ผลของสารหน่วงไฟต่อสมบัติทางกายภาพของพอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว

นางสาวรพิศา จารปัญญาชีพ

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
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EFFECT OF FLAME RETARDANTS ON PHYSICAL PROPERTIES OF SAWDUST/RECYCLED HIGH DENSITY POLYETHYLENE COMPOSITES

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EFFECT OF FLAME RETARDANTS ON PHYSICAL PROPERTIES OF SAWDUST/RECYCLED HIGH DENSITY POLYETHYLENE COMPOSITES

Suranaree University of Technology has approved this thesis submitted in partial fulfillment of the requirements for a Master’s Degree.

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รพิศา จารปัญญาชีพ : ผลของสารหน่วงไฟต่อสมบัติทางกายภาพของพอลิเมอร์คอมโพสิท
ระหว่างผงขี้เลื่อยและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว
(EFFECT OF FLAME RETARDANTS ON PHYSICAL PROPERTIES OF
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งานวิจัยนี้ ผงขี้เลื่อยถูกนำมาใช้เป็นสารตัวเติมเสริมแรงสำหรับการเตรียมพอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว ผงขี้เลื่อยถูกเตรียมในรูปผงขี้เลื่อยที่ไม่ผ่านการดัดแปรและผ่านการดัดแปรอัลคาไลน์ ความเข้มข้นของสารละลายโซเดียมไฮดรอกไซด์คือ 2 5 และ 10 เปอร์เซ็นต์โดยน้ำหนักต่อปริมาตร และเวลาในการดัดแปรคือ 30 และ 60 นาที ผลของสมบัติทางความร้อน สัณฐานวิทยา การวิเคราะห์หมู่ฟังก์ชันและองค์ประกอบของสารตัวเติม พบว่าการดัดแปรอัลคาไลน์ลดปริมาณเฮมิเซลลูโลสและลิกนินจากผงขี้เลื่อยลง ภาพถ่ายจากกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดแสดงให้เห็นว่า ผงขี้เลื่อยที่ผ่านการดัดแปรอัลคาไลน์มีพื้นผิวที่สะอาดและขรุขระกว่า สภาวะที่เหมาะสมในการดัดแปรอัลคาไลน์ คือความเข้มข้นของสารละลายโซเดียมไฮดรอกไซด์ 2 เปอร์เซ็นต์โดยน้ำหนักต่อปริมาตร เป็นเวลา 30 นาที

ผลของปริมาณของผงขี้เลื่อยและการดัดแปรอัลคาไลน์ ต่อสมบัติทางกล สมบัติการต้านทานต่อไฟ สมบัติทางความร้อนและสัณฐานวิทยาของพอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้วถูกวิเคราะห์ ปริมาณผงขี้เลื่อยคือ 30 40 และ 50 เปอร์เซ็นต์โดยน้ำหนัก ความต้านทานต่อแรงดึง การยืดสูงสุด ณ จุดขาด ความต้านทานต่อแรงดัด ความต้านทานต่อแรงกระแทก ความต้านทานต่อไฟ และความเสถียรต่อร้อนลดลงเมื่อปริมาณผงขี้เลื่อยที่ไม่ผ่านการดัดแปรอัลคาไลน์เพิ่มขึ้น ในขณะที่มอดูลัสแรงดึงและมอดูลัสแรงดัดเพิ่มขึ้น การดัดแปรอัลคาไลน์ปรับปรุงสมบัติทางกล สมบัติการต้านทานต่อไฟ และสมบัติทางความร้อนของพอลิเมอร์คอมโพสิทที่ทุกปริมาณของผงขี้เลื่อย พอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยที่มีการปรับปรุงพื้นผิวชนิดอัลคาไลน์และพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว ที่มีปริมาณผงขี้เลื่อย 30 เปอร์เซ็นต์โดยน้ำหนัก แสดงสมบัติทางกล สมบัติการต้านทานต่อไฟ ความเสถียรต่อความร้อนที่สูงที่สุด ภาพถ่ายจากกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดแสดงให้เห็นถึงการปรับปรุงการยึดติดที่อินเตอร์เฟสระหว่างผงขี้เลื่อยและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้วโดยการดัดแปรอัลคาไลน์

อลูมินัมไตรไฮเดรทถูกนำมาใช้เป็นสารหน่วงไฟ ที่ปริมาณ 10 20 และ 30 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์คอมโพสิท สมบัติการต้านทานต่อไฟและความเสถียรต่อความร้อนของพอลิ-

เมอร์คอมโพสิทถูกปรับปรุงเมื่อเพิ่มปริมาณของอลูมินัมไตรไฮเดรท ในขณะที่สมบัติทางกลไม่ส่งผลกระทบมากนัก ภาพถ่ายจากกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดแสดงให้เห็นการยึดติดที่อินเทอร์เฟสที่ไม่ดีระหว่างอลูมินัมไตรไฮเดรทในพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว พอลิเมอร์คอมโพสิทที่มีอลูมินัมไตรไฮเดรทในปริมาณ 30 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์คอมโพสิท มีสมบัติการต้านทานต่อไฟและความเสถียรต่อความร้อนที่สูงที่สุด

ซิงค์บอเรทถูกนำมาใช้ร่วมกันกับอลูมินัมไตรไฮเดรท อัตราส่วนของอลูมินัมไตรไฮเดรทต่อซิงค์บอเรท คือ 2 ต่อ 1 1 ต่อ 1 และ 1 ต่อ 2 การร่วมกันของอลูมินัมไตรไฮเดรทและซิงค์บอเรทเพิ่มสมบัติทางกลของพอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยผ่านการดัดแปรอัลคาไลน์และพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว ที่มีปริมาณผงขี้เลื่อย 30 เปอร์เซ็นต์โดยน้ำหนักเล็กน้อย เมื่อเปรียบเทียบกับพอลิเมอร์คอมโพสิทที่มีการใส่อลูมินัมไตรไฮเดรท พอลิเมอร์คอมโพสิทระหว่างผงขี้เลื่อยที่ผ่านการดัดแปรอัลคาไลน์และพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว ที่มีปริมาณผงขี้เลื่อยที่ผ่านการดัดแปรอัลคาไลน์ 30 เปอร์เซ็นต์โดยน้ำหนักและมีปริมาณซิงค์บอเรท 30 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์ มีสมบัติทางกล ความเสถียรต่อความร้อนที่สูงที่สุด แต่มีสมบัติการต้านทานต่อไฟต่ำที่สุด การร่วมกันของอลูมินัมไตรไฮเดรทและซิงค์บอเรท ที่อัตราส่วน 2 ต่อ 1 และ 1 ต่อ 1 แสดงผลการเสริมกันในการปรับปรุงสมบัติการต้านทานต่อไฟพอลิเมอร์คอมโพสิท พอลิเมอร์คอมโพสิทที่มีปริมาณอลูมินัมไตรไฮเดรท 20 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์คอมโพสิท และมีปริมาณซิงค์บอเรท 10 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์คอมโพสิท มีสมบัติการต้านทานต่อไฟที่ดีที่สุด

นอกจากนี้ มาเลอิกแอนไฮไดรกราฟท์พอลิเอทิลีนถูกนำมาใช้เป็นสารช่วยให้เข้ากันที่ปริมาณ 1 3 และ 5 ส่วนในหนึ่งร้อยส่วนของพอลิเมอร์คอมโพสิท เมื่อเพิ่มปริมาณมาเลอิกแอนไฮไดรกราฟท์พอลิเอทิลีน สมบัติทางกลของพอลิเมอร์คอมโพสิทเพิ่มขึ้นอย่างมีนัยสำคัญ ในขณะที่สมบัติการต้านทานต่อไฟ และความเสถียรต่อความร้อนเพิ่มขึ้นเล็กน้อย ภาพถ่ายจากกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดแสดงให้เห็นไม่เพียงแต่การปรับปรุงการยึดติดที่อินเตอร์เฟสระหว่างสารตัวเติมและพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว แต่ยังแสดงการกระจายตัวที่ดีของสารหน่วงไฟและผงขี้เลื่อยในพอลิเอทิลีนชนิดความหนาแน่นสูงที่ผ่านการใช้งานแล้ว

สาขาวิชา วิศวกรรมพอลิเมอร์ ลายมือชื่อนักศึกษา____________________

ปีการศึกษา 2556 ลายมือชื่ออาจารย์ที่ปรึกษา______________

RAPISA JARAPANYACHEEP : EFFECT OF FLAME RETARDANTS ON PHYSICAL PROPERTIES OF SAWDUST/RECYCLED HIGH DENSITY POLYETHYLENE COMPOSITES. THESIS ADVISOR : ASST. PROF. KASAMA JARUKUMJORN, Ph.D., 131 PP.

RECYCLED HIGH DENSITY POLYETHYLENE/SAWDUST/COMPOSITE/ ALUMINUM TRIHYDRATE/ZINC BORATE/ALKALI TREATMENT/MALEIC ANHYDRIDE GRAFTED POLYETHYLENE

In this study, sawdust was used as reinforcing filler for preparing sawdust/recycled high density polyethylene (rHDPE) composites. Sawdust was prepared as untreated sawdust (UT) and alkali treated sawdust (AT). Sodium hydroxide (NaOH) concentrations were 2, 5 and 10% w/v and the treatment times were 30 and 60 min. Thermal properties, morphological properties, functional group analysis and filler composition determination results exhibited that alkali treatment removed hemicellulose and lignin from sawdust. SEM micrographs revealed cleaner and rougher surface of alkali treated sawdust. The optimum alkali treatment condition was 2% w/v NaOH for 30 min

Effects of sawdust content and alkali treatment on mechanical, flame retarding, thermal and morphological properties of sawdust/rHDPE composites were investigated. Sawdust contents were 30, 40 and 50 wt%. Tensile strength, elongation at break, flexural strength, impact strength, flame retardancy and thermal stability of the composites were decreased with increasing untreated sawdust content while tensile modulus and flexural modulus were increased. Alkali treatment enhanced mechanical, flame retarding and thermal properties of the composites at all sawdust contents. rHDPE/30AT composite showed the highest mechanical properties, flame retardancy and thermal stability. SEM micrographs revealed the improvement of interfacial adhesion between sawdust and rHDPE matrix by alkali treatment.

Aluminum trihydrate (ATH) was used as a flame retardant at contents of 10, 20 and 30 phr. Flame retardancy and thermal stability of the composites were improved with increasing ATH content while mechanical properties were not much affected. SEM micrographs showed the poor interfacial adhesion between ATH and rHDPE matrix. The composite containing 30ATH showed the highest flame retardancy and thermal stability.

Zinc borate (ZB) was used in the combination with ATH. ATH/ZB ratios were 2:1, 1:1 and 1:2. The combination of ATH and ZB slightly increased mechanical properties of rHDPE/30AT composite comparing to the composite containing ATH. The composite containing 30ZB had the highest mechanical properties and thermal stability but the lowest flame retardancy. The combination of ATH and ZB at ATH:ZB ratios of 2:1 and 1:1 exhibited the synergistic effect in enhancing flame retardancy of the composites. The composite containing ATH/10ZB at the ratio of 2:1 showed the highest flame retardancy.

In addition, maleic anhydride grafted polyethylene (MAPE) was used as a compatibilizer and its contents were 1, 3 and 5 phr. With increasing MAPE content, mechanical properties of the composites were significantly improved while flame retardancy and thermal stability were slightly increased. SEM micrographs revealed not only the enhancement of interfacial adhesion between fillers and rHDPE matrix but also the good distribution of flame retardants and sawdust in rHDPE matrix.

School of Polymer Engineering Student’s Signature__________________

Academic Year 2013 Advisor’s Signature__________________

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Rapisa Jarapanyacheep

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SYMBOLS AND ABBREVIATIONS

AT = Alkali treated sawdust

ATH = Aluminum trihydrate

DTA = Differential thermal analysis

FTIR = Fourier transform infrared spectrometer

MAPE = Maleic anhydride grafted polyethylene

NaOH = Sodium hydroxide

rHDPE = Recycled high density polyethylene

SEM = Scanning electron microscope

TGA = Thermogravimetric analysis

UT = Untreated sawdust

ZB = Zinc borate

CHAPTER I

INTRODUCTION

1.1 General introduction

Lignocellulosic fillers/polymer composites have attracted much attention from a number of researchers and manufactures. In comparison with other fillers such as traditional glass fiber and inorganic mineral fillers, lignocellulosic fillers have received much interest for being used as reinforcing materials because of their biodegradability, renewable resources, low cost and light weight. Sawdust, by-product obtaining from the wood industry, is one the most used lignocellulosic fillers for the manufacture of lignocellulosic fillers/polymer composites. The use of sawdust in producing lignocellulosic fillers/polymer composites benefits to the environment and also adds value to sawdust.

Nowadays, plastics are widely used as packaging, furniture, electronic parts, and housewares. This has caused an increasing concern regarding the environment problem of plastic waste disposal. Plastic recycling is an alternative solution to handling plastic waste disposals. High density polyethylene (HDPE) is one of the most commonly used thermoplastic and constitutes large portion of the post-consumer household wastes. Moreover, HDPE is easy to separate from the post-consumer household wastes and offers many advantages such as high toughness and good chemical resistance.

Sawdust/recycled high density polyethylene (rHDPE) composites have gained increasing interest over decades because they offer interesting combinations of cost and properties (Khedari et al., 2004; Gosselin et al., 2006; Khalil et al., 2006; Yang et al., 2007). Najafi et al. (2006) and Adhikary et al. (2008) reported that the composites made from rHDPE and sawdust showed similar mechanical properties but were much cheaper than the composites made from virgin HDPE.

The main use of sawdust/rHDPE composites is in building and construction to replace impregnated wood in outdoor applications such as decking, railings, window and door frames. Sawdust/rHDPE composites are also use as automotive interior panels and interior materials such as flooring and furniture. However, the major limitation of sawdust/rHDPE composites is poor interfacial adhesion between hydrophobic PE and hydrophilic sawdust resulting in the composites with poor mechanical properties (Wang et al., 2003). The compatibility of the composites can be improved by various methods such as filler surface treatment, matrix modification and addition of compatibilizer. Alkali treatment is one of the most used chemical surface modifications. The treatment removes lignin, hemicellulose, pectin and impurity covering the surface of the filler. Therefore, the effective filler surface area available for interaction with the matrix would increase (Morsyleide et al., 2009). This results in the improvement of the mechanical properties of the composites (Lopattananon, et al., 2006; Suardana et al., 2011; Elzubair et al., 2012).

Another drawback of sawdust/rHDPE composites is their high flammability because both rHDPE and sawdust are flammable (Stark et al., 2010). Generally, the method to improve fire resistance of flammable materials is to use flame retardants which are directly incorporated into the materials. Flame retardants are often preferred to provide low flammability to polymeric materials because the uses of flame retardants compromise between cost and properties (Bourbigot et al., 2010).

Aluminum trihydrate (ATH) is a wildly used flame retardant for polymer. The particular benefits of ATH include low health hazard, good purity, cost effectiveness, non-corrosion and smoke suppressant. However, high loading level of ATH is required to meet flame retardant properties. This could be deteriorated mechanical properties of flame retardant materials (Chen et al., 2005). Many investigations found that the incorporation of ATH enhanced flame resistance of the polymer composites (Schartel et al., 2006; Ramazani et al; 2008; Formicola et al., 2009). The decomposition mechanism of ATH can be explained as follows. ATH provides flame retarding effects by producing char insulating layer and releasing water at about 200°C (Klyosov, 2007).

$$2Al(OH)_3 \xrightarrow{200°C} Al_2O_3 + 3H_2O \qquad (1.1)$$

Zinc borate (ZB) is one of the flame retardants to use in combination with halogen free flame retardants in polymers resulting in the polymers with enhanced flame retardancy and mechanical properties. (Ning and Guo 2000; Carpentier et al., 2007; Formicola et al., 2009). The decomposition of ZB is shown as follows. At about 320°C, it decomposes endothermically and releases water (Samyn et al., 2007).

$$2ZnO.3B_2O_3.3H_2O \xrightarrow{320°C} 2ZnO.3B_2O_3 + 3H_2O \qquad (1.2)$$

The effectiveness of flame retardants can be enhanced by synergistic effect. The combination of flame retardants leads to the improvement of flame retarding properties (Garcia et al., 2009; Kurt and Mengeloglu, 2009; Bai, Guo and Li, 2014). Ning and Guo (2000) found that the combination of ATH and ZB can result in synergistic benefits in enhancing flame retardancy of polyvinyl chloride (PVC) composites. The interaction of ATH with ZB can lead to a hard residue with porous structure which acts as a thermal insulator or barrier protecting the polymer. High quality of chars can separate the polymer from heat and oxygen. Kurt and Mengeloglu (2011) reported that the use of ZB as a synergist improved mechanical and flame retardancy of wood flour/polypropylene (PP) composites filled with ammonium polyphosphate (APP). Jeencham et al. (2010) found that the combination of APP and ZB showed synergistic effect in enhancing flame retardantcy of the sisal/PP composites without significant change in mechanical properties of the composites.

In addition, in order to improve the distribution of flame retardants in rHDPE matrix and the compatibility between flame retardants and rHDPE matrix, maleic anhydride grafted polyethylene (MAPE) is incorporated. The anhydride group of MAPE forms the chemical bonds with the hydroxyl groups of flame retardants. Long hydrocarbon tails of MAPE are able to anchor to HDPE matrix through physical entanglements and van der waals interactions. Therefore, the interfacial adhesion between flame retardants and HDPE can be improved by adding MAPE compatibilizer (Hausmann et al., 1997; Hippi et al., 2003).

1.2 Research objectives

The main objectives of this research are as below:

(i) To study effect of sawdust content on mechanical, thermal, flame retarding and morphological properties of sawdust/rHDPE composites.

(ii) To study effect of alkali treatment on mechanical, thermal, flame retarding and morphological properties of sawdust/rHDPE composites.

(iii) To study effect of ATH as a flame retardant on mechanical, thermal, flame retarding and morphological properties of sawdust/rHDPE composites.

(iv) To study effect of the combination of ATH and ZB on mechanical, thermal, flame retarding and morphological properties of sawdust/rHDPE composites.

(v) To study effect of MAPE as a compatibilizer on mechanical, thermal, flame retarding and morphological properties of sawdust/rHDPE composites containing the combination of ATH and ZB.

1.3 Scope and limitation of the study

In this study, sawdust was prepared as untreated sawdust (UT) and alkali treated sawdust (AT). Sodium hydroxide (NaOH) concentrations were varied as 2, 5 and 10% w/v at the treatment time of 30 and 60 min at room temperature. Thermal properties, morphological properties, functional group analysis and filler composition determination were investigated.

