2010).

Study conducted by Hafizuddin *et al.* (2014) concluded that the amount of RH may not exceed the optimum loading. The plausible reason was due to weak interfacial adhesion between r-matrix and rice husk. Results obtained in compliance with previous study suggested that RH present in thermoplastic composites may enhanced its mechanical properties. RH was found to improve the stiffness properties by hindering the movement of matrix molecules. The movement of matrix molecules was more difficult with increasing filler content. The stiffness of composites will decrease drastically after the optimum points have been reached.

The SEM micrographs in Figure 4.9 (b) to (d) show that composite with higher loading of RH (30 percent), which exhibited poor flexural properties, has more visible cracks compared to the composite with lower loading of RH (20 percent) which exhibited good flexural properties. During bending process, the surface of the composite underwent tension stress, which resulted in shear cracks. This crack was initiated from voids, which are near the fillers or space between fillers and matrix. Higher volume of shear cracks formed in PE-RH composite with poor flexural properties (Figure 4.9 (d)) as compared to that of PE-RH composite with good flexural properties (Figure 4.9 (b)). The formation of shear cracks weakens the yield strength of the composites.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EMUPM
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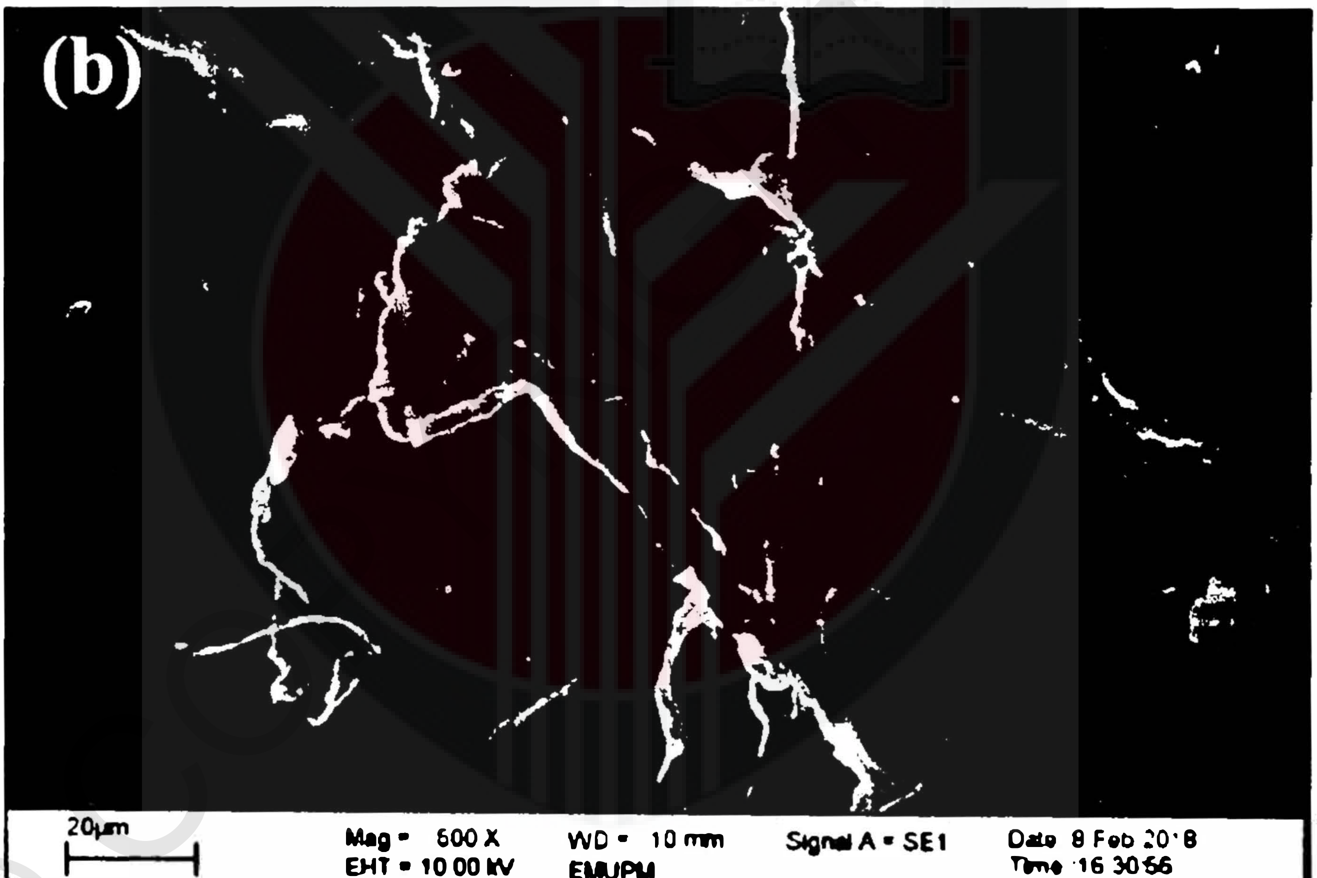
Date 9 Feb 2018
Time 11:57:49