Sawdust/rHDPE composites at sawdust contents of 30, 40 and 50 wt% were prepared using a twin screw extruder. Test specimens were molded using a compression molding machine. Alkali treatment was used to improve the compatibility between sawdust and rHDPE. Effects of sawdust content and alkali treatment on mechanical, flame retarding, thermal and morphological properties of sawdust/rHDPE composites were investigated. According to mechanical, thermal and flame retarding properties, the composite giving the optimum properties was chosen to study the effect of flame retardant on the properties of the composites.

ATH was used as a flame retardant and its contents were 10, 20 and 30 phr. Mechanical, thermal, flame retarding and morphological properties of the composites were examined. Based on the mechanical, thermal, flame retarding properties of the composites, the composite giving the optimum properties was selected to study the effect of the combination of flame retardants on the properties of sawdust/rHDPE composites.

The effect of the combination of ATH and ZB on the properties of the composites was also investigated. The ratios of ATH:ZB were 1:1, 1:2 and 2:1. The composite having optimum mechanical, flammability and thermal properties was chosen to study the effect of compatibilizer on the properties of sawdust/rHDPE composites containing flame retardants. Moreover, maleic anhydride grafted polyethylene (MAPE) was used as a compatibilizer. MAPE contents were 1, 3 and 5 phr.

CHAPTER II

LITERATURE REVIEW

2.1 Characteristics of wood

Wood is classified as a lignocellulosic material. It is composed of three main components; cellulose, hemicellulose and lignin as shown in Table 2.1. Cellulose is the major component of wood providing the wood strength and structural stability. It is derived from glucose units and linked by β-1,4 glycosidic bonds (Hendriks and Zeeman, 2009) as illustrated in Figure 2.1 (a). Hemicellulose is a complex carbohydrate structure that consists of pentose, hexose and sugar acid. Hemicellulose has a lower molecular weight than cellulose and branches with short chains which are easy to hydrolysis (Hendriks and Zeeman, 2009) as display in Figure 2.1 (b). Lignin is a binding agent that holds cellulose fibers together. It consisted of three different phenylpropane units; p-coumaryl, coniferyl and sinapyl alcohol (Hendriks and Zeeman, 2009) as shown in Figure 2.1 (c). The main propose of lignin is to give the plant structural support, impermeability and resistance against microbial attack.

Table 2.1 Chemical compositions of wood (Kozlowski and Przybylak, 2008).

Wood type	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Hardwood	38-49	19-26	23-30
Softwood	40-45	7-14	25-35

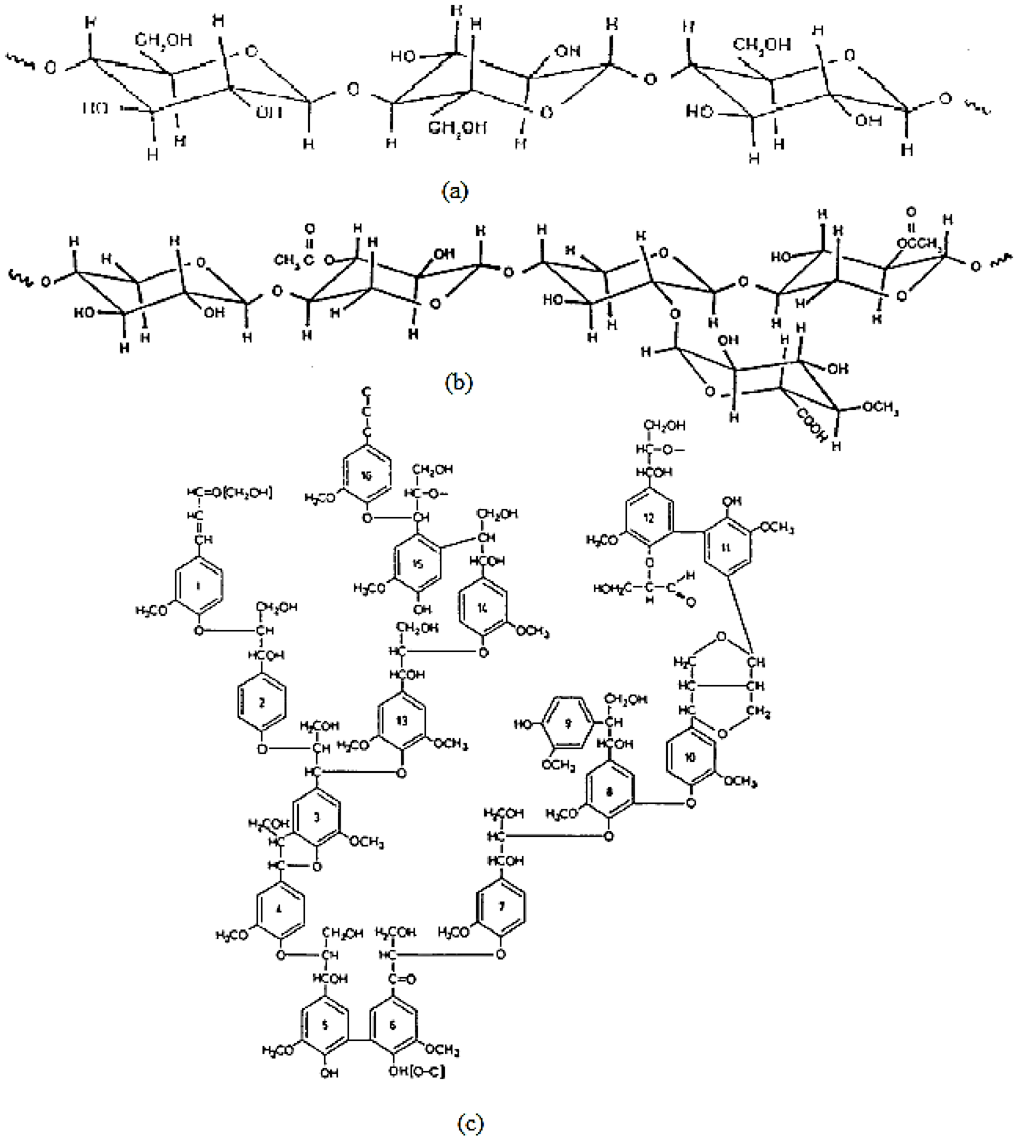


Figure 2.1 Chemical structure of (a) cellulose, (b) hemicellulose and (c) lignin (Pandey, 1999).

2.2 Wood/polymer composites

Wood/polymer composites (WPCs) have gained considerable attention from many researchers. WPCs can be made from virgin or recycled polymers and wood filler obtaining from wood industry. WPCs containing recycled plastics and wood filler offer interesting combinations of cost and properties. Khalil et al. (2006)

examined the effects of sawdust content and particle size on mechanical properties of sawdust/recycled polypropylene (rPP) composites. The composites were prepared at five different filler loadings (0, 10, 20, 30, 40, and 50 wt%) with three particles sizes (100, 212, and 300 mm). The results showed that composites with a smaller particle size (100 mm) had remarkably higher properties compared to others (212 and 300 mm). Mechanical properties of the composites continuously increased with increasing sawdust content but the value dramatically decreased with adding above 30% filler loading.

Najafi et al. (2006) investigated mechanical properties of the composites containing recycled high density polyethylene (rHDPE) and rPP. The composites were prepared at sawdust loading of 50 wt%. Tensile properties, flexural properties and impact strength were examined. The composites made from rPP exhibited higher stiffness and strength than those of rHDPE composites but their impact strength was lower. Moreover, mechanical properties of composites containing recycled polymers were similar and comparable to those of composites made from virgin polymer.

Adhikary et al. (2008) studied the effect of wood flour content on mechanical properties of wood flour/plastic composites based on rHDPE and virgin high-density polyethylene (vHDPE). Wood flour contents were 30, 40 and 50 wt%. Tensile and flexural properties of the composites based on rHDPE were equivalent to those based on vHDPE. In addition, with increasing wood flour content, tensile strength, elongation at break and flexural strength of the composites decreased while tensile modulus and flexural modulus increased.

Nourbakhsh and Ashori (2009) studied the effects of wood fiber content (55, 70, and 85 wt%) and compatibilizing agent content (0, 2, and 4 wt%) on flexural properties of wood fiber/rHDPE composites. Flexural properties of the composites increased with increasing wood fiber content. Maleic anhydride grafted polyethylene (MAPE) improved the compatibility between wood fiber and rHDPE leading to a significant improvement of flexural properties.

2.3 Compatibility improvement between lignocellulosic fillers and polymer matrix

The main drawback of the lignocellulosic fillers/polymer matrix is the incompatibility between hydrophilic lignocellulosic fillers and hydrophobic polymer matrix leading to poor mechanical properties of the composites. The interfacial adhesion between lignocellulosic fillers and polymer can be improved by filler surface treatment, matrix modification and adding compatibilizer.

2.3.1 Filler surface treatment

2.3.1.1 Physical treatment of lignocellulosic fillers

The physical modifications such as heat treatment, plasma treatment and corona treatment have been applied to enhance the compatibility between lignocellulosic fillers and polymer matrix.

2.3.1.1.1 Heat treatment

Heat treatment is an environmental friendly method without impregnating with additional chemicals. Heat treatment of lignocellulosic fillers is an effective method to improve mechanical properties, dimensional stability, hygroscopic properties and resistance to fungal decay for above-ground applications (Marcos et al., 2009).

Hakkou et al. (2005) studied the change of wettability of sawdust during thermal treatment. Heat treatment of sawdust were performed in an oven in a range of temperature between 130 and 160°C for 8 h. Fourier transform infrared spectroscopy (FTIR) results showed that thermal treatment led to an important decrease in the carbonyl band at 1730 cm^{-1} indicating hemicelluloses degradation. The hydrophilic wood became rather hydrophobic after thermal treatment.

Cao et al. (2007) examined the effect of heat treat temperature on tensile properties of kenaf fibers. Kenaf fibers were treated at 130, 140, 160, 190 and 220°C for 10 h. The maximum tensile strength of the fiber was obtained at heating temperature of 140°C. Tensile strength of heat treated fibers was decreased when heating temperature was over 140°C. With increasing heat treatment temperature, the appearance color of the fibers gradually became brown and black by the observation of naked eye and the fibers became easily brittle and broken.

Kaewkuk et al. (2010) studied the effect of heat treatment temperature on tensile and morphological properties of sisal fibers. Sisal fibers were heated at 150, 170 and 200°C for 30 min under an atmospheric pressure and a presence of air. Tensile strength of heat treated sisal fiber was higher than untreated sisal fiber. Tensile properties of treated fiber at 170°C were higher than those of treated fiber at 150 and 200°C. SEM micrographs revealed that the surface of heat treated fibers were cleaner and smoother than that of untreated fiber due to the removal of impurities and some hemicellulose.

Ochi (2012) investigated the effect of heat treatment on mechanical properties of bamboo fibers. Bamboo fibers were heated in air at 140, 160, 180 and 200°C for 15, 30, 60 and 120 min. Tensile strength of heat treated bamboo fibers at 140°C was unchanged even with longer heating times. Tensile strength of heat-treated bamboo fibers at 160°C gradually decreased with increasing heating time. Tensile strength of bamboo fibers rapidly decreased at heating temperatures of 180 and 200°C when heating time was decreased. At the same heating time, heat treated bamboo fibers at 140°C showed the highest tensile strength.

2.3.1.1.2 Plasma treatment

Plasma treatment is used to enhance the wetting characteristics of a variety of materials (plastic, metal, glass and wood). Yuan et al. (2004) studied the effect of plasma treatment on surface characteristics of wood fibers. Wood fiber was modified by Argon (Ar) and air-plasma treatments with discharge power of 60 W and treatment time of 30 s. X-ray photoelectron spectroscopy (XPS) was used to investigate surface characteristics of wood fibers. XPS results showed that O/C ratios of wood fibers increased after both Ar-plasma and air-plasma treatments indicating that oxidation occurred during the plasma treatment. Air-plasma treated wood fiber surface contained the highest oxygen content and O/C ratio because oxygen in air was excited by plasma into a highly energetic and reactive atomic and diatomic molecular state. Ar-plasma treated samples also showed an increase in both oxygen content and O/C ratio due to the fact that the free radicals generated in Ar-plasma treatment on the surface of fibers reacted readily with the atmospheric oxygen and moisture when samples were left in laboratory conditions. This was suggested that Ar and air-plasma treatment led to the generation of identical surface functionalities on pure cellulose surfaces. Since cellulose and hemicellulose were more reactive to plasma, they were more easily etched away by plasma treatment. On the other hand, non-polar lignin was left on the fiber surface.

Bozaci et al. (2009) examined the effect of plasma treatment parameters on surface characteristics and mechanical properties of jute fiber. Jute fiber was treated with air-plasma at discharge power of 60, 90 and 120 W for 1, 3 and 6 min. SEM micrographs revealed that surface roughness of jute fiber was increased with increasing plasma power and treatment time. Plasma treatment caused damages on the fiber surface resulting in decreased fiber strength because micropits and voids were generated. With increasing power and time input, more damages on the fiber surfaces were observed. This led to a decrease in fiber strength.

Anwer and Bhuiyan (2012) studied the effect of Ar-plasma treatment on morphological and electrical properties of jute fibers. The discharge powers were adjusted at 50 and 100 W with the duration of exposure time of 10 and 20 min. SEM micrographs revealed that the surface of raw jute fiber was smooth. Surface roughness of the treated fiber was increased with increasing discharge power and exposure time because of physical sputtering and chemical etching effect. Electrical resistivity of the fibers increased with increasing discharge power and treatment time because the fibers were dry and hydrophobic.

2.3.1.1.3 Corona treatment

Corona treatment of lignocellulosic filler leads to an increase in the polarity of filler, mainly due to increasing the content of carboxyl and hydroxyl groups. Gassan and Gutowski (2000) studied the effect of corona treatment on the fiber characteristic of jute fibers. Power output was ranged from 40 to 100 W and treatment speed was varied from 10 to 88 cm/min. With increasing power output and treatment speed, the tensile strength of the fiber was decreased due to the oxidation of the fiber surface.

Ragoubi et al. (2012) studied the effect of corona treatment on surface characteristics of miscanthus fiber. The fiber was modified by corona treatment with power of 15 kV at 50 Hz. The treatment times were 15, 30 and 45 min. XPS results showed that O/C ratio of treated fiber was enhanced when treatment time was increased. This was attributed to the surface oxidation generated by the corona treatment. SEM micrographs revealed the fiber surface was progressively modified when the treatment time was increased. After 15 min of treatment time, some little cracks appeared on the fiber surface. The fibrils were pulled off from the fiber when treatment time was 30 min. Some cavities were observed after 45 min of treatment. This was caused by the bombardment of the surface by the oxygen plasma species. The corona discharge produced many activated sites all along the polymeric chains of the fiber which could react with oxygen to give etching effect.

2.3.1.2 Chemical treatment of lignocellulosic fillers

The chemical modifications e.g. alkali treatment, silanization and acetylation were used to decrease the hydrophilic nature of lignocellulosic fillers and enhance the compatibility between lignocellulosic fillers and polymer matrix.

2.3.1.2.1 Alkali treatment

Alkali treatment is one of the most common treatment of the lignocellulosic fillers. This process is a purifying treatment that removes waxes, pectin, hemicellulose and lignin from the filler surface. Sharifah et al. (2004) examined the effect of alkali treatment on fiber density and surface topography of hemp fiber. Hemp fibers were treated with 6 wt% of sodium hydroxide (NaOH). After alkali treatment, fiber densities were increased due to cell wall densification. SEM micrographs revealed that the fiber surface of alkali treated fibers was rougher than that of untreated fibers due to the removal of wax, oil, surface impurities, lignin and hemicellulose from the fiber surface.

Lopattananon et al. (2006) treated pineapple leaf fibers with 1, 3, 5 and 7% w/v NaOH solution. FTIR results showed that after alkalization the peak of lignin (1436 cm^{-1}) was reduced and hemicellulose (1254 cm^{-1}) structure was absent due to complete decomposition of hemicellulose and partial leaching out of lignin by NaOH. This indicated that the ester type in hemicellulose was more easily removed by alkali solution. SEM micrograph of the treated fiber showed the separation of the microfibrillar structure of the fiber because lignin and hemicellulose were removed. The removal of lignin and hemicellulose appeared to increase with increasing concentration of alkali solution. Consequently, the effective fiber surface area which was available for bonding with the matrix would be increased.

Mwaikambo (2009) studied the effect of alkali treatment on tensile properties of sisal fiber bundles. Sisal fibers were soaked in NaOH solution with concentrations of 0.03, 0.08, 0.16, 0.24 and 0.32 wt% for 48 h. Tensile strength of alkali treated fiber was increased with increasing NaOH concentration due to an increase in cellulose content. The maximum tensile strength was obtained at 0.24 wt% NaOH. However, at 0.32 wt% NaOH solution, tensile strength of the fiber was decreased due to cell wall degradation. From SEM micrographs, the untreated jute fibers showed smooth surface while alkali treated jute fibers revealed rough and void regions between fiber cells.

Edeerozey et al. (2007) investigated morphology and tensile properties of NaOH treated kenaf fibers. The fibers were immersed in NaOH solution with different concentrations (3, 6 and 9 wt% NaOH) for 3 h at room temperature. SEM micrographs showed that wax, pectin, hemicellulose and lignin were removed from the fiber surface. Tensile properties of kenaf fiber were significantly improved by the alkalization treatment when compared to untreated kenaf fiber. The highest tensile strength was obtained at 6 wt% NaOH solution. Tensile strength significantly decreased when NaOH concentration was increased to 9 wt%. This indicated that 9 wt% of NaOH was too strong and damaged the fibers resulting in lower tensile strength.

Suardana et al. (2010) studied the effect of alkali treatment on physical properties of hemp fibers. NaOH concentrations were varies as 2%, 4% and 6 wt%. Alkali treatment increased weight loss but decreased density and tensile strength of the fibers. When the NaOH concentration was increased density and tensile strength of the fibers were decreased. SEM micrographs revealed that the surface of the untreated hemp fiber was covered with a layer of substances including pectin, lignin, and other impurities. After NaOH treatment, almost of the lignin and pectin were removed resulting in a rougher surface with some fibrils. FTIR spectra of untreated fiber showed that C=O (carbonyl) peak at 1732 cm^{-1} was reduced when the alkali treatment concentration was increased and totally disappeared at 6 wt% NaOH because of the removal of hemicelluloses from the fiber surfaces. Moreover, the peak at 1240 cm^{-1} (C-O stretching of the acetyl groups of lignin) was reduced when the

NaOH concentration was increased. This suggested that lignin was partially removed from the fiber surface after alkali treatment.