(b)
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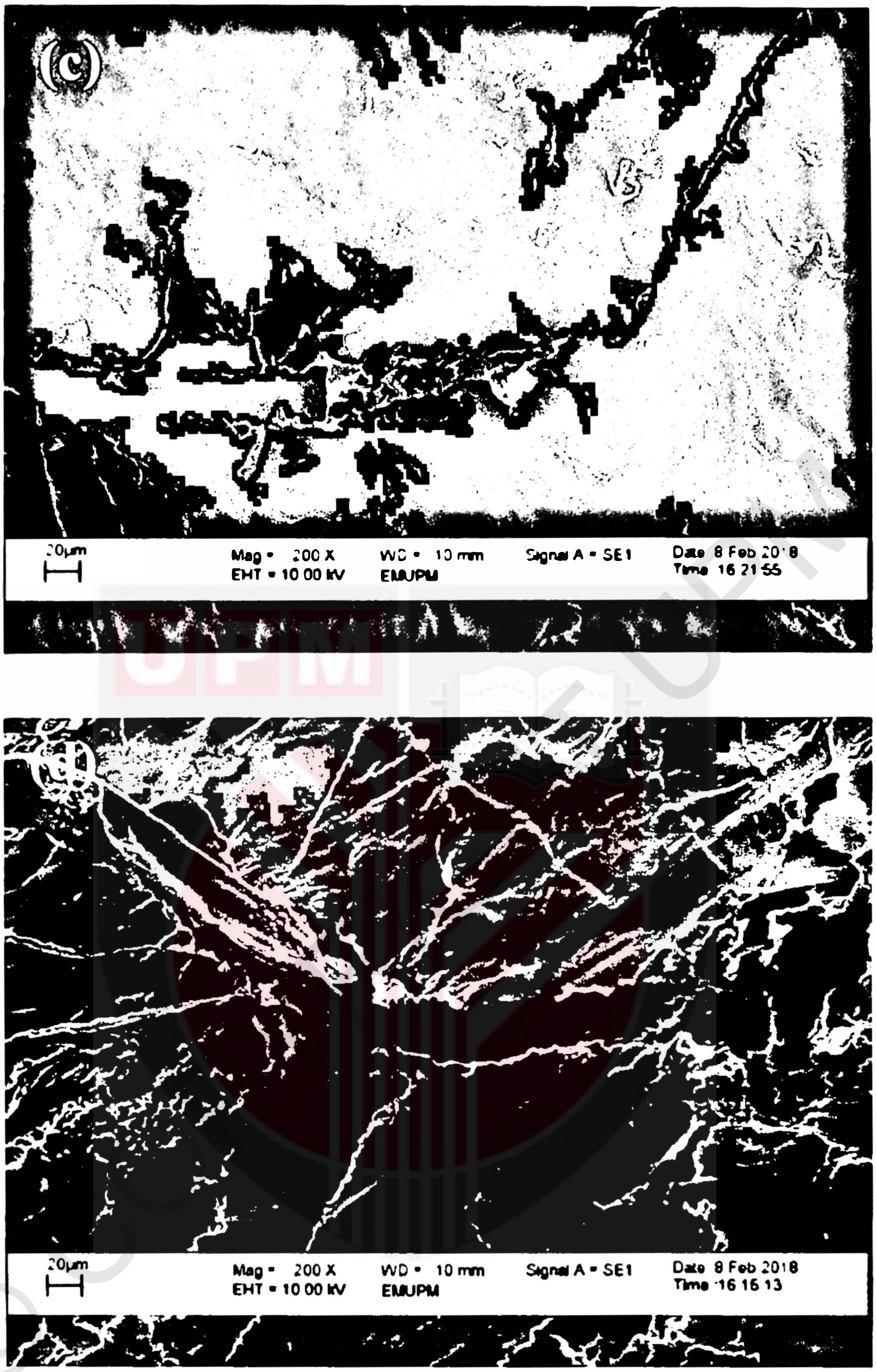


Figure 4.9 (a) Polyethylene film after flexural test (b) Good flexural properties PE-RH composite (c) Moderate flexural properties PE-RH composite (d) Poor flexural properties PE-RH composite

Finer size of filler particle resulted in fewer voids formed in the composite structure. Smaller particles are not desirable as they require more polymers for effective binding and therefore resulted in lower flexural strength. Too large particles will form more voids. However, discontinuous inter-molecular bonding between matrix and filler weaken the overall strength of the composite and therefore resulted in lower flexural strength (Nur *et al.*, 2015).