Reddy et al. (2012) studies the effect of alkali treatment on mechanical, thermal and chemical composition of Borassus fruit fibers. The fibers were treated with different concentrations of NaOH solution (2%, 5%, 10%, 15% and 20% w/v) for 30 min. Tensile properties of the fibers were improved by alkali treatment. The maximum tensile properties were obtained when fibers was treated with 15% w/v NaOH. With further increasing NaOH concentration, tensile properties of the fibers were decreased due to the degradation of cellulose at higher concentrations of alkali solution. Thermal stability of the alkali treated fibers was higher than that of the untreated fibers. With increasing NaOH concentration, thermal stability of alkali treated fibers were increased and found to be maximum at 15% w/v NaOH. However, the thermal stability of the fibers treated with 20% w/v NaOH was lower even to that of untreated fibers due to the degradation of cellulose. Chemical composition results indicated that the percentage of cellulose was maximum when the fibers were treated with 15% w/v NaOH and then decreased at 20% w/v NaOH. Thus, the optimum concentration of NaOH was 15% w/v for alkali treatment of Borassus fibers.

2.3.1.2.2 Silanization

Silane coupling agents are hydrophilic compounds with different groups attached to silicon. One end can interact with matrix and the other end can react with hydrophilic fiber. This reaction performs as a bridge between them. Gonzalez et al. (1999) modified short henequen fibers with 1.0% w/w vinyltris (2-methoxy-ethoxy) silane and 0.5% w/w dicumyl peroxide. The fibers were immersed in the solution for 1 h. Its deposition mechanism on the fiber surface was investigated. Fourier Transform Infrared Spectroscopy (FTIR) result showed the characteristic absorption bands of -Si-O-Si- at 700, 1030, 1145 and 1187 cm^{-1} which confirmed the presence of polysiloxanes. The shoulders at 965 and 1200 cm^{-1} corresponding to the characteristic of the -Si-O-C- bonds seemed to confirm the reaction between the hydrolyzed silane and the henequen fiber. The absorption bands at 860 and 930 cm^{-1} corresponding to the -Si-OH bond revealed the presence of residual hydrolyzed silane. These results confirmed that the silane was effectively deposited on the fiber surface.

Pickering et al. (2003) studied the effect of dichlorodiethylsilane (DCS) on fiber characteristics of radiata pine wood fiber. X-ray photoelectron spectroscopy (XPS) and nuclear magnetic resonance spectroscopy (NMR) were used to characterize the modification of the wood fiber. From XPS results, the spectrum of the untreated fiber was associated with carbon C_{1s} and O_{1s} photoelectrons. For treated fiber, there was a clear evidence from the observation of the Si_{2p} peak at 105 eV indicating that the reaction between the hydroxyl groups on the fibers and the silane was taken place. NMR results indicated that coupling had occurred between the wood fibers and DCS by reaction which produced from ether linkages between the hydroxyl groups on the wood fibers and the silane.

2.3.1.2.3 Acetylation

Acetylation is a chemical reaction that introduces an acetyl functional group into filler surface. The reaction involves the replacement of the hydrogen atom of a hydroxyl group of the filler with an acetyl group, therefore the filler surface is more hydrophobic. Albano et al. (1999) examined the effect of acetylation on thermal degradation of sisal fibers. TGA results showed that hemicellulose decomposition of acetylated fiber was missing and weight loss at the major degradation step (385ºC) was increased. This implied that the acetylation increased the thermal stability of the fiber. The residue left at 427ºC of the acetylated fiber was less than the untreated fiber because of the loss of volatile products that did not contribute to char formation. SEM micrographs revealed that fiber surface was cleaner and rougher upon acetylation when compared to the raw sisal fiber due to the removal of wax, lignin and hemicellulose. Moreover, a number of small voids on the treated fiber surface was observed.

Luz et al. (2008) studied the effect of acetylation on fiber characteristics of cellulose fibers attained from sugarcane bagasse. After fiber acetylation, FTIR results showed that there was a decrease in OH band. This indicated that the hydrogen of hydroxyl group was substituted by acetyl groups resulting in lower hydroxyl groups. In comparison with untreated fibers, the band at 1758 cm^{-1} of the treated fiber was appeared. This band was proceeded from wide C=O carbonyl peak from bonded acetyl group. SEM micrographs of the untreated cellulose fiber showed cylindrical shape fibers with 10 μm diameter and the presence of fiber bundles. After acetylation, the fibers became agglomerate with globular shapes. This indicated that the chemical modification was not so soft and promoted the hard changes in the morphology of fiber**.**

2.3.2 Matrix modification

Balasuriya et al. (2001) prepared wood flake/high density polyethylene (HDPE) composites at 50/50 w/w. HDPE matrix was modified with 0.5, 1, 2, 3 and 5 wt% maleic anhydride (MA). Wood flake was modified with 0.5, 1, 2, 3, and 4 wt%

cationic vinyl benzyl silane (CVBS). Both CVBS-modified wood flake/HDPE and wood flake/MAPE composites showed a significant increase in tensile strength, elongation at break and flexural strength when compared to the unmodified composites. Furthermore, matrix modification and wood treatment showed a slight increase in tensile modulus and flexural modulus of the composites. From mechanical properties, it indicated that HDPE modification with 1-2 wt% MA and wood flake treatment with 1-3 wt % CVBS provided the optimum properties of the composites.

Marcovich and Villar (2003) investigated the effect of matrix modification on thermal and mechanical properties of wood flour/linear low density polyethylene (LLDPE) composites. The composites were prepared at wood flour content of 30 wt%. LLDPE was modified with 1 wt% MA. Degree of crystallinity was reduced in the modified LLDPE but it increased with the addition of wood flour for the formation of the composites. MA modified LLDPE composites had better yield strength, elongation at break and toughness than the unmodified LLDPE composites due to the improvement of interfacial adhesion between wood flour and matrix.

Arbelaiz et al. (2005) prepared flax fiber/polypropylene (PP) composites and their mechanical properties were investigated. PP was modified with maleic anhydride (MA) and vinyltrimethoxy silane (VTMO). Matrix modification with MA and VTMO resulted in an enhancement of mechanical properties of the composites. The composite modified with MA had higher mechanical properties than the composite modified with VTMO.

2.3.3 Adding compatibilizer

Oksman and Lindberg (1997) investigated the effect of compatibilizer on mechanical properties of wood flour/recycled low density polyethylene (rLDPE) composites. The composites were prepared at wood flour content of 40 wt%. Maleated triblock copolymer styrene-ethylene/butylene-styrene (MA-SEBS) was used as a compatibilizer and its content was varied from 2 to 10 wt%. Tensile strength, elongation at break and impact strength of the composites were improved with increasing MA-SEBS content but kept constant when MA-SEBS content was 4 wt%. Tensile modulus of the composites was decreased with increasing MA-SEBS content.

Rana et al. (1998) studied the effects of fiber and MAPP contents on mechanical properties of jute fiber/PP composites. The fiber loadings were 30, 40, 50, and 60 wt%, and at each fiber loading, compatibilizer concentrations were 0, 1, 2, 3 and 4 wt %. Tensile properties and flexural properties of the composites were studied. With the addition of MAPP, tensile strength and flexural strength of the composites were increased while tensile modulus and flexural modulus were insignificantly changed. When MAPP content was higher than 1 wt%, no significant change of tensile strength and flexural strength was observed.

Lai et al. (2003) examined the effect of compatibilizer type on mechanical properties of wood fiber/HDPE composites. Four kinds of functionalized polyolefins, i.e, maleic anhydride grafted linear low-density polyethylene (MA-LLDPE), maleic anhydride grafted high-density polyethylene (MA-HDPE), MA-SEBS, and maleic anhydride grafted polypropylene (MAPP) were used in this study. MA-LLDPE and MA-HDPE were more effective in enhancing tensile strength and impact strength of the composites than MA-SEBS and MAPP because of their better compatibility with the HDPE matrix.

Lu et al. (2005) examined the effect of compatibilizer type and concentration on mechanical properties of wood fiber/HDPE composites. The composites were prepared at wood fiber content of 30 wt%. Maleic anhydride grafted polyethylene (MAPE) and oxidized polyethylene (OPE) were used as compatibilizers and their contents were 0, 1, 3, 5 and 10%. Tensile properties and flexural properties were investigated. With increasing compatibilizer contents, mechanical properties of the composites were increased and kept constant when compatibilizer content was higher than 3 wt%. Furthermore, MAPE was more effective than OPE in improving mechanical properties of the composites.

Pracella et al. (2006) studied the effect of glycidyl methacrylate grafted polypropylene (GMA-PP) on mechanical properties of hemp fiber/PP composites. The composites were prepared at fiber content of 10 and 20 wt%. GMA-PP content was 5 wt%. With increasing fiber content, tensile modulus of the composites was increased while elongation at break and tensile strength were reduced. The addition of GMA-PP resulted in an increase in tensile strength and tensile modulus of the composites because of the improvement of interfacial adhesion between hemp fiber and PP matrix. Moreover, the compatibilized composites showed better fiber dispersion in the PP matrix when compared to the uncompatibilized composites.

Lei et al. (2007) studied the effect of MAPE content on mechanical properties of wood fiber/rHDPE composites. The composites were prepared at wood fiber content of 30 wt%. MAPE content was varied from 0 to 2.4 wt%. Tensile modulus of rHDPE was increased about 50% when wood fiber was added into rHDPE matrix but the tensile strength and impact strength were obviously decreased. Tensile strength, impact strength and storage modulus of the composites were increased with increasing MAPE content and kept constant when MAPE content was higher than 1.8%.

Adhikary et al. (2008) investigated the effect of wood flour content and MAPP on mechanical properties of wood flour/rHDPE composites. Wood flour contents were 30, 40 and 50 wt%. Tensile properties and flexural properties were examined. The mechanical properties of the composites made from rHDPE were similar to the composites made from the virgin HDPE. With increasing wood flour content, tensile strength and flexural strength were decreased while tensile modulus and flexural modulus were increased. The addition of MAPP (5 wt%) into the composites significantly improved mechanical properties of the composites at all wood flour contents.

2.4 Flame retardancy improvement of polymer and lignocellulosic fillers/polymer composites

Nowadays, lignicellulosic fillers/polymer composites are used in many areas such as automotive interiors, furnitures, building materials and etc. However, these materials are highly flammable. Therefore, fire hazards associated with the use of the composites causing the loss of life and property should be concerned. Generally, the method to improve fire resistance of flammable materials is to use flame retardants which are directly incorporated into the materials. Flame retardants are often preferred to provide low flammability to polymeric materials because the use of flame retardants is an acceptable compromise between cost and properties

2.4.1 Flame retardant for polymer

Flame retardancy of polymer may be achieved in one of the following ways (Klyosov, 2007).

(1) By changing the pyrolysis reactions to form a carbonaceous char, which will block heat transformation and hence reduce the volatile formation and protect the polymer from further oxidation. This method poses real challenges for polymer because it has no reactive side chains thereby preventing char formation following elimination of these groups.

(2) By inhibiting the initiating radicals in the pre-flame and flame zones; typically, antimony–halogen based additives are effective here.

(3) By adding some hydrated inorganic additives which decompose endothermically and release water and hence withdraw heat from the substrate and dilute the combustible volatiles.

(4) By modifying the chemical structure of the polymer to change its decomposition procedure and/or to improve the compatibility with other flame retardants.

(5) By addition of char-forming additives preferably having an intumescent property.

Flame retardants are generally classified according to their chemical compositions. The most common classes of flame retardants are halogen compounds, phosphorus compounds, silicon compound, metal hydroxides compound and nanoparticles.

2.4.1.1 Halogen compounds flame retardants

Various formulations for the flame retardancy of polymer consisting of halogen compounds have been developed over the years. The nature of the group of halogen atom is very important because it determines the carbon-halogen ratio and carbon-halogen bond energy and hence the amount of halogen releases during burning. In general, aliphatic or alicyclic halogen compounds are more effective than aromatic halogen compounds due to the lower carbon-halogen bond energies and hence relatively easier halogen-release. The hydrogen halides released by decomposition of halogenated flame retardants are believed to react with these radicals to produce radicals which are much less active and thus inhibit the propagation of fire. The activity of halogenated flame retardants occurs mainly in the gas phase but they are thought to inhibit further burning in the solid or liquid phase where the heavy halogenated molecular fragments help to exclude oxygen from burning material and possibly encourage char formation (Horrocks and Price, 2001). Halogen compounds are an effective flame retardant. However the halogen compounds have a high smoke density and emit corrosive gases on burning and thus limit their use in some applications (Sen et al. 1991).

2.4.1.2 Phosphorus compounds flame retardants

Phosphorus compounds flame retardants used in polymer may be both inorganic and organic compounds. The reactive type organophosphorus flame retardants which have attracted more attention in flame retardant chemistry can be broadly divided into three categories: (a) simple reactive phosphate monomers; (b) linear polyphosphazenes; and (c) aromatic cyclic phosphazenes (Lu and Hamerton, 2002). Phosphorus compounds flame retardants produce a char during burning resulting in further flame retardant protective and less smoke generation. Phosphorus compounds products do not emit corrosive gases in a fire.

2.4.1.3 Silicon compounds flame retardants

Silicon compounds flame retardants such as silanes, siloxanes and silsesquioxanes are considered to be ‘environmentally friendly’ additives because their use is claimed to lead to a reduction in the harmful impact on environment. The researchers have been reported that the silicon in conjunction with a metal soap can provide a degree of flame retardancy for certain thermoplastics (Maelaury and Holub, 1981). Silicon in combination with a metal stearate shows some flame retardancy but tends to give flaming drips. Another advantage of the silicon-based additives is that they can dramatically improve the impact resistance of thermoplastics (Zhang and Horrocks, 2003).

2.4.1.4 Metal hydroxides and oxides flame retardants

The main interest of metal hydroxide flame retardants for polymer composites lies in their low toxicity, corrosion properties and emission of smoke during processing and burning. Typical metal hydroxide flame retardants are aluminum trihydrate (ATH), magnesium carbonate ($MgCO_3$) and magnesium hydroxide (MgOH). ATH and $MgCO_3$ are known to have a decomposition temperature in the range of 180-340°C, with a peak at about 320°C. MgOH has a peak decomposition temperature at about 410°C. Among the aluminium compounds, ATH is the most widely used one (Wang, Liuand Wang, 2010). These metal hydroxides decompose and release water during burning and thus reduce the heat and temperature from the substrate. Moreover, metal hydroxides provide effective flame retarding effects by generating a metal oxide coating that can act as an insulating protective layer during combustion. However, very high metal hydroxide concentrations are usually required to achieve an acceptable degree of flame retardancy (Rothon, 1990).

2.4.1.5 Nanoparticles flame retardants

Fire retardancy is one of the fields where the nanomaterials could be used successfully. Nanoparticles, e.g. nanoscale (fumed) silica, nanoclay and carbon nanotubes are increasingly used to improve flame retardancy of polymers (Zanetti et al, 2002). Nanoparticles embedded various polymer matrices reduce considerably thermal decomposition of these materials (Lee et al., 2008). Flame retardant mechanism of nanoparticles is a consequence of high performance carbonaceous-silicate char on the surface during burning which insulates the underlying material and slows down the mass loss rate of decomposition products. Moreover, nanoparticles such as carbon nanotube and nanoclay significantly increase mechanical properties and electric conductivity of polymers (Lee et al., 2008).

2.4.2 Effect of flame retardants on properties of lignocellulosic fillers/polymer composites

Flame retardants are generally used to improve the flame retardancy of lignocellulosic fillers/polymer composites. The effect of flame retardants on the properties of lignocellulosic fillers/polymer composites has been studied. Jang and Lee (2004) investigated the effect of flame retardants on flame retardant properties of paper sludge/polypropylene (PP) composites. Triphenyphosphate (TPP), ethylenebispentabromodiphenylethane (Saytex8010), $Mg(OH)_2$ and antimony trioxide were used as flame retardants. Flame retardancy of the composites was measured by the UL-94 test. The paper sludge/PP composites without flame retardants were highly flammable. With the incorporation of flame retardants, the flame retardancy of the composites was enhanced. Burning time of the composites containing TPP was 180 s. On the other hand, burning time of the composites containing $Mg(OH)_2$ was more than 300 s. The result of the UL-94 test exhibited that Saytex8010 highly enhanced the flame retardancy of the composites. In order to investigate the effect of hybrid flame retardant on flame retardancy of paper sludge/PP composites, Saytex8010 30 phr and antimony trioxide 10 phr were introduced. The results showed that hybrid flame retardant gave the most powerful flame retardancy to the composite via a synergistic effect. It was identified by TGA investigation that the flame retardancy of the composite was mainly through a gas phase mechanism. The paper sludge/PP composite with Saytex8010 25 phr and antimony trioxide 15 phr showed the highest flexural strength. The flexural strength of hybrid flame retardant systems increased as the proportion of antimony trioxide increased.

Sain et al. (2004) studied the effect of $Mg(OH)_2$, boric acid (BA) and zinc borate (ZB) as flame retardants on flammability of sawdust/PP composites and rice husk/PP composites. It was found that $Mg(OH)_2$ could effectively reduce the flammability (almost 50%) of sawdust filled PP composites. Partial replacement of about 5% $Mg(OH)_2$ with BA or ZB showed a retarding effect of flame retardant property of $Mg(OH)_2$ rather than a synergetic effect. Moreover, the mechanical properties of the composites filled with $Mg(OH)_2$ or $Mg(OH)_2$/BA or $Mg(OH)_2$/ZB showed a marginal decrease compared to the composites without a flame retardant.

García et al. (2007) studies the effect of flame retardant on the flame retarding properties of wood fiber (WF)/high density polyethylene (HDPE) composites. Aluminum trihydrtae (ATH) was used as a flame retardant and its content was 9 wt%. The burning test results showed that HDPE and wood were flammable. With the addition of ATH into the composites, the flame burnt much more slowly. The external surface of the specimens was burnt, while the internal zone was not altered. Additionally, the composites extinguished after the flame was went out. The composites just caught fire when the flame was directly falling on them. This indicated that these composites were auto-extinguishing.