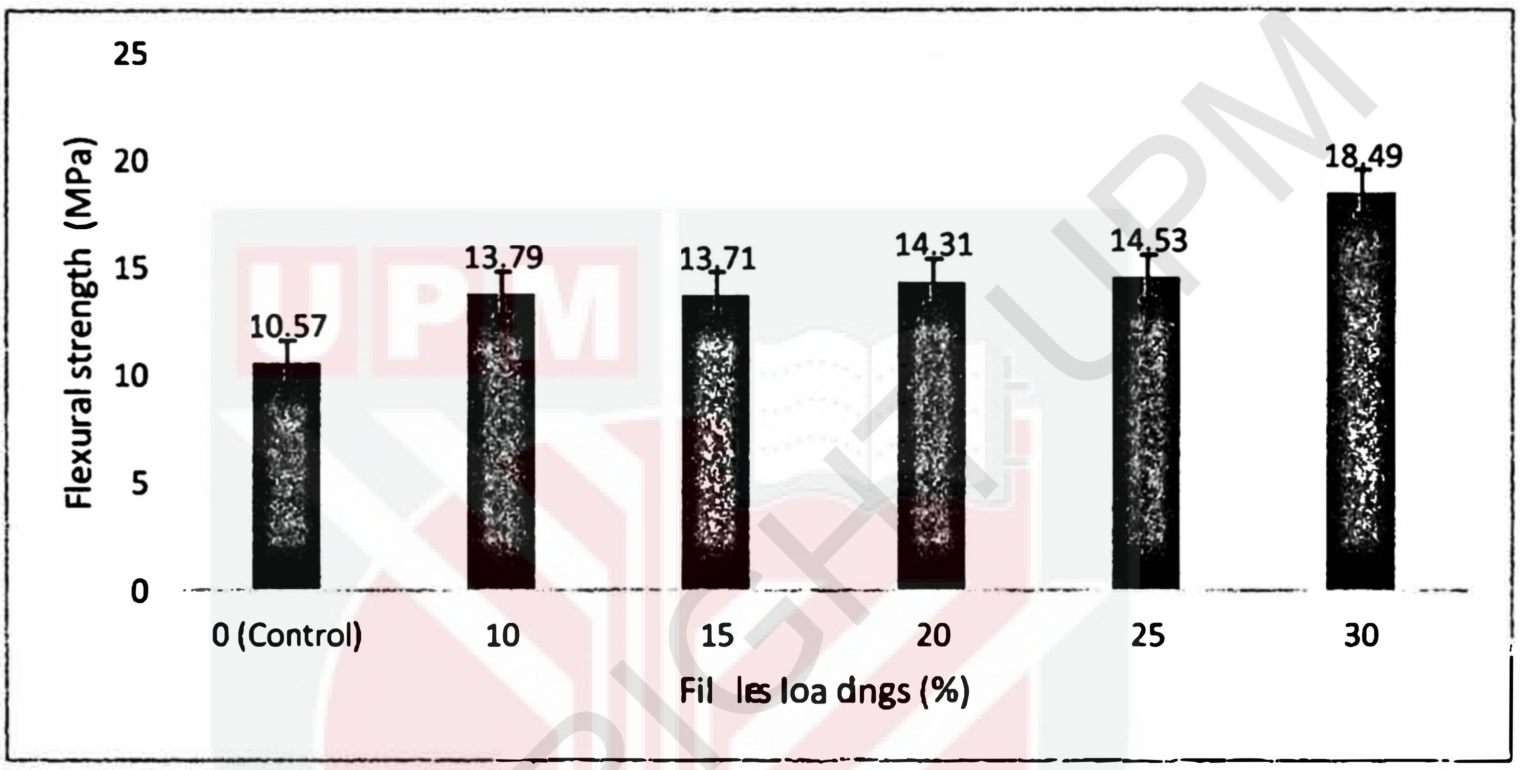
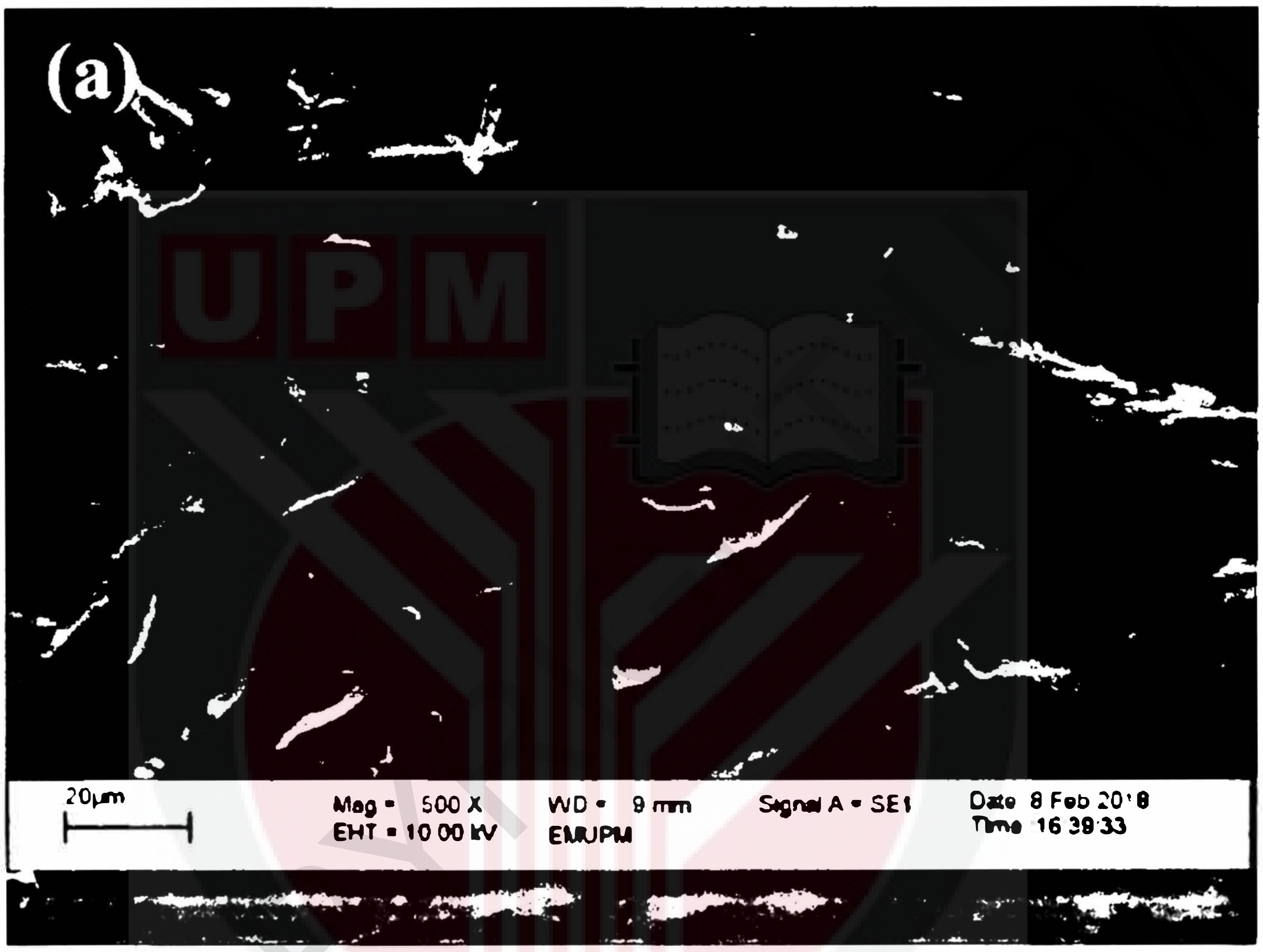
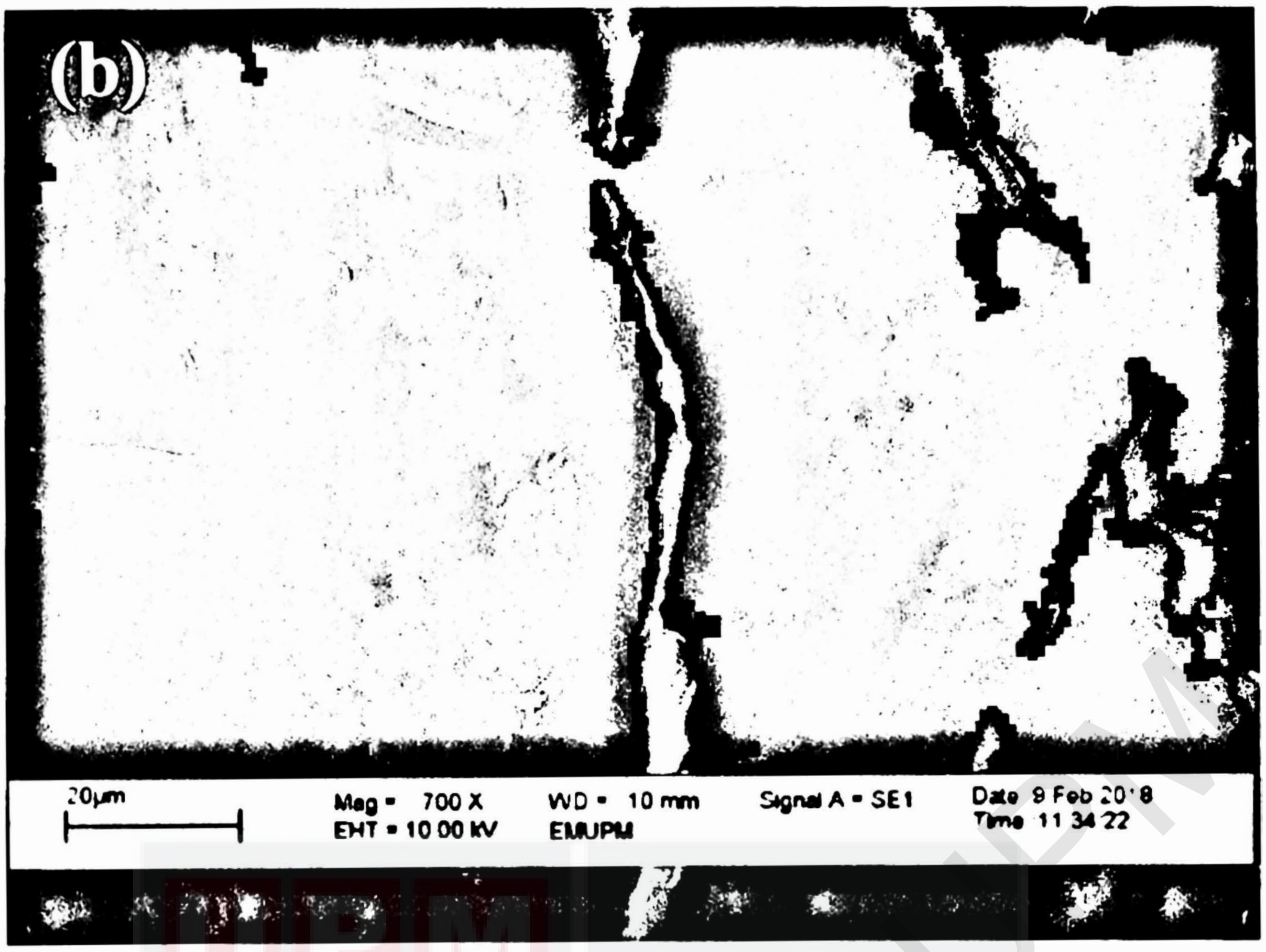