Asaad et al. (2008) investigated the effect of maleic anhydride grafted polyethylene (MAPE), aluminum trihydrate (ATH) and a mixture of them on mechanical and thermal properties of wood flour/low density polyethylene (LLDPE) composites. MAPE, ATH and MAPE/ATH contents were 10, 10 and 5/5 wt%, respectively. Compressive strength of wood flour/polyethylene generally improved when ATH was added. In addition, compressive and bond strength of the composites were enhanced with the presence of MAPE, ATH and a mixture of them. MAPE and ATH also enhanced thermal stability of the composites. The combination of MAPE and ATH led to better mechanical and thermal properties of the composites compared to the use of MAPE or ATH alone.

Abu Bakar et al. (2009) studied the effect of flame retardant on flammability and mechanical properties of wood flour (WF)/polypropylene (PP) composites. Ammonium polyphosphate (APP) was used as a flame retardant to improve the flame retardancy of the composites. Thermal gravimetric analysis (TGA) data showed that 30 wt% of APP could enhance the thermal stability of the PP composites and effectively increased the char residue formation. APP could effectively reduce the flammability of the PP composites by achieving V-0 (UL-94 classification). LOI of WF/PP composites also increased with adding APP.

Suppakarn and Jarukumjorn (2009) examined the effect of $Mg(OH)_2$ and zinc borate on flammability and mechanical properties of sisal fiber/PP composites. Maleic anhydride grafted polypropylene (MAPP) was also used as a compatibilizer. With the addition of flame retardants into sisal fiber/PP composites, burning rate of the composites was decreased while thermal stability was increased. However, the addition of $Mg(OH)_2$ reduced the burning rate of the PP composite more effectively than the addition of zinc borate. No synergistic effect was observed when both $Mg(OH)_2$ and zinc borate were incorporated in the sisal fiber/PP composites. Morphology of the PP composites revealed good distribution of flame retardants and sisal fiber in PP matrix. All PP composites had lower impact strength than the neat PP. However, the sisal fiber/PP composites with the addition of $Mg(OH)_2$ and zinc borate exhibited comparable tensile and flexural properties to the sisal fiber/PP composites without adding those flame retardants. Therefore, the addition of $Mg(OH)_2$ and zinc borate enhanced flame retardancy of sisal/PP composites without deteriorating their mechanical properties.

Jeencham et al. (2010) studied the effect of combination of flame retardants on flammability and tensile properties of sisal fiber/PP composites. APP and ZB were used as flame retardants and ratios of APP and ZB were varied. The combination of APP and ZB enhanced flame retardantcy of the composites without significant change in mechanical properties of the composites. The composite containing 30 phr of APP and 10 phr of ZB exhibited better flame retardancy than the composites containing other ratios of APP to ZB.

Kurt and Mengeloglu (2011) investigated the use of ZB as a synergist with APP in wood flour/PP composites. APP and APP/ZB contents were 25 and 22/3 wt%, respectively. The addition of APP reduced tensile properties of the composites while impact strength was not much affected. The incorporation of the combination of APP and ZB increased mechanical properties of the composites without the deterioration of flame retardancy. Therefore, ZB acted as a synergist to the wood flour/PP composites containing APP.

Norzali et al. (2011) investigated the effect of ATH on compression properties of oil palm empty fruit bunch fibre (EFB)/polyurethane composites. The hybrid composite was prepared by adding 10 wt% of EFB followed by ATH at 2, 4 and 6 wt% of the overall mass of the resin. Compression stress and modulus showed the highest values at 2 wt% loading of ATH, then decreased drastically at 4 wt% and further decreased at 6 wt%. Moreover, the highest burning rate was observed at 2 wt% ATH after that the burning rate was decreased with increasing ATH content.

CHAPTER III

EXPERIMENTAL

3.1 Materials

Recycled high density polyethylene (rHDPE) was purchased from a local recycling plant. Indian Walnut *(Albizia lebbeck Benth)* sawdust was obtained from Huathalae sawmill, Nakhon Ratchasima, Thailand. Sodium hydroxide (NaOH, Carlo Erba) was purchased from Italmar (Thailand) Co., Ltd. Maleic anhydride grafted polyethylene (MAPE, Fusabond$^{®}$E MB100D, DuPont) was used as a compatibilizer. Aluminum trihydrate (ATH, APYRAL$^{®}$ 40CD, Nabaltec, average diameter of 1.3 μm) and zinc borate (ZB, Firebrake$^{®}$ ZB, Rio Tinto Mineral, average diameter of 9 μm) were used as flame retardants.

3.2 Experimental

3.2.1 rHDPE preparation

Recycled milk bottles were cleaned by water and ground by a mechanical grinder (Retsch).

3.2.2 Sawdust preparation

Sawdust was sieved using 40 mesh and later dried in an oven at 60°C for 24 h. This sawdust was called “untreated sawdust (UT)”. Some of UT was treated with sodium hydroxide (NaOH). The concentrations of NaOH solution was varied as 2, 5 and 10% w/v. The treatment time were also varied as 30 and 60 min. The sawdust was completely immersed and stirred in the NaOH solution at room temperature. Then the sawdust was washed with water several times until all adhered alkali was removed from the surface of the sawdust. The sawdust was tested for neutralization using pH meter to maintain a pH of 7. Then the sawdust was dried by an oven at 60°C for 24 h. This sawdust was called “alkali treated sawdust (AT)”

3.2.3 Sawdust characterization

3.2.3.1 Filler composition determination

Sawdust compositions were determined according to wood industry sector method (Chattopadhyay and Sarkar 1946; Sarkar et al. 1948). The sawdust was dewaxed with a mixture of benzene/ethanol taken in the ratio of 2:1 at 70ºC for 3 h. Filler to liquor ratio was 1:50. The dewaxed sawdust was washed with ethanol for 30 min, dried and weighed.

The dewaxed sawdust was boiled for 2 h in 0.7% aqueous sodium chlorite solution (adjusted to pH 4 using buffer solutions) by maintaining sawdust to liquor ratio of 1:50. Then, it was washed with 5% aqueous sodium bisulphate solution and distilled water and dried at 105°C in a hot air oven. In this step, the lignin was removed and the weight difference after this step corresponded to the lignin content whereas the remaining weight was holocellulose content (hemicellulose and cellulose). The percentage of lignin was calculated using the equation (3.1):

$$\%\ \text{Lignin} = \frac{W(g)-W_1(g)}{W(g)} \times 100 \qquad (3.1)$$

Where W is dry weight of dewaxed sample, W_1 is dry weight remaining holocellulose in sample.

The remaining holocellulose was treated with 17.5% aqueous sodium hydroxide solution to eliminate alkali soluble hemicellulose. Then, the samples were washed with acetic acid solution and distilled water and dried at 105°C in a hot air oven. The weight loss and remaining weight corresponded to the contents of hemicellulose and cellulose, respectively. The percentage of hemicellulose and cellulose was calculated using the equation (3.2) and (3.3), respectively:

$$\%\ \text{Hemicellulose} = \frac{W_1(g)\text{-}W_2(g)}{W(g)} \times 100 \qquad (3.2)$$

$$\%\ \text{Cellulose} = \frac{W_2(g)}{W(g)} \times 100 \qquad (3.3)$$

Where W is the dry weight of dewaxed sample, W_1 is the dry weight remaining holocellulose in sample and W_2 is the dry weight remaining cellulose in sample.

3.2.3.2 Functional groups analysis

FTIR spectra of untreated and alkali treated sawdust were recorded in an air atmosphere using Fourier transform infrared spectrometer (Bruker, Tensor 27) in the wavenumber range from 4000 to 400 cm^{-1}, operating in attenuated total reflectance (ATR) mode. Thirty two scans were used at a resolution of 4 cm^{-1}.

3.2.3.3 Thermal properties

Thermal decomposition temperature and weight loss of untreated and alkali treated sawdust were analyzed by a thermogravimetric analyzer

(Perkin Elmer, STD 2960). The temperature range was from room temperature to 800°C with a heating rate of 10°C/min under a nitrogen atmosphere. The weight of specimen between 10 to 15 g was used.

3.2.3.4 Morphological properties

Surface morphologies of untreated and alkali treated sawdust were examined using a scanning electron microscope (SEM, JEOL JCM-6010) at 10 kV. The samples were coated with goal before examination.

3.2.4 Composite preparation

The composites of recycled high density polyethylene (rHDPE) and UT or AT were prepared at sawdust loading of 30, 40 and 50 wt%. The composites were mixed in a twin screw extruder (Brabender, DSE 35/17D). The temperatures of the four processing zones were 175, 180, 185 and 190°C, respectively. The screw speed was 20 rpm. The test specimens were molded by a compression molding machine (Labtech, LP20-B) at 190°C under a constant pressure of 120 psi for 10 min. According to mechanical, thermal and flame retarding properties, the composite with the optimum properties was chosen to study the effect of flame retardant on the properties of the composites.

ATH was used as a flame retardant for sawdust/rHDPE composites. ATH contents were varied as 10, 20 and 30 phr. The composite with the optimum mechanical, flame retarding and thermal properties was selected to study the effect of the combination of flame retardants on the properties of sawdust/rHDPE composites.

The effect of the combination of ATH and ZB on the properties of the composites was also investigated. The ratios of ATH:ZB were 1:1, 1:2 and 2:1. The composite having optimum mechanical, flammability and thermal properties was chosen to study the effect of compatibilizer on the properties of sawdust/rHDPE composites containing flame retardant. Maleic anhydride grafted polyethylene (MAPE) was used as a compatibilizer. MAPE contents were 1, 3 and 5 phr.

3.2.5 Composite characterization

3.2.5.1 Mechanical properties

Tensile properties of rHDPE and rHDPE composites were tested by following ASTM D638 using a universal testing machine (Instron, 5565) with a load cell of 5 kN, a crosshead speed of 50 mm/min and a guage length of 115 mm.

Flexural properties of rHDPE and rHDPE composites were tested by following ASTM D790 using a universal testing machine (Instron, 5565) with a span length of 56 mm and a crosshead speed of 15 mm/min.

Izod impact strength of rHDPE and rHDPE composites was performed according to ASTM D256 using an impact testing machine (Atlas, BPI). Notched impact strength was tested using the impact pendulum with impact energy of 2.7 J.

All the mechanical properties were performed at room temperature and five specimens were tested for each composition.

3.2.5.2 Flammability

Flammability of rHDPE and rHDPE composites was studied by vertical and horizontal burning tests according to ASTM D3801 and ASTM D635, respectively. In the case of horizontal burning rate, the specimen was held horizontally and flame was applied to one end of the specimen. The time for flame to reach from the first reference mark (25 mm from the end) to second reference mark, which was at 100 mm from the end, was measured. Then, burning rate of the composites was calculated by using the equation (3.4):

$$V = 60L/t \tag{3.4}$$

Where V is the burning rate (mm/min). L is the burned length (mm). t is the time (s) of burning.

Flammability of rHDPE and rHDPE composites were classified by the vertical burning test. The specimen was held vertically at 300 ± 10 mm above a horizontal layer of cotton. Distance between the lower end of the specimen and the burner tube was 10 ± 1 mm. The flame was applied for 10 s and then was removed until flaming stopped at which time the flame was reapplied for another 10 s and then removed. The time was recorded. Five specimens from each composite were tested. The V-0, V-1 and V-2 classification according to ASTM D3801 marks self-extinguishing behavior of the materials in the vertical burning test. The lowest flame retardancy rating is V-2 (flaming time ≤ 30 s and cotton indicator ignited by flaming particles or 23 drops) and V-0 (no dripping, flaming time reduced (≤ 10 s) represents a strongly flame retarded material, while V-1 (flaming time ≤ 30 s) is intermediate term between V-0 and V-2. The flammable materials which cannot be classified according to this method, are marked with code NC (no classification, complete combustion of the specimen)

3.2.5.3 Thermal properties

Thermal behaviors of rHDPE and rHDPE composites were analyzed using a thermogravimetric analyzer (Perkin Elmer, STD 2960). Thermal decomposition of each sample was examined under nitrogen atmosphere. The weight of specimens between 10 to 15 mg was used. The specimens were heated from room temperature to 800°C with a heating rate of 10°C/min.

3.2.5.4 Morphological properties

A scanning electron microscope (SEM, JEOL JCM6010) was used to examine morphology of tensile fracture surface of rHDPE composites. The specimens were coated with gold before analysis. Acceleration voltage of 10 kV was used to collect SEM images of the samples.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Effect of alkali treatment on physical properties of sawdust

4.1.1 Chemical compositions

The chemical compositions of untreated sawdust and alkali treated sawdust are shown in Table 4.1. After alkali treatment at 2% w/v NaOH for 30 min, hemicellulose and lignin of alkali treated sawdust was remarkablely decreased while cellulose content was increased. This was because hemicellulose and lignin were removed during alkali treatment (Ndazi et al., 2007).

When NaOH concentration was increased to 5% and 10% w/v, hemicellulose and lignin contents of alkali treated sawdust were slightly decreased whereas cellulose content was increased. Reddy et al. 2012 also reported that hemicellulose and lignin contents of alkali treated jute fiber were reduced with increasing NaOH concentration from 2 to 20% w/v while cellulose content was increased due to the removal of hemicellulose and lignin.

At the same concentration of NaOH solution, hemicellulose and lignin contents of alkali treated sawdust at treatment time of 60 min slightly reduced while cellulose content increased when compared to alkali treated sawdust at treatment time of 30 min. Ray and Sakar (2000) also observed that with increasing treatment time, hemicellulose and lignin contents were reduced but cellulose content was increased.

From the results, it was clearly seen that the treatment at low concentration of NaOH (2% w/v) for short time (30 min) was successfully removed hemicellulose and lignin from sawdust.

Table 4.1 Chemical compositions of untreated sawdust and alkali treated sawdust.

Treatment conditions	**Cellulose (%)**	**Hemicellulose (%)**	**Lignin (%)**
Untreated sawdust (UT)	56.62	12.29	27.38
Alkali treated 2% w/v for 30min (2%AT_30min)	58.09	10.26	25.83
Alkali treated 5% w/v for 30min (5%AT_30min)	59.01	9.00	25.11
Alkali treated 10% w/v for 30min (10%AT_30min)	59.90	8.43	25.71
Alkali treated 2% w/v for 60min (2%AT_60min)	58.56	9.94	25.23
Alkali treated 5% w/v for 60min (5%AT_60min)	59.09	8.28	25.21
Alkali treated 10% w/v for 60min (10%AT_60min)	59.64	8.02	25.12

4.1.2 Functional groups analysis

Fourier transform infrared spectroscopy (FTIR) spectrum of untreated sawdust is shown in Figure 4.1 and its characteristic bands are listed in Table 4.2. The absorption band at 3339 cm^{-1} was a characteristic of hydroxyl (O-H) stretching vibration originating from cellulose, hemicellulose and lignin. The absorption band at 2909 cm^{-1} was due to the C-H stretching vibration of CH in cellulose, hemicellulose and lignin. There was an absorption band at 1732 cm^{-1} belonged to the carbonyl (C=O) stretching vibration of ester group in hemicellulose or carboxylic acid in lignin. The absorption bands were found at 1600 and 1505 cm^{-1} indicating the aromatic ring stretching vibration of lignin. The absorption band at 1454 cm^{-1} was assigned to CH_2 asymmetric bending in cellulose. The absorption band at 1420 cm^{-1} was attributed to C-H deformation in lignin. The absorption band at 1367 cm^{-1} was attributed to C-H deformation in hemicellulose. The absorption bands at and 1322 and 1231 cm^{-1} were related to C-O stretching vibration of aromatic ring and acetyl group of lignin, respectively. Little shoulder observed at 1154 and 1103 cm^{-1} were due to C-O-C and O-H stretching in cellulose, respectively. The absorption band at 1029 cm^{-1} was described to the C-O stretching of hydroxyl functionality in cellulose. Finally, the absorption band at 894 cm^{-1} was associated β-glycosidic linkage of polysaccharide (Ray and Sarkar, 2000; Lai et al., 2003; Muller et al., 2009; Reddy et al., 2012).

FTIR spectra of untreated sawdust and alkali treated sawdust at various NaOH concentrations and treatment times are shown in Figure 4.2. After alkali treatment, the changes in absorption bands at 3339, 2909, 1732, 1600, 1505, 1367, 1322 and 1231 cm^{-1} were observed. The intensity of the peak at 3339 and 2909 cm^{-1} of the alkali treated sawdust was decreased comparing to the untreated sawdust due to the removal of hemicellulose and lignin (Ray et al., 2001). The peak at 1732 cm^{-1} was remarkably reduced after alkali treatment. This indicated that the alkali treatment significantly removed hemicellulose and lignin (Ray et al., 2001). The reduction of the peak intensity at 1367 cm^{-1} assigned for C-H deformation in hemicellulose was found after alkali treatment (Sinha et al., 2009). Moreover, the peak intensities at 1600, 1505, 1332 and 1231 cm^{-1} were decreased indicating that amount of lignin was decreased by alkali treatment (Reddy et al., 2012). From FTIR results, it can be concluded that alkali treatment removed hemicellulose and lignin from sawdust.

Ramadevi et al. (2012) also reported that after alkali treatment, the peak intensity at 3347, 1732, 1593, 1310 and 1245 cm^{-1} of abaca fiber was reduced due to the removal of hemicellulose and lignin components.

Nonetheless, NaOH concentration and treatment time were slightly reduced the peak intensity of alkali treated sawdust. This result was corresponded to filler composition results in previous section.

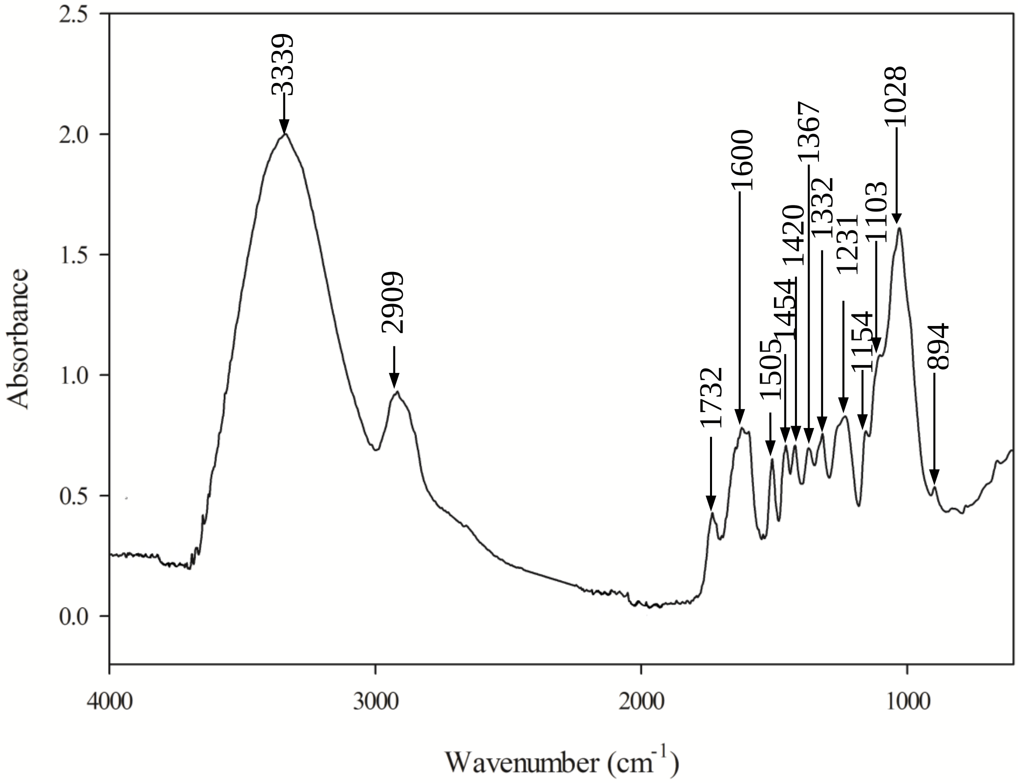


Figure 4.1 ATR-FTIR spectrum of untreated sawdust.