Figure 4.10 Flexural strength of PE-RHA composite

Figure 4.10 shows the effect of increasing fillers loading on the flexural strength of PE-RHA composites. Flexural strength of the composites increased steadily with the fillers loading. The highest flexural strength was recorded at 30 percent of fillers loading. Compared to PE film without fillers (control), the flexural strength was improved by approximately 75 percent. It can be concluded that increase in filler loadings will resulted in better flexural strength and vice versa.

As shown in Figure 4.11, cracks formation can be seen in PE-RHA composites which have significant influence on the flexural strength of the composite. The flexural strength is affected by the number of shear cracks formed and the width of the cracks. The width of shear cracks formed on samples with good and poor flexural strength are approximately 11µm and 23µm, respectively. Higher number and wider shear cracks formed as shown in Figure 4.11 (d) resulted in poor flexural properties and vice versa. Poor flexural strength of the composite may probably due to the gap surrounding the reinforcement which indicates the poor bonding reinforcement and matrix. Poor interfacial bonding results in a decrease in the strength of a composite as reported by Sachudhanandan *et al.* (2015).

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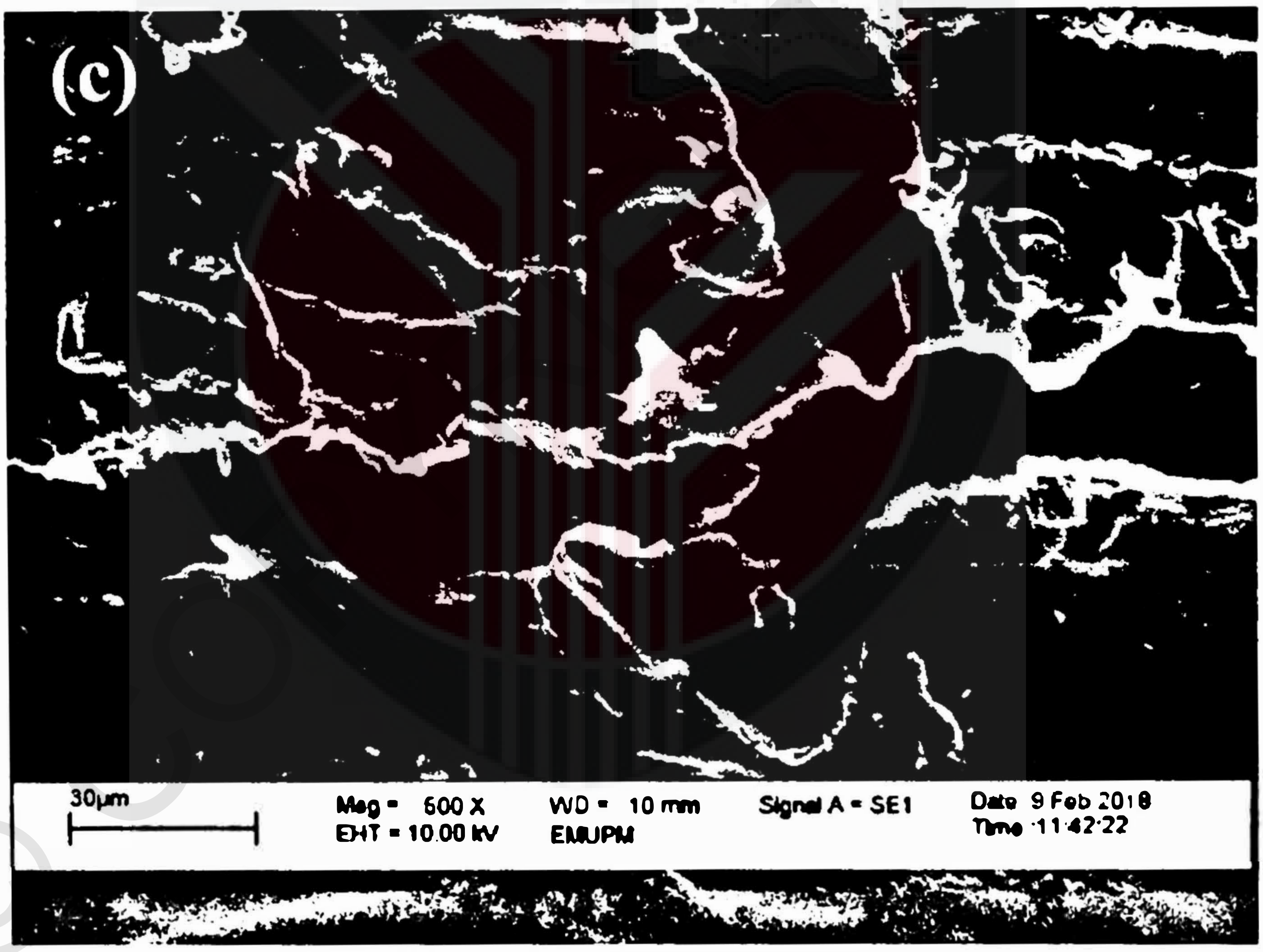
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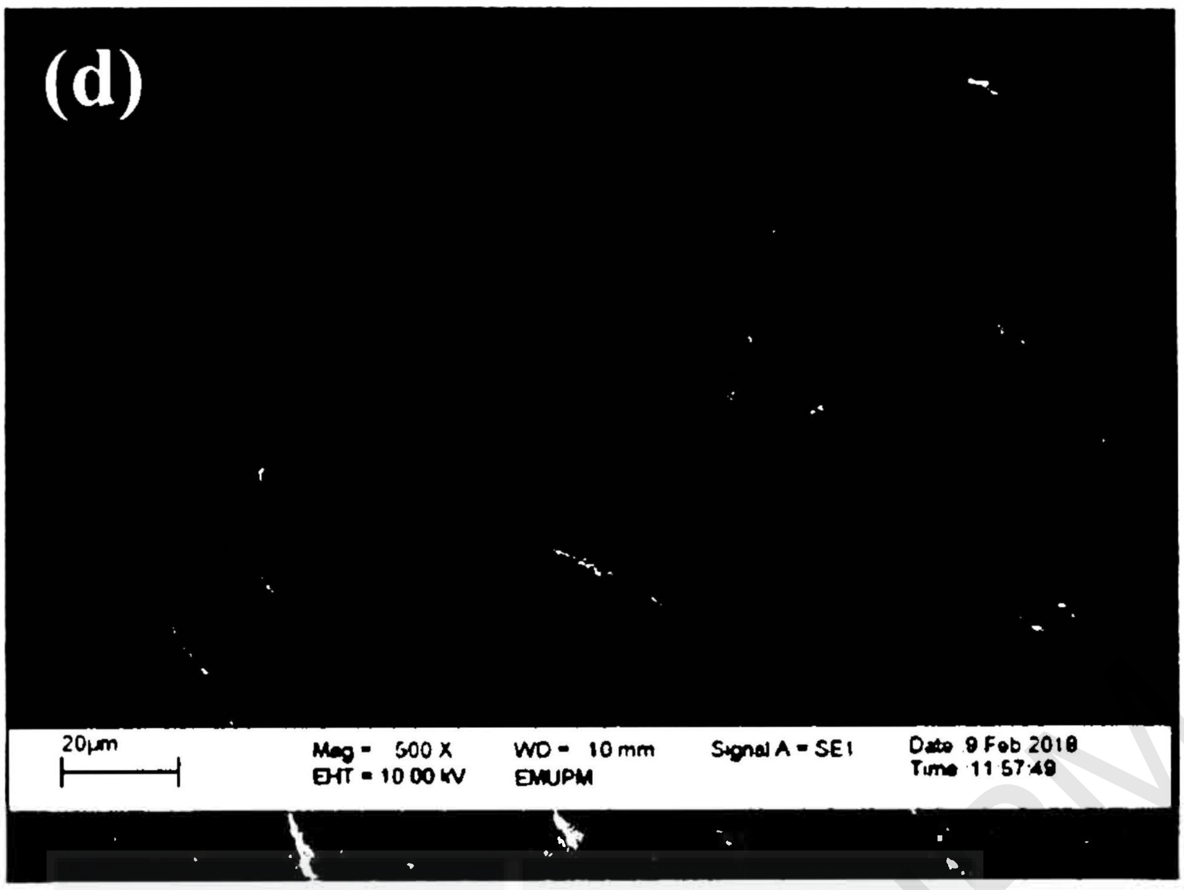