Table 4.2 FTIR peak positions of untreated sawdust (Ray and Sarkar, 2000; Lai et al., 2003; Muller et al., 2009; Reddy et al. 2012).

Wavenumber (cm^{-1})	**Assignment**	**Source**
3339	O-H stretching	Cellulose, hemicellulose and lignin
2909	C-H stretching	Cellulose and hemicellulose
1732	C=O stretching	Carboxylic acid in lignin or ester group in hemicellulose
1600	C=O stretching	Aromatic ring of lignin
1505	C=C stretching	Aromatic ring of lignin
1454	CH_2 symmetric bending	Cellulose
1420	C-H deformation	Aromatic ring in lignin
1367	C-H deformation	Hemicellulose
1322	C-O stretching	Aromatic ring in lignin
1231	C=O stretching	Acetyl group of lignin
1154	C-O-C antisymmetric stretching	Cellulose
1103	O-H stretching	Cellulose
1029	C-O and O-H stretching	Cellulose
894	β-glycosidic linkage	Polysaccharide

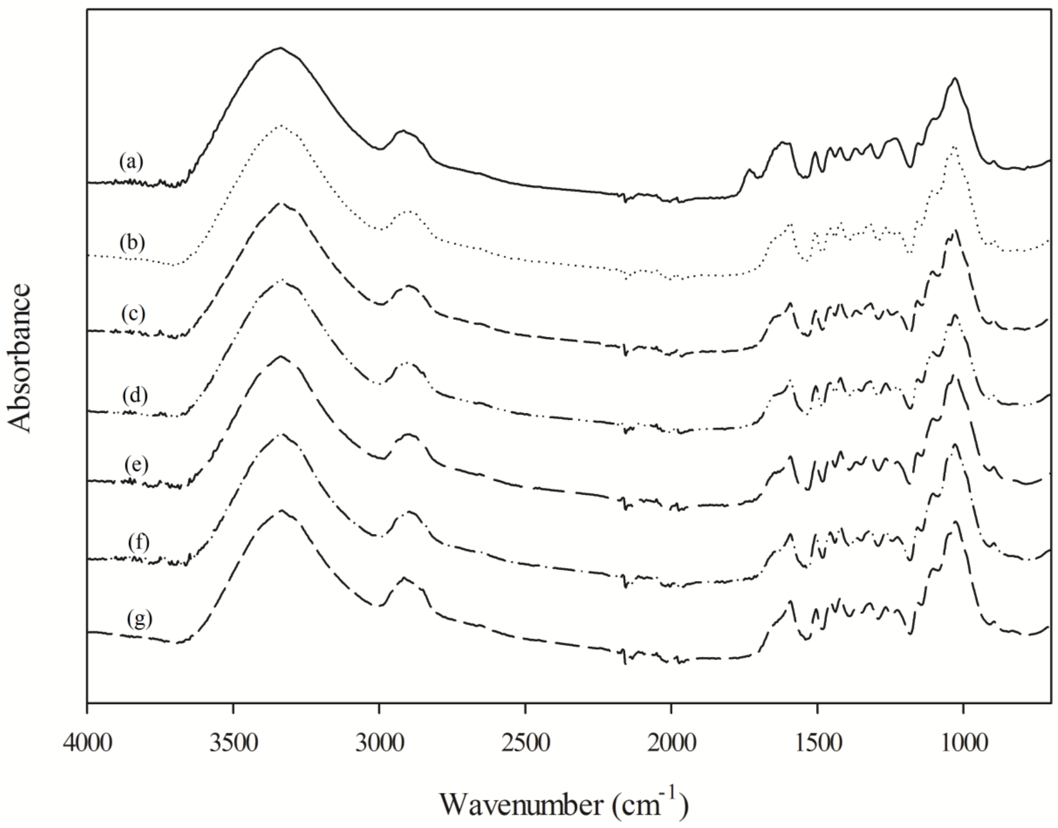


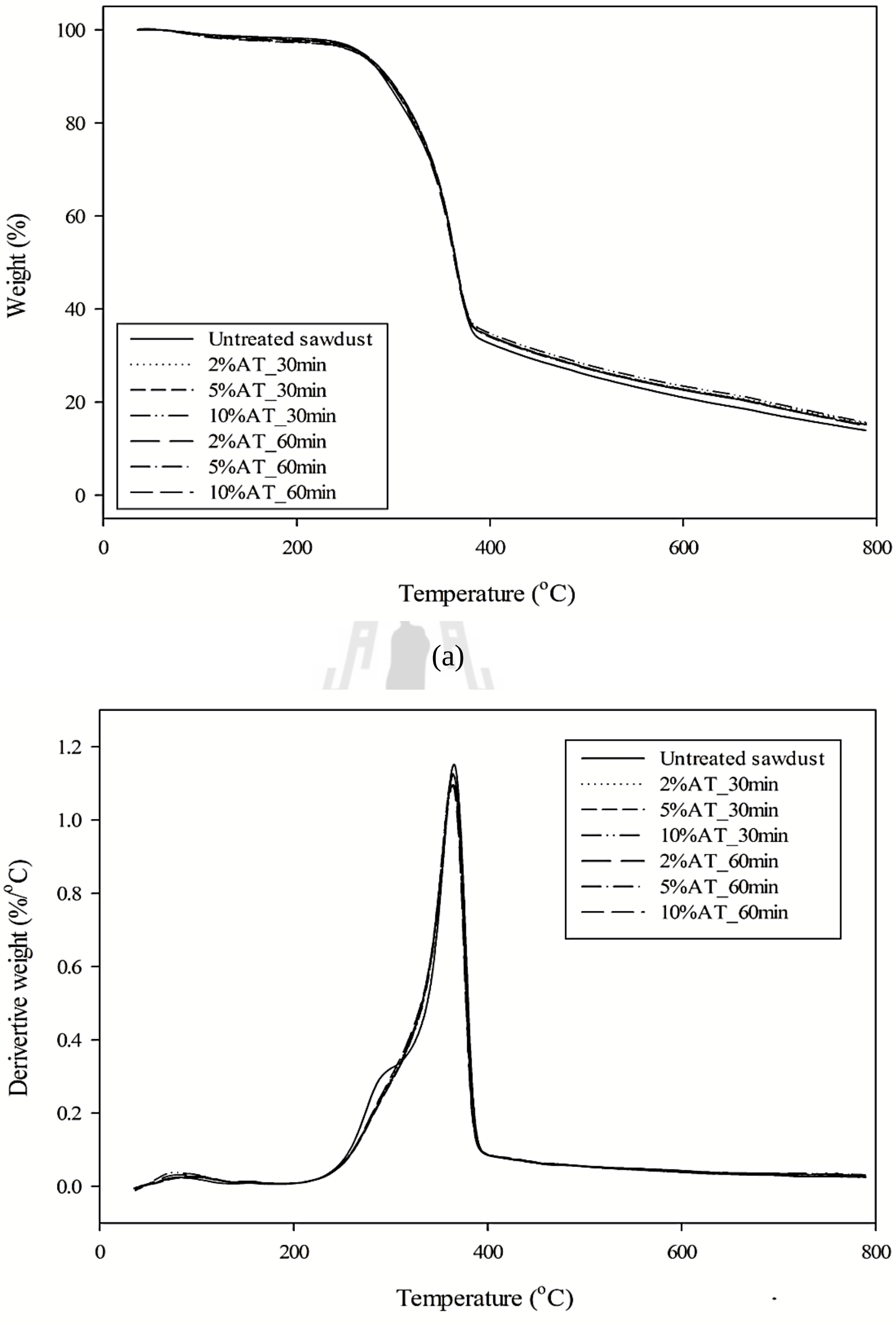
Figure 4.2 ATR-FTIR spectra of (a) UT, (b) 2%AT_30min, (c) 5%AT_30 min, (d) 10%AT_30 min, (e) 2%AT_60 min, (f) 5%AT_60 min and (g) 10%AT_60 min.

4.1.3 Thermal properties

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) curves of untreated sawdust and alkali treated sawdust are shown in Figure 4.3 and thermal decomposition characteristics of untreated and alkali treated sawdust summarized in Table 4.3. It was found that untreated sawdust had three stages of the decomposition process. The 1st weight loss step of untreated sawdust was observed below 100°C due to the evaporation of moisture. The 2nd weight loss step observed as a shoulder peak at 283.97°C was caused by hemicellulose degradation (Ouajai et al., 2004). The 3rd weight loss step at 344.68°C indicated the degradation of cellulose containing in sawdust (Ouajai et al., 2004). Lignin degradation was unable to see in the TGA and DTA curves because it was overlapped with the hemicellulose and cellulose decomposition (Manfredi et al., 2006). Lignin decomposed between 250 and 500°C (Kim et al., 2003). Ndazi et al. (2007) also observed three stage decomposition of rice husk. The 1st weight loss was found at around 60°C due to the evaporation of moisture. The 2nd weight loss observed at 229°C was involved the decomposition of hemicellulose. The 3rd weight loss observed at 341°C was the decomposition of cellulose. In addition, char residue of untreated sawdust at 800°C was 12.78%.

After alkali treatment at 2% w/v for 30 min, the 1st weight loss temperature of alkali treated sawdust was observed below 100°C due to the evaporation of moisture. The 2nd weight loss temperature (shoulder peak) of alkali treated sawdust was disappeared. This indicated that hemicellulose was removed by alkali treatment (Ouajai et al., 2004). The 3rd weight loss temperature (cellulose decomposition temperature) of alkali treated sawdust was remarkably shifted to higher temperature from 344.68°C to 353.65°C indicating the enhancement of the thermal stability of alkali treated sawdust. This was because some components of sawdust such as impurities, wax and hemicellulose which degraded at low temperature may be removed during alkali treatment (Albano et al., 1999). Khan et al. (2012) also found that the cellulose decomposition temperature of alkali treated coconut husk fiber was shifted to higher temperature when compared to untreated coconut husk fiber. Moreover, char residue of alkali treated sawdust was increased. Ray et al. (2001) found that char residue of jute fiber was increased after alkali treatment. Ndazi et al. (2001) also found that char residue of rice husks was slightly increased after alkali treatment due to an increase in cellulose content after alkali treatment.

However, the decomposition temperature and char residue of alkali treated sawdust slightly changed with increasing NaOH concentration and treatment time. This indicated that the optimum alkali treatment condition was with 2% w/v NaOH for 30 min. Reddy et al. (2012) also reported no significant change in decomposition temperature of alkali treated Borassus fruit fiber with increasing NaOH concentration from 2 to 15% w/v. In addition, Ray et al. (2002) also observed that no significant change of the decomposition temperature of alkali treated jute fiber when treatment time was increased from 2 to 8 h.



(b)

Figure 4.3 Thermal decomposition characteristics of untreated and alkali treated sawdust with different NaOH concentrations and treatment times (a) TGA and (b) DTA.

Table 4.3 Thermal decomposition characteristics of untreated and alkali treated sawdust with different NaOH concentrations and treatment times.

Sample name	**1st weight loss temperature (°C)**	**2nd weight loss temperature (°C)**	**3rd weight loss temperature (°C)**	**Residue (%)**
Untreated sawdust	81.60	283.97	344.68	12.78
2%AT_30min	91.43	-	353.65	15.48
5%AT_30min	91.84	-	353.86	16.30
10%AT_30min	92.28	-	354.28	16.42
2%AT_60min	92.96	-	353.81	16.07
5%AT_60min	92.81	-	354.33	17.05
10%AT_60min	93.30	-	354.46	17.87

4.1.4 Morphological properties

SEM micrographs of untreated and alkali treated sawdust are illustrated in Figure 4.4 (a)-(h). It can be seen that the surface of untreated sawdust was covered by a layer of surface impurities on its surface as displayed in Figure 4.4 (b). After alkali treatment at 2% w/v NaOH for 30 min, the surface of alkali treated sawdust was cleaner than that of untreated sawdust as shown in Figure 4.4 (c) due to the removal of impurities, oil and wax (Karthikeyan et al., 2012). It was also found that roughness of the sawdust surface was obviously enhanced due to the removal of hemicellulose and lignin (Lui et al., 2009). After alkali treatment at 5% and 10% w/v NaOH for 30 min, an insignificant change of surface roughness of sawdust was found as displayed Figure 4.5 (d)-(e).

After alkali treatment at 2% w/v NaOH for 60 min, the surface roughness of sawdust was insignificantly changed when compared to sawdust treated with 2% w/v NaOH for 30 min as illustrated in Figure 4.5 (f). In addition, no significant change of surface roughness of sawdust was observed with increasing NaOH concentration as shown in Figure 4.5 (g)-(h). Therefore, the optimum condition of alkali treatment was 2% w/v NaOH for 30 min.

From chemical composition, functional group analysis, thermal properties and morphological properties, the optimum alkali treatment condition was 2% w/v NaOH for 30 min. Therefore, sawdust treated with 2% w/v NaOH for 30 min was selected to study the effect of sawdust content on the properties of sawdust/rHDPE composites.

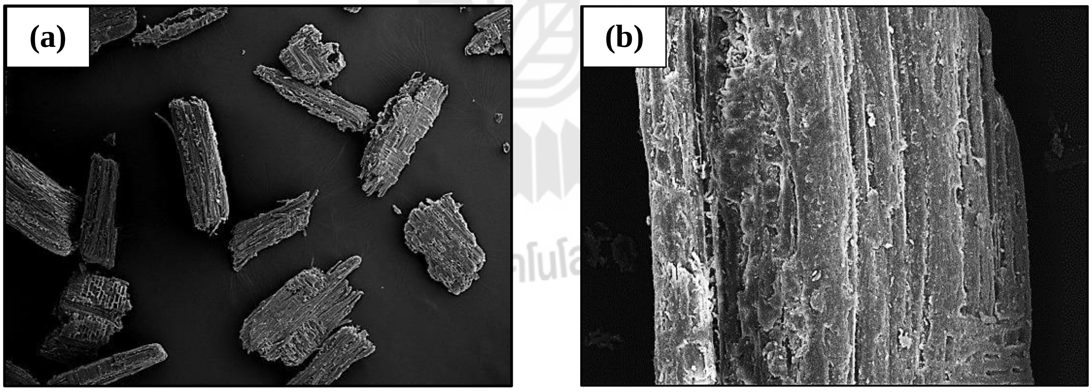


Figure 4.4 SEM micrographs of (a) UT (50x), (b) UT (200x), (c) 2%AT_30min (200x), (d) 5%AT_30 min (200x), (e) 10%AT_30 min (200x), (f) 2%AT_60 min (200x), (g) 5%AT_60 min (200x) and (h) 10%AT_60 min (200x).