Figure 4.9 (a) Polyethylene film after flexural test (b) Good flexural properties PE-RHA composite (c) Moderate flexural properties PE-RHA composite (d) Poor flexural properties PE-RHA composite

To identify samples which possessed a combination of good oil absorption and flexural properties, a concept scoring matrix was used. A scale from 1 to 5 was implemented which indicated the sample with very poor, poor, moderate, good, and very good properties, respectively. Sample with the highest total score will be considered as the best sample which has a combination of good oil absorption and flexural properties. From Table 4.1, it shows that PE-RH composite with 20 percent of RH and PE-RHA composite with 25 percent of RHA are the best sample.

Table 4.1 Selection of the best sample

		Filler loading (%)				
		10	15	20	25	30
PE-RH	Oil absorption	1	2	4	5	3
	Flexural strength	3	4	5	2	1
	Total score	4	6	9	7	4
	Rank	**4**	**3**	**1**	**2**	**4**
PE-RHA	Oil absorption	2	3	4	5	1
	Flexural strength	2	1	3	4	5
	Total score	4	4	7	9	6
	Rank	**4**	**4**	**2**	**1**	**3**

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusion

Polyethylene-rice husk (PE-RH) and polyethylene-rice husk ash (PE-RHA) were prepared based on the experimental design and tested for oil absorption and flexural properties. In overall, PE-RH composite with 20 percent of RH and PE-RHA composite with 25 percent of RHA are the best sample which possessed a combination of good oil absorption and flexural properties. Based on the results obtained from both tests, addition of fillers (RH and RHA) showed significant improvement on the properties of the composites. Although further increase in filler loadings, above optimum value, resulted in a decrease of the properties value of the composites such as for PE-RH composites for both tests, the total oil uptake and flexural strength are still above the control sample.

In terms of microstructure characteristics of PE-RH and PE-RHA composites, higher amount of fillers resulted in less homogenous composite mixture while lower amount of fillers make the structure appears more homogeneous and the matrix system become denser. Based on the results from oil absorption test, an increased in the content of fillers would lead to some larger agglomerates due to the formation of hydrogen bonds among the abundant hydroxyl groups and adsorbed water on their surface. Higher amount of spaces between rice husk and polyethylene may allow more oil to be absorbed into the composite. It indicates that higher filler content in the composite resulted in higher percentage of oil uptake.