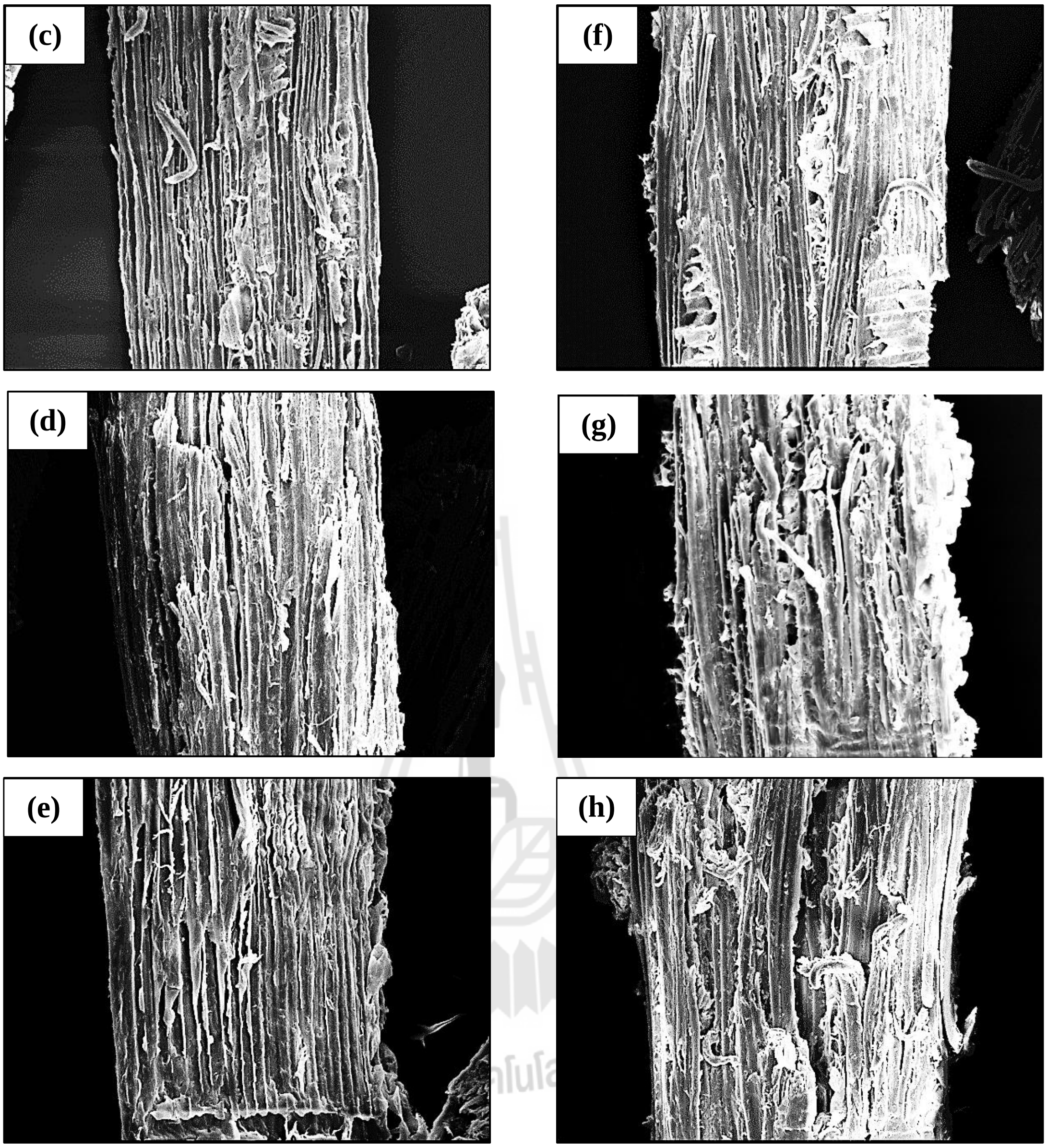


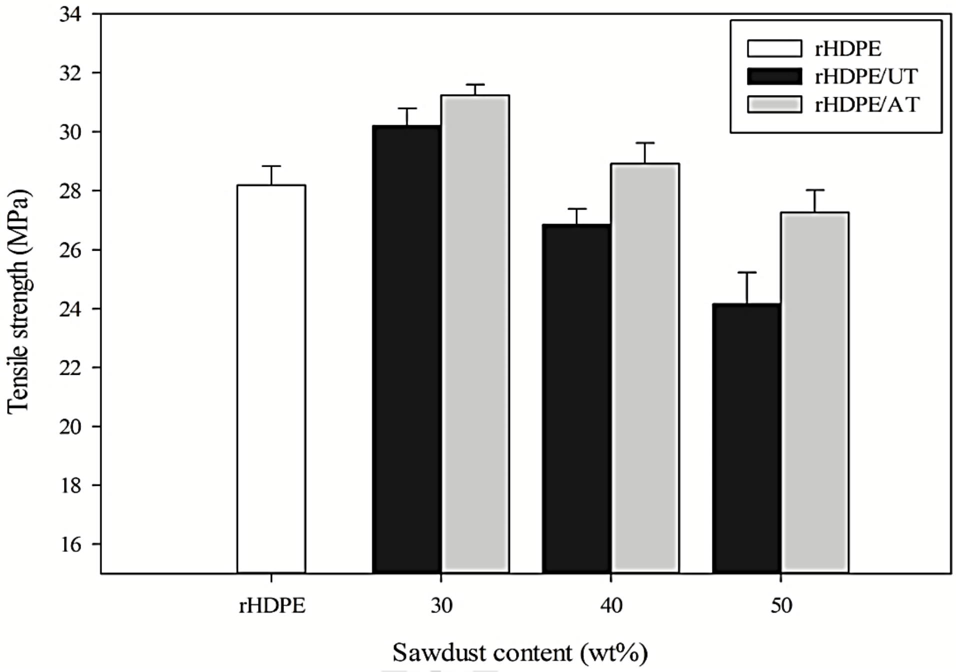
Figure 4.4 SEM micrographs of (a) UT (50x), (b) UT (200x), (c) 2%AT_30min (200x), (d) 5%AT_30 min (200x), (e) 10%AT_30 min (200x), (f) 2%AT_60 min (200x), (g) 5%AT_60 min (200x) and (h) 10%AT_60 min (200x) (continued).

4.2 Effects of sawdust content and alkali treatment on physical properties of sawdust/rHDPE composites

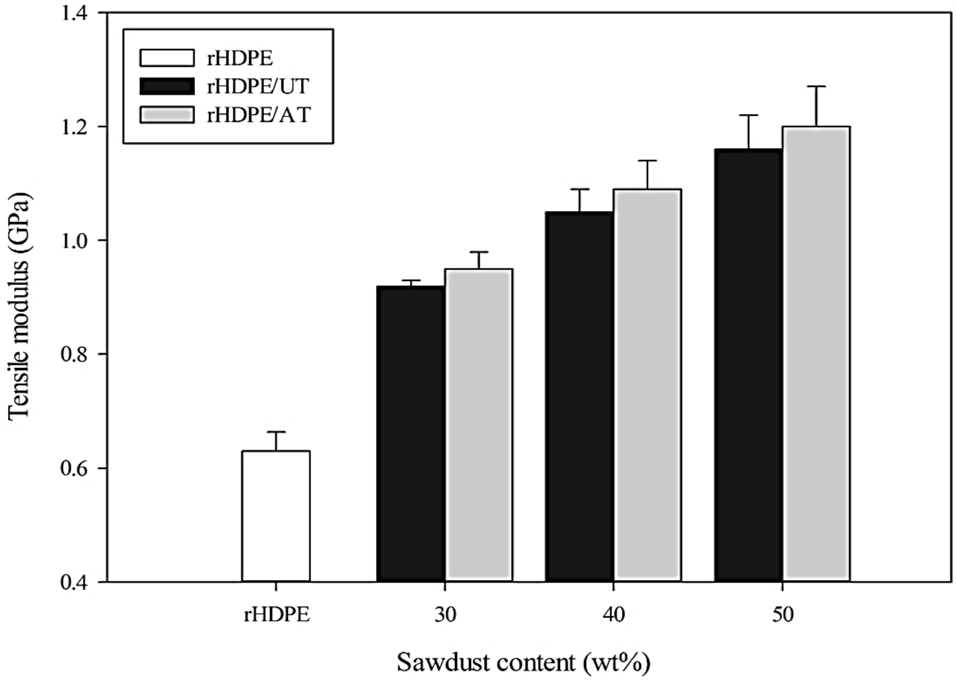
4.2.1 Mechanical properties

Tensile properties of rHDPE and rHDPE composites are shown in Table 4.4. With the addition of untreated sawdust, tensile strength and tensile modulus of rHDPE were improved but elongation at break was reduced. This result suggested that sawdust acted as reinforcing filler in the system (Danyadi et al., 2007). In addition, tensile strength of the composites decreased with increasing sawdust content as illustrated in Figure 4.5 (a). This may be due to the incompatibility between sawdust and rHDPE matrix (Atuanya et al., 2011). From Figure 4.5 (b), tensile modulus of the composites enhanced with increasing sawdust content indicating the ability of sawdust to impart greater stiffness to the composites (Atuanya et al., 2011). The reduction of elongation at break of the composites was found when sawdust content was increased as shown in Figure 4.5 (c). This was because the presence of sawdust caused matrix to lose its ability against elastic deformation (Li et al., 2004).

Tensile strength, tensile modulus and elongation at break of alkali treated sawdust/rHDPE composites were slightly higher than those of untreated sawdust/rHDPE composites at all sawdust contents due to the enhancement of interfacial adhesion between sawdust and rHDPE matrix. Alkali treatment increased the filler surface roughness by a removal of impurities, waxes, hemicellulose and lignin on filler surface resulting in better mechanical interlocking between sawdust and rHDPE matrix (Joseph et al., 1996; Ikhlef et al., 2012).

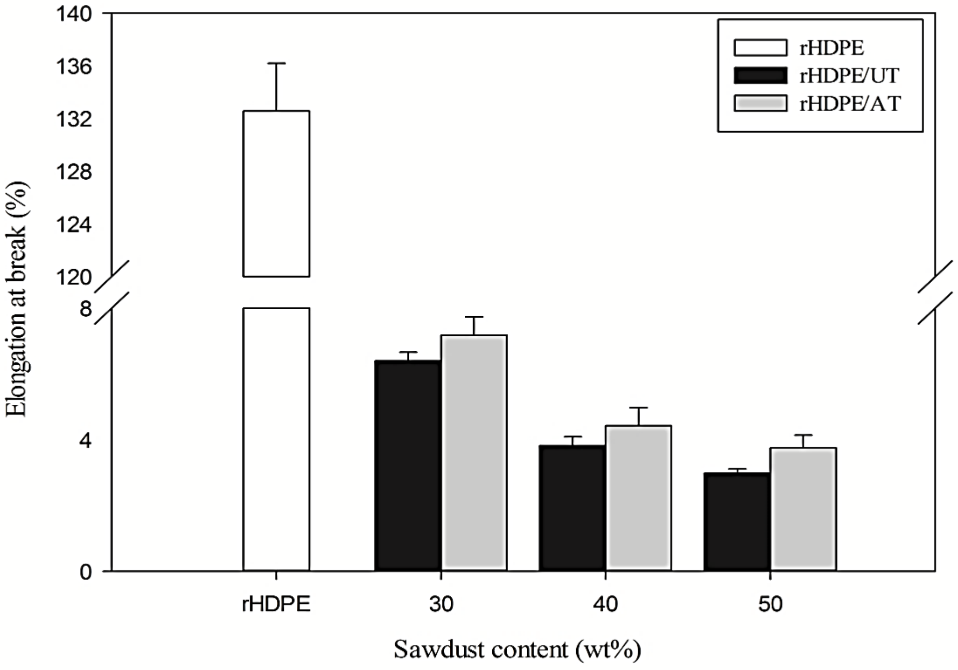


(a)



(b)

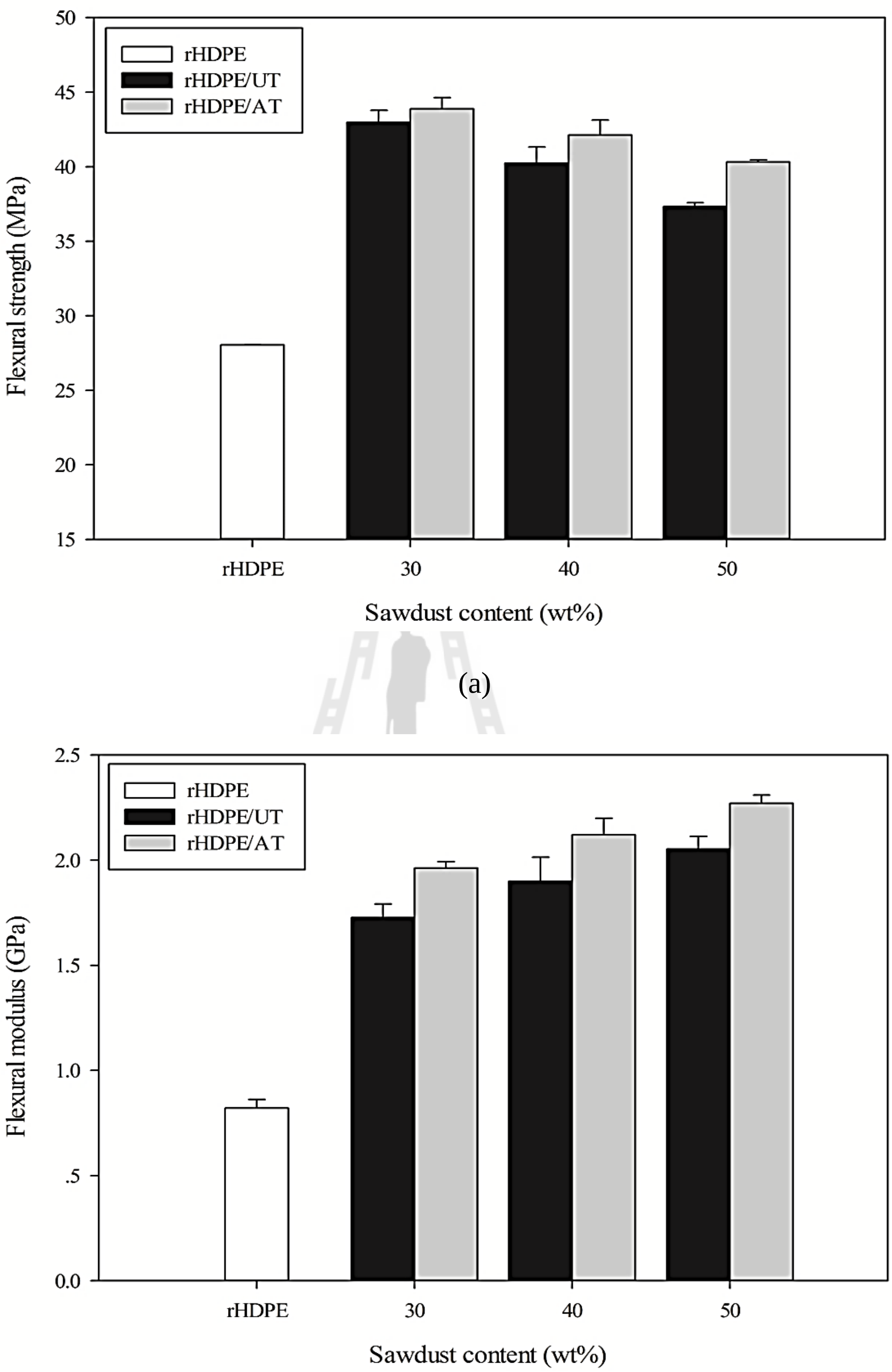
Figure 4.5 Tensile properties of rHDPE and rHDPE composites with different sawdust contents (a) tensile strength, (b) tensile modulus and (c) elongation at break.



(c)

Figure 4.5 Tensile properties of rHDPE and rHDPE composites with different sawdust contents (a) tensile strength, (b) tensile modulus and (c) elongation at break (continued).

Flexural strength and flexural modulus of rHDPE and rHDPE composites are shown in Table 4.4. Flexural properties of rHDPE composites showed the same trend as tensile properties. From Figure 4.6 (a)-(b), flexural strength and flexural modulus of rHDPE enhanced with incorporating sawdust. With increasing sawdust content, flexural strength of the composites decreased whereas flexural modulus increased. Flexural strength and flexural modulus of alkali treated sawdust/rHDPE composites were slightly higher than those of untreated composites at all sawdust contents due to the improvement of interfacial adhesion between sawdust and rHDPE.



(a)

(b)

Figure 4.6 Flexural properties of rHDPE and rHDPE composites with different sawdust contents (a) flexural strength and (b) flexural modulus.

Impact strength of rHDPE and rHDPE composites is shown in Table 4.4. It was seen that all composites showed lower impact strength than rHDPE because the addition of sawdust created stress concentration that required less energy to initiate cracking. From Figure 4.7, with increasing sawdust content, impact strength of the composites was decreased due to an increase in filler agglomeration creating more regions of stress concentration (Yao et al., 2008). The improvement of impact strength was observed in the composites prepared from alkali treated sawdust. This was due to the improvement of interfacial adhesion between sawdust and matrix resulting in better stress transfer during testing.

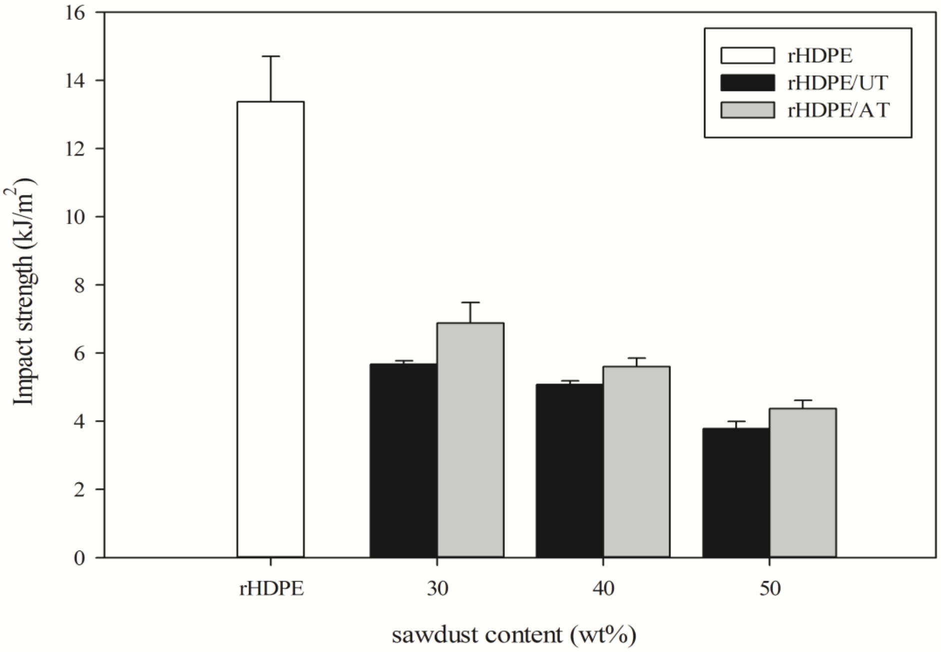


Figure 4.7 Impact strength of rHDPE and rHDPE composites with different sawdust contents.

Table 4.4 Mechanical properties of rHDPE and rHDPE composites with different sawdust contents.

Sample name	**Tensile strength (MPa)**	**Tensile modulus (GPa)**	**Elongation at break (%)**	**Flexural strength (MPa)**	**Flexural modulus (GPa)**	**Impact strength (kJ/m^2)**
rHDPE	28.19±0.65	0.63±0.03	132.57±3.63	28.06±0.01	0.82±0.04	13.68±0.88
rHDPE/30UT	30.21±0.59	0.91±0.01	6.40±0.26	42.99±0.78	1.73±0.06	5.67±0.10
rHDPE/40UT	26.86±0.53	1.05±0.04	3.82±0.27	40.25±1.07	1.90±0.11	5.08±0.10
rHDPE/50UT	24.16±1.07	1.17±0.07	2.98±0.13	37.32±0.27	2.05±0.05	3.78±0.21
rHDPE/30AT	31.24±0.36	0.95±0.03	7.18±0.55	43.88±0.76	1.96±0.03	6.88±0.61
rHDPE/40AT	28.92±0.70	1.09±0.04	4.42±0.55	42.13±0.99	2.12±0.08	5.60±0.25
rHDPE/50AT	27.27±0.76	1.20±0.07	3.75±0.39	40.31±0.14	2.27±0.04	4.37±0.24

4.2.2 Flammability

UL-94 test results and horizontal burning rate of rHDPE and rHDPE composites are shown in Table 4.5. From UL-94 test, rHDPE and rHDPE composites were completely combusted and were not classified in UL-94 classification. This indicated that rHDPE and sawdust/rHDPE composites had high sensitivity to flame (Bakar et al., 2010). Alkali treated sawdust/rHDPE composites were not also classified in UL-94 classification.

Sawdust/rHDPE composites had higher burning rate than rHDPE and the burning rate increased with increasing sawdust content as illustrated in Figure 4.8. This also indicated that sawdust/rHDPE composites had high sensitivity to flame (Li et al., 2004). Alkali treated sawdust/rHDPE composites showed slightly lower burning rate than untreated sawdust/rHDPE composites. This may be due to an increase in char residue of the composites by alkali treatment as observed from TGA results (section 4.2.3). When the char layer built up, it may separate the composites from heat and oxygen (Chapple and Anandjiwala, 2012)

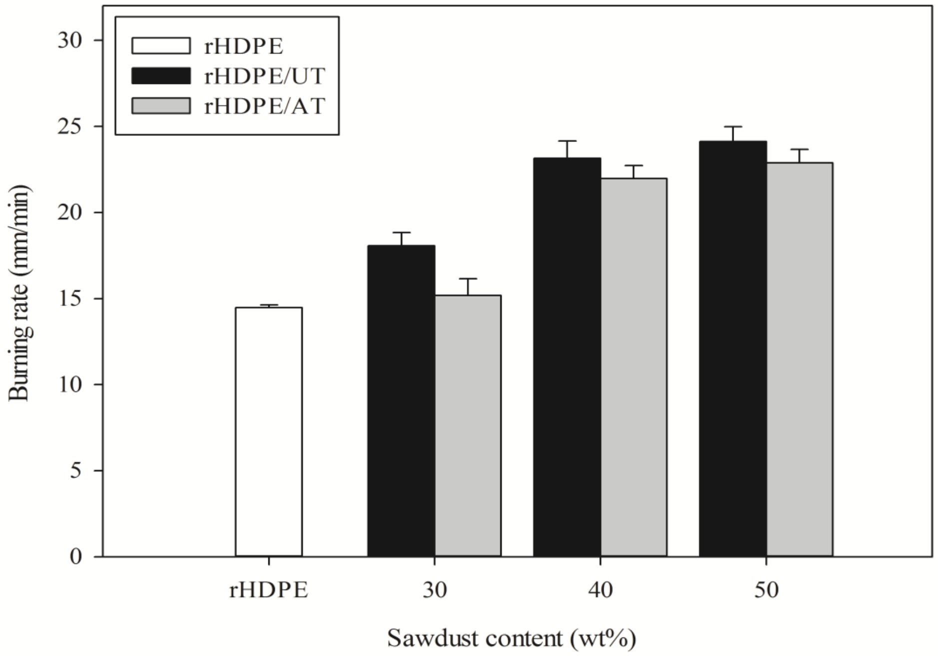


Figure 4.8 Burning rate of rHDPE and rHDPE composites with different sawdust contents.

Table 4.5 UL-94 classification and horizontal burning rate of rHDPE and rHDPE composites with different sawdust contents.

Sample name	**UL-94 classification**	**Horizontal burning rate (mm/min)**
rHDPE	NC	15.27±1.38
rHDPE/30UT	NC	21.58±0.61
rHDPE/40UT	NC	24.38±0.30
rHDPE/50UT	NC	26.00±0.86
rHDPE/30AT	NC	18.07±0.77
rHDPE/40AT	NC	22.59±0.29
rHDPE/50AT	NC	23.67±0.42

4.2.3 Thermal properties

TGA and DTA curves of rHDPE and rHDPE composits are shown in Figure 4.9. Sawdust/rHDPE composites showed two weight loss processes at about 345.17°C and 475.50°C. The 1st weight loss was attributed to thermal degradation of sawdust (Li et al., 2004). The 2nd weight loss was associated to decomposition of rHDPE. The 1st weight loss temperature of the composites continuously reduced when sawdust content was increased. Kim et al. (2003) also found that the 1st weight loss temperature of rice husk flour (RHF)/polypropylene (PP) composites decreased with increasing RHF loading due to the low thermal decomposition temperature of RHF. The addition of untreated sawdust into rHDPE matrix reduced the decomposition temperature of rHDPE. Li and He et al. (2003) also found that an increase in fiber content led to a decrease in linear low density polyethylene (LLDPE) decomposition temperature of wood fiber/LLDPE composites. This was because free radicals were produced in thermal degradation of wood at high temperature and accelerated the reactions of the thermal degradation of LLDPE.

Alkali treatment increased the sawdust decomposition temperature of the composites. This may be due to the fact that some components in sawdust which degraded at low temperature were extracted out during alkali treatment resulting in the composites with higher thermal stability (Kaewkuk et al., 2010). Moreover, char residue of alkali treated sawdust/rHDPE composites was slightly higher than that of untreated sawdust/rHDPE composites as seen in Table 4.6. The char residue of the composites was attributed to the presence of sawdust in rHPDE matrix. The increase in char residue of alkali treated sawdust/rHDPE composites was due to the increase in char residue of sawdust after alkali treatment as observed in Figure 4.3 (section 4.1.3).

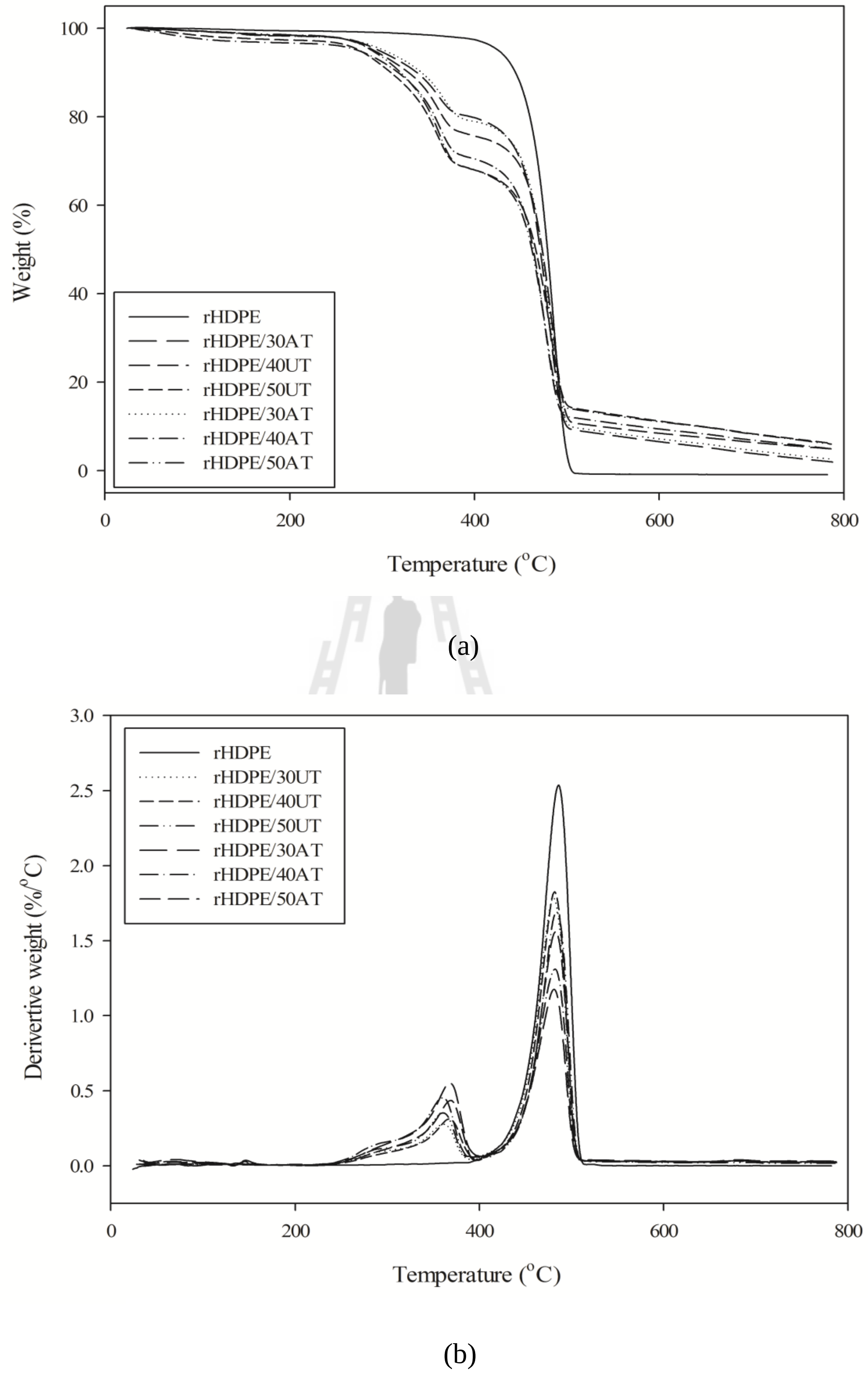


Figure 4.9 Thermal decomposition characteristics of rHDPE and rHDPE composites with different sawdust contents (a) TGA and (b) DTA.

Table 4.6 Thermal degradation characteristics of rHDPE and rHDPE composites with different sawdust contents.

Sample name	**1st weight loss temperature (°C)**	**2nd weight loss temperature (°C)**	**Residue (%)**
rHDPE	-	477.67	0
rHDPE/30UT	345.17	475.50	5.11
rHDPE/40UT	344.50	474.50	7.57
rHDPE/50UT	343.83	473.83	8.60
rHDPE/30AT	353.83	476.33	6.71
rHDPE/40AT	352.67	475.85	8.32
rHDPE/50AT	351.50	474.67	8.85

4.2.4 Morphological properties

SEM micrographs of tensile fracture surface of untreated and alkali treated sawdust/rHDPE composites at 30 wt% of sawdust are shown in Figure 4.10. As shown in Figure 4.10 (a), gap between rHDPE and sawdust was observed. This indicated the poor compatibility between sawdust and rHDPE matrix leading to the reduction of mechanical properties of rHDPE composites. From Figure 4.10 (b), alkali treatment enhanced the interfacial adhesion between sawdust and rHDPE matrix resulting in the improvement of the mechanical properties of the composites.

SEM micrographs of tensile fracture surface of untreated sawdust/rHDPE composites at various sawdust contents are illustrated in Figure 4.11 (a)-(c). It can be seen that holes which left after sawdust pull out from rHDPE matrix were observed. With increasing sawdust content, the agglomeration of untreated sawdust in rHDPE matrix was found. On the other hand, good interfacial adhesion between alkali treated sawdust and rHDPE matrix was observed as shown in Figure 4.11 (d)-(f). This suggested that alkali treatment enhanced the compatibility between sawdust and rHDPE matrix. In addition, sawdust agglomeration was not seen in alkali treated sawdust/rHDPE composites. This result was well corresponded with the mechanical properties.

According to the mechanical properties, flammability and thermal properties, the composite filled with 30 wt% of alkali treated sawdust was selected to investigate the effect of aluminum trihydrate (ATH) as a flame retardant on the properties of the alkali treated sawdust/rHDPE composites.

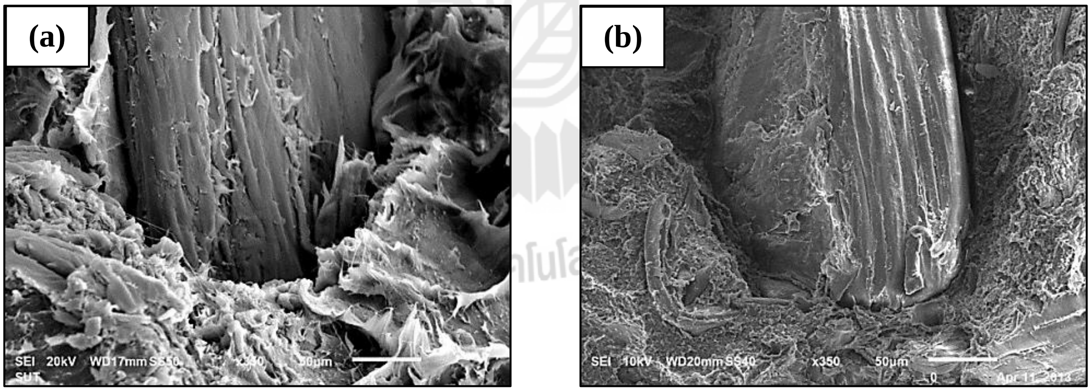


Figure 4.10 SEM micrographs of tensile fracture surface of the sawdust/rHDPE composites at 350x magnification (a) rHDPE/30UT and (b) rHDPE/30AT.

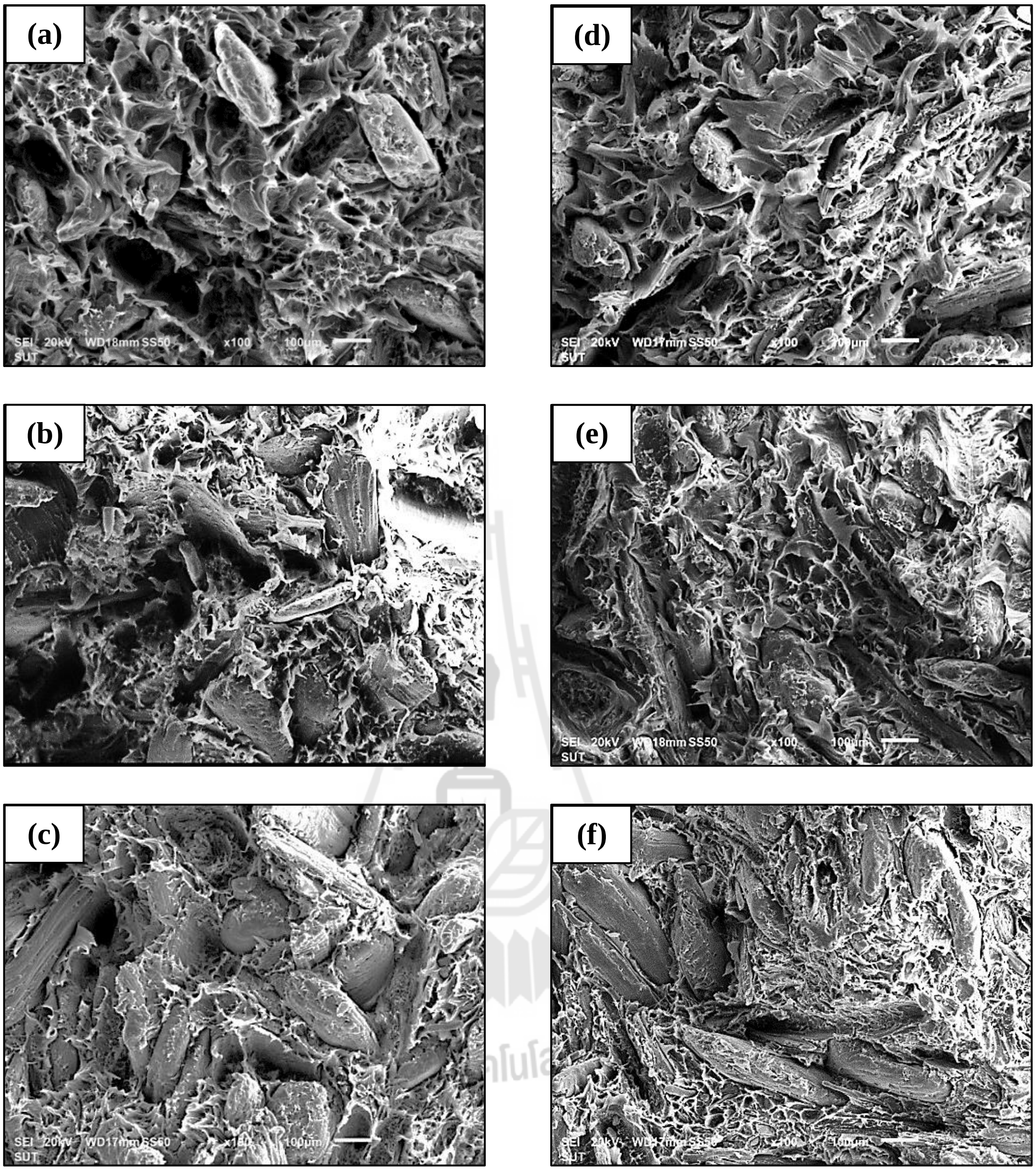
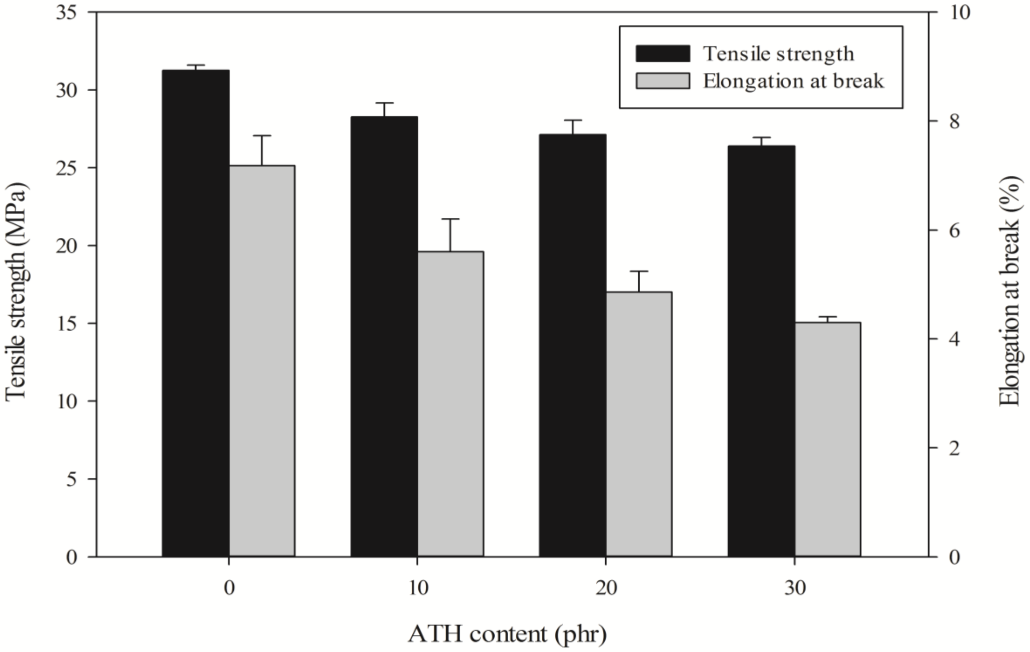


Figure 4.11 SEM micrographs of tensile fracture surface of the sawdust/rHDPE composites at 100x magnification (a) rHDPE/30UT, (b) rHDPE/40UT, (c) rHDPE/50UT, (d) rHDPE/30AT, (e) rHDPE/40AT and (f) rHDPE/50AT.