As for flexural properties of the composite, the surface of the composite underwent tension stress during bending process, which resulted in shear cracks. This crack was initiated from voids, which are near the fillers or space between fillers and matrix. Higher volume of shear cracks formed in PE-RH composite resulted in poor flexural properties. The formation of shear cracks weakens the yield strength of the composites. From the results, it proved that PE-RH and PE-RHA composites have great potential to be a good oil absorbent.

5.2 Recommendations

Several potential improvement were identified during the research that may improve on the PE composite. The followings are recommended for future investigation:

1) To incorporate nanocellulose filler (extracted from rice husk) into polyethylene to possibly improve its properties.
2) Other tests can be performed including bacterial resistance, waterproof, and tensile to widen its potential applications.
3) Using statistical analysis tool in designing the experiment and further obtain the optimum formulation of the composite.

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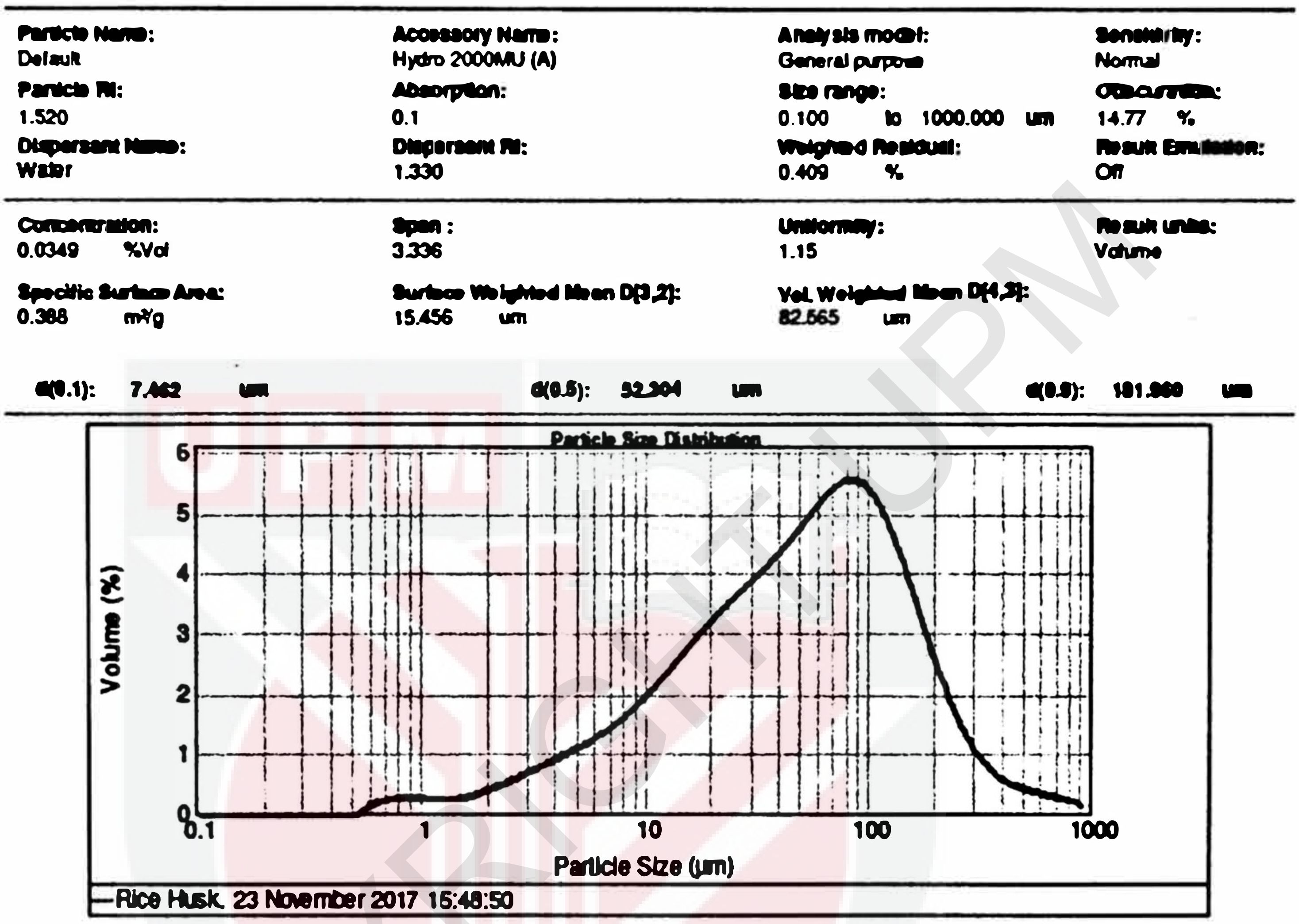
APPENDICES

Appendix A

Particle size analysis for rice husk powder

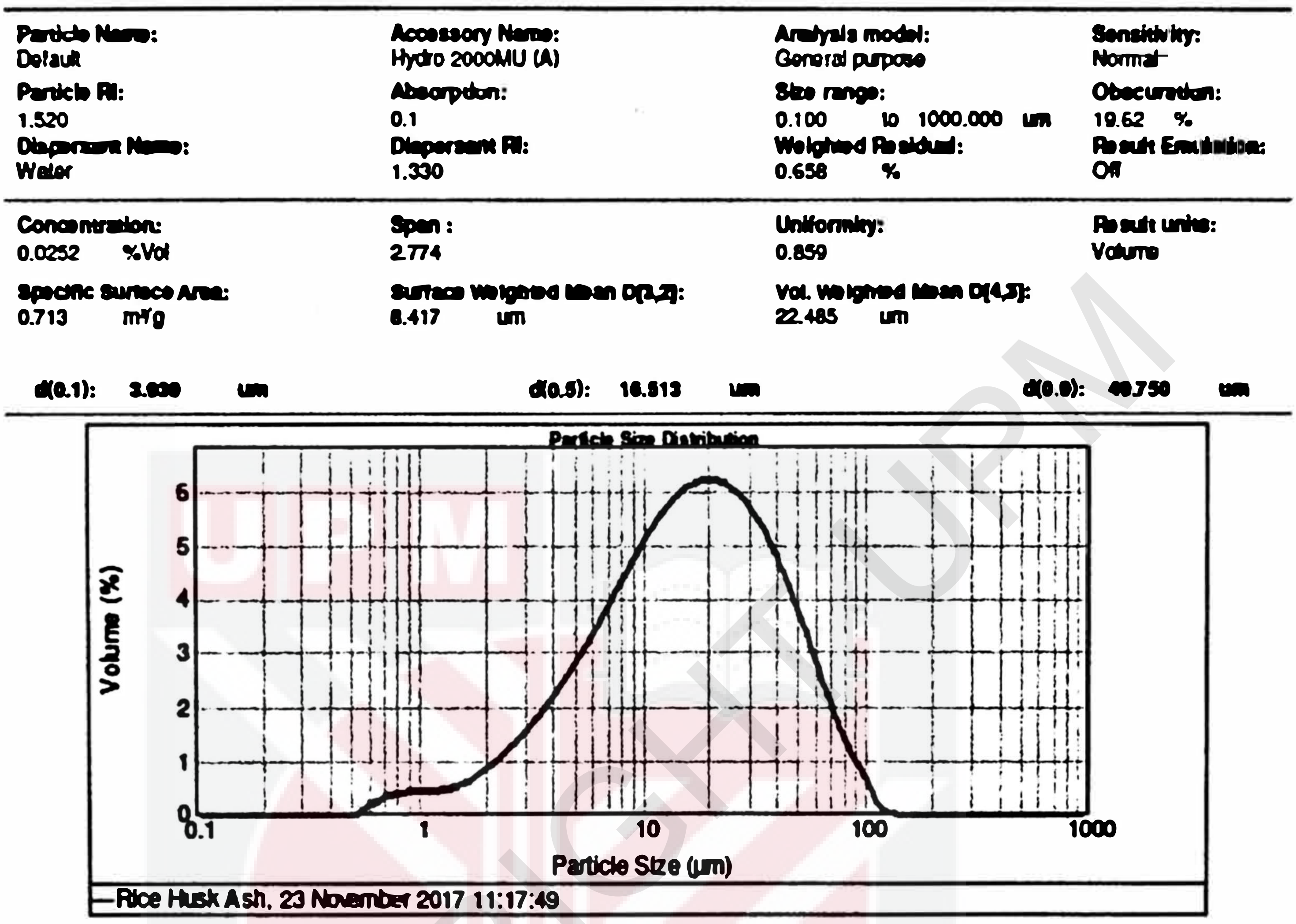


Particle Name: Default	**Accessory Name:** Hydro 2000MU (A)	**Analysis model:** General purpose	**Sensitivity:** Normal
Particle RI: 1.520	**Absorption:** 0.1	**Size range:** 0.100 to 1000.000 um	**Obscuration:** 14.77 %
Dispersant Name: Water	**Dispersant RI:** 1.330	**Weighted Residual:** 0.409 %	**Result Emulation:** Off
Concentration: 0.0349 %Vol	**Span :** 3.336	**Uniformity:** 1.15	**Result units:** Volume
Specific Surface Area: 0.388 m²/g	**Surface Weighted Mean D[3,2]:** 15.456 um	**Vol. Weighted Mean D[4,3]:** 82.565 um	

d(0.1): 7.462 um **d(0.5):** 52.304 um **d(0.9):** 181.960 um

Appendix B

Particle size analysis for rice husk ash powder



Particle Name: Default
Accessory Name: Hydro 2000MU (A)
Analysis model: General purpose
Sensitivity: Normal

Particle RI: 1.520
Absorption: 0.1
Size range: 0.100 to 1000.000 um
Obscuration: 19.62 %

Dispersant Name: Water
Dispersant RI: 1.330
Weighted Residual: 0.658 %
Result Emulation: Off

Concentration: 0.0252 %Vol
Span : 2.774
Uniformity: 0.859
Result units: Volume

Specific Surface Area: 0.713 m²/g
Surface Weighted Mean D[3,2]: 8.417 um
Vol. Weighted Mean D[4,3]: 22.485 um

d(0.1): 3.930 um
d(0.5): 16.513 um
d(0.9): 49.750 um