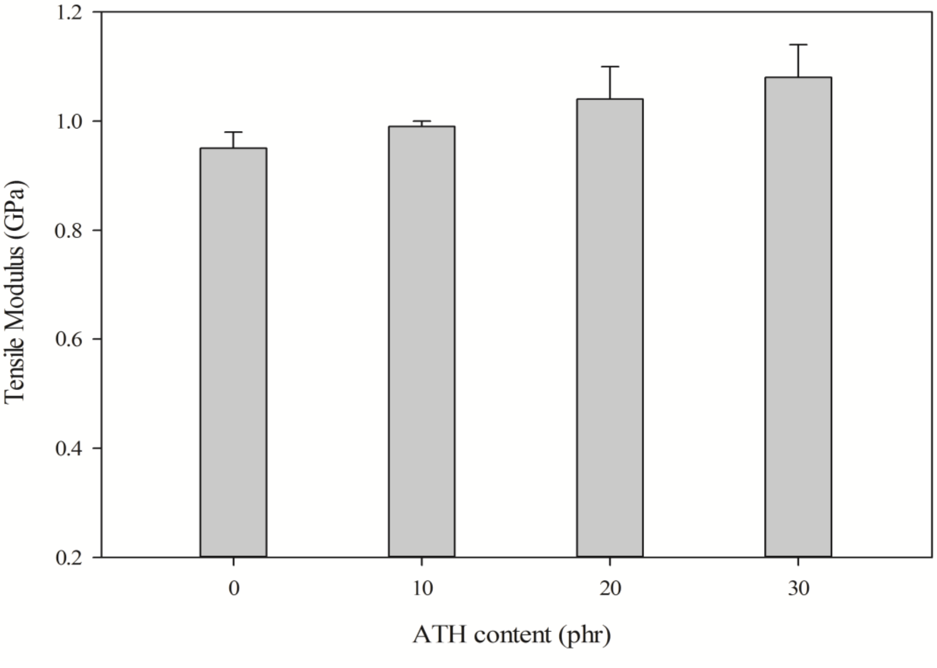
4.3 Effect of aluminum trihydrate (ATH) on physical properties of alkali treated sawdust/rHDPE composites

4.3.1 Mechanical properties

Tensile properties of alkali treated sawdust/rHDPE composites with different ATH contents are listed in Table 4.7. Tensile strength and elongation at break of the composites were continuously decreased with increasing ATH content as shown in Figure 4.12 (a). This was due to poor interfacial adhesion between ATH and rHDPE matrix and agglomeration of ATH in rHDPE matrix confirmed by SEM micrographs of the rHDPE composites as shown in Figures 4.17 (a)-(c). Tensile modulus of the composites improved when ATH content was increased as illustrated in Figure 4.12 (b). ATH was a rigid small particle enhancing the stiffness of the composites (Tai et al., 2000). Ramazani et al. (2008) also reported that tensile strength and elongation at break of polypropylene composites were decreased with increasing ATH content while tensile modulus was increased.



(a)



(b)

Figure 4.12 Tensile properties of alkali treated sawdust/rHDPE composites with different ATH contents (a) tensile strength and elongation at break and (b) tensile modulus.

Flexural properties of alkali treated sawdust/rHDPE composites with different ATH contents are listed in Table 4.7. Flexural strength of the composites slightly decreased with increasing amount of ATH while flexural modulus increased as shown in Figure 4.13 due to the agglomeration and the poor interfacial adhesion between ATH and rHDPE matrix. Shah et al. (2013) also found that with increasing ATH content, flexural strength of polypropylene composites decreased whereas flexural modulus increased.

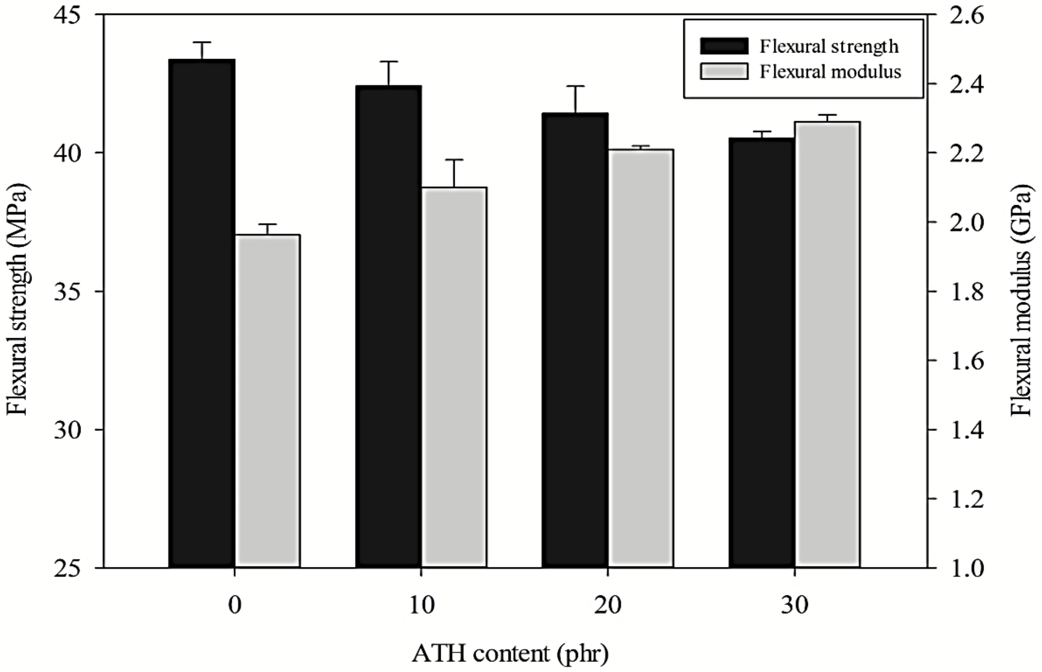


Figure 4.13 Flexural strength and flexural modulus of alkali treated sawdust/rHDPE composites with different ATH contents.

Impact strength of alkali treated sawdust/rHDPE composites with different ATH contents is shown in Figure 4.14. Impact strength of the composites continuously reduced with increasing ATH content. This was due to the poor interfacial adhesion between ATH and rHDPE matrix and the agglomeration of ATH in rHDPE matrix creating more regions of stress concentration. Ramazani et al. (2008) also reported that impact strength of polypropylene (PP) composites decreased with increasing ATH content because of the poor compatibility between ATH and PP matrix.

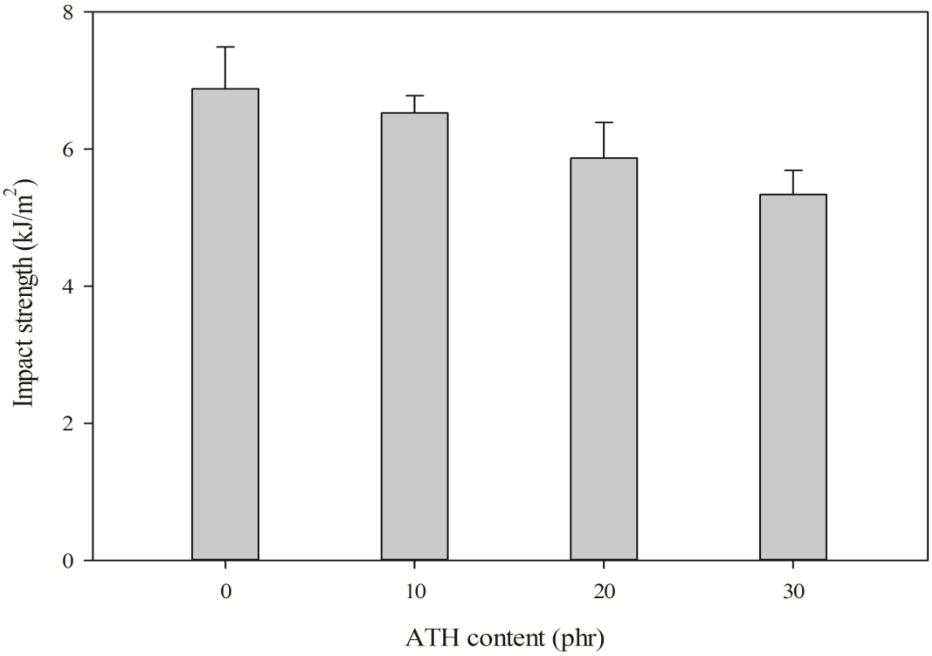


Figure 4.14 Impact strength of alkali treated sawdust/rHDPE composites with different ATH contents.

Table 4.7 Mechanical properties of alkali treated sawdust/rHDPE composites with different ATH contents.

Sample name	**Tensile strength (MPa)**	**Tensile modulus (GPa)**	**Elongation at break (%)**	**Flexural strength (MPa)**	**Flexural modulus (GPa)**	**Impact strength (kJ/m^2)**
rHDPE/30AT	31.24±0.36	0.95±0.03	7.18±0.55	43.88±0.76	1.96±0.03	6.88±0.61
10ATH	28.26±0.90	0.99±0.01	5.60±0.60	42.42±0.87	2.10±0.08	6.53±0.25
20ATH	27.12±0.92	1.04±0.06	4.86±0.38	41.43±0.98	2.21±0.01	5.87±0.52
30ATH	26.39±0.54	1.08±0.06	4.30±0.10	40.54±0.24	2.29±0.02	5.34±0.11

4.3.2 Flammability

UL-94 test results and horizontal burning rate of alkali treated sawdust/rHDPE composites with different ATH contents are shown in Table 4.8. From UL-94 test, alkali treated sawdust/rHDPE composite was combustible materials and was not classified in UL-94 classification. Moreover, the addition of ATH did not have a significant effect on UL-94 test results of the composites at all ATH contents. The composites were also not classified in UL-94 classification. Plentz et al. (2006) found that polypropylene containing with 30-50 wt% ATH could not be classified in UL-94 classification. However, the composite containing 60 wt% ATH was classified as V-2 grade. This indicated that ATH was an effective flame retardant of polymer at a high loading.

Burning rate of alkali treated sawdust/rHDPE composites containing ATH was lower than that of alkali treated sawdust/rHDPE composite without ATH. The burning rate of the composites decreased with increasing ATH content as illustrated in Figure 4.15. This indicated that ATH enhanced the flame retardancy of alkali treated sawdust/rHDPE composites. Norzali et al. (2011) also found that the burning rate of oil palm empty fruit bunch fiber/polyurethane composites with ATH was lowered than the composites without ATH. This was because ATH released some amount of water when ATH was combustible resulting in the dilution of the combustible gases. Moreover, ATH also produced char protective layer on the polymer surface to prevent the polymer from being in contact with heat and oxygen.

Table 4.8 UL-94 classification and horizontal burning rate of alkali treated sawdust/rHDPE composites with different ATH contents.

Sample name	**UL-94 classification**	**Horizontal burning rate (mm/min)**
rHDPE/30AT	NC	18.07±0.77
10ATH	NC	17.88±0.90
20ATH	NC	13.36±1.83
30ATH	NC	11.09±0.84

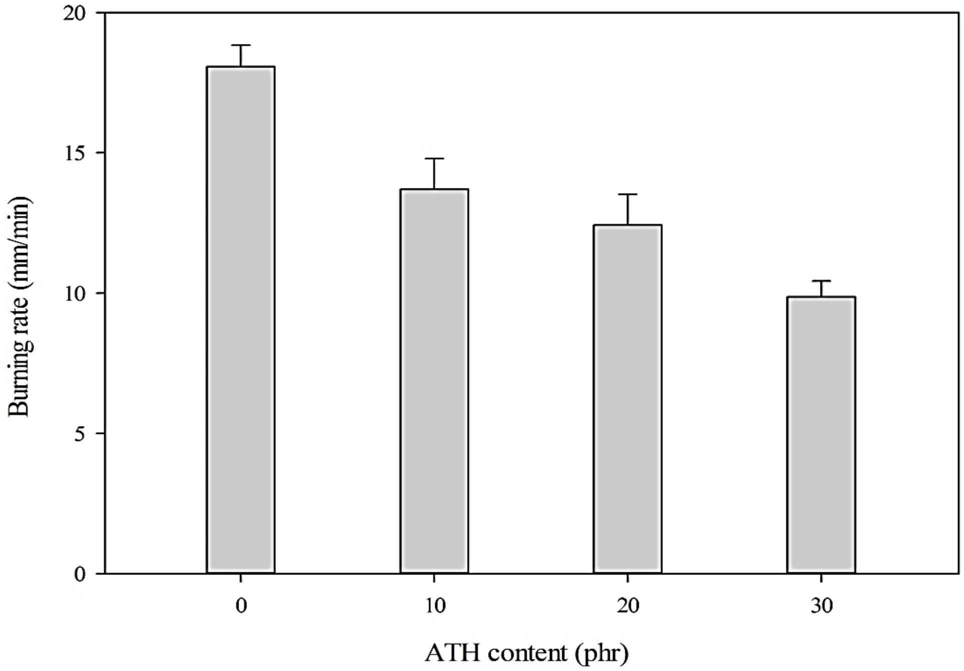


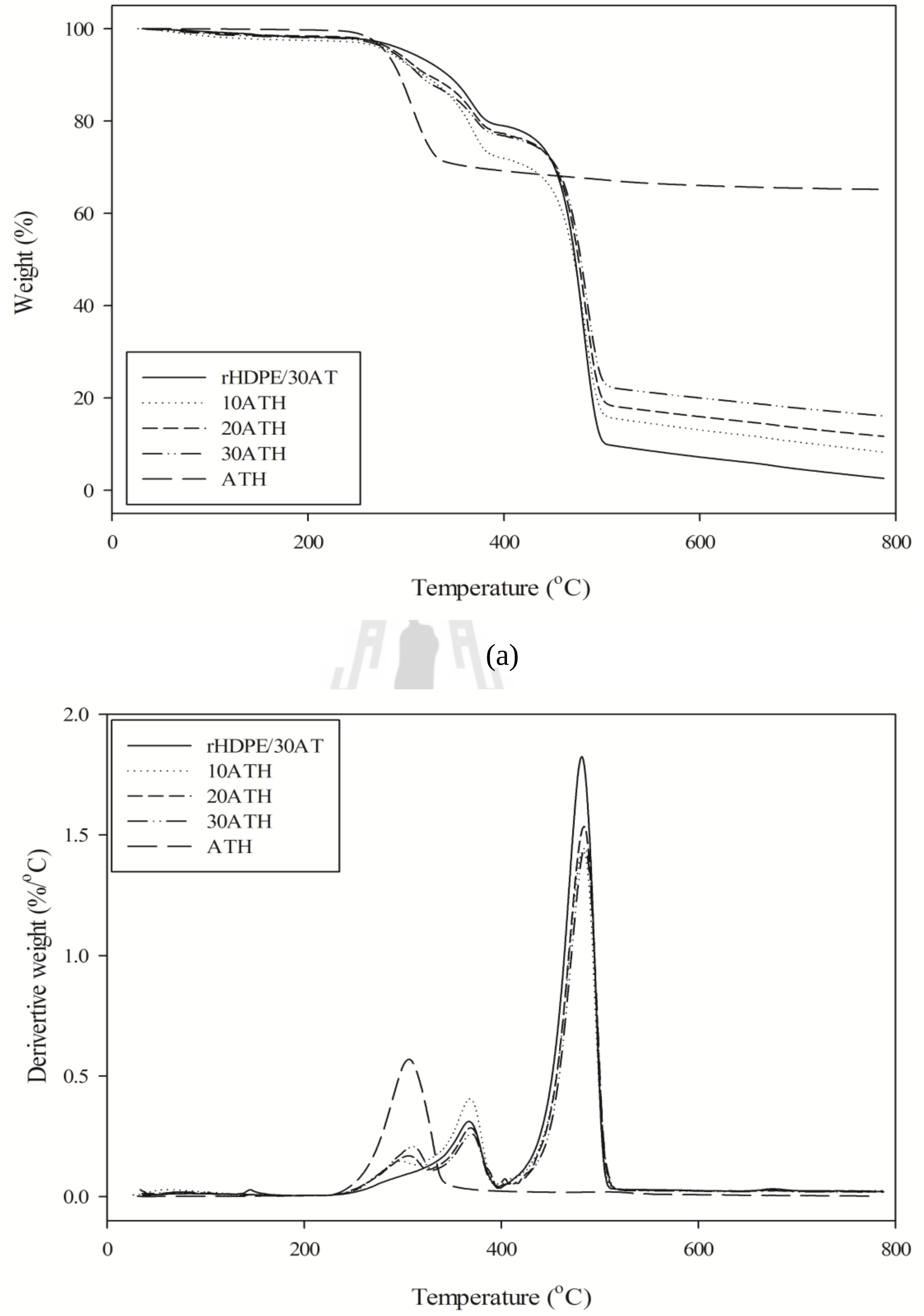
Figure 4.15 Horizontal burning rate of alkali treated sawdust/rHDPE composites with different ATH contents.

4.3.3 Thermal properties

TGA and DTA thermograms of ATH and alkali treated sawdust/rHDPE composites with different ATH contents are shown in Figure 4.16 ATH had only one weight loss step at 291.67°C and left very high amount of char residue around 65.21% at 800°C. During the decomposition process, ATH released water and produced very high amount of char residue (Chen et al., 1989). When ATH at content of 10 phr was added into alkali treated sawdust/rHDPE composites three weight loss steps were found at 292.24, 358.40 and 477.01°C due to the decomposition of ATH, sawdust and rHDPE, respectively. The 2nd and 3rd decomposition temperature of the composites slightly increased with increasing ATH content because the release of water and the formation of char residue during the thermal decomposition of ATH delayed thermal decomposition of the composites by acting as an insulating protective layer (Ramazani et al., 2008). In addition, the char residue continuously increased with increasing ATH content.

Table 4.9 Thermal decomposition characteristics of ATH and alkali treated sawdust/rHDPE composites with different ATH contents.

Sample name	1st weight loss temperature (°C)	2nd weight loss temperature (°C)	3rd weight loss temperature (°C)	Residue (%)
rHDPE/30AT	-	353.83	476.33	6.71
10ATH	292.24	358.40	477.01	8.31
20ATH	292.32	359.57	477.90	11.70
30ATH	292.03	360.40	478.66	15.07
ATH	291.67	-	-	65.21



(a)

(b)

Figure 4.16 Thermal decomposition characteristics of ATH and alkali treated sawdust/rHDPE composites with different ATH contents (a) TGA and (b) DTA.

4.3.4 Morphological properties

SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with different ATH contents are shown in Figure 4.17 (a)-(c). The gap between ATH and rHDPE matrix was clearly observed in Figure 4.17 (a) indicating poor compatibility between rHDPE and ATH. Moreover, the agglomeration of ATH was found when ATH content was increased as shown in Figure 4.17 (b)-(c). This result corresponded well with the mechanical properties of the composites.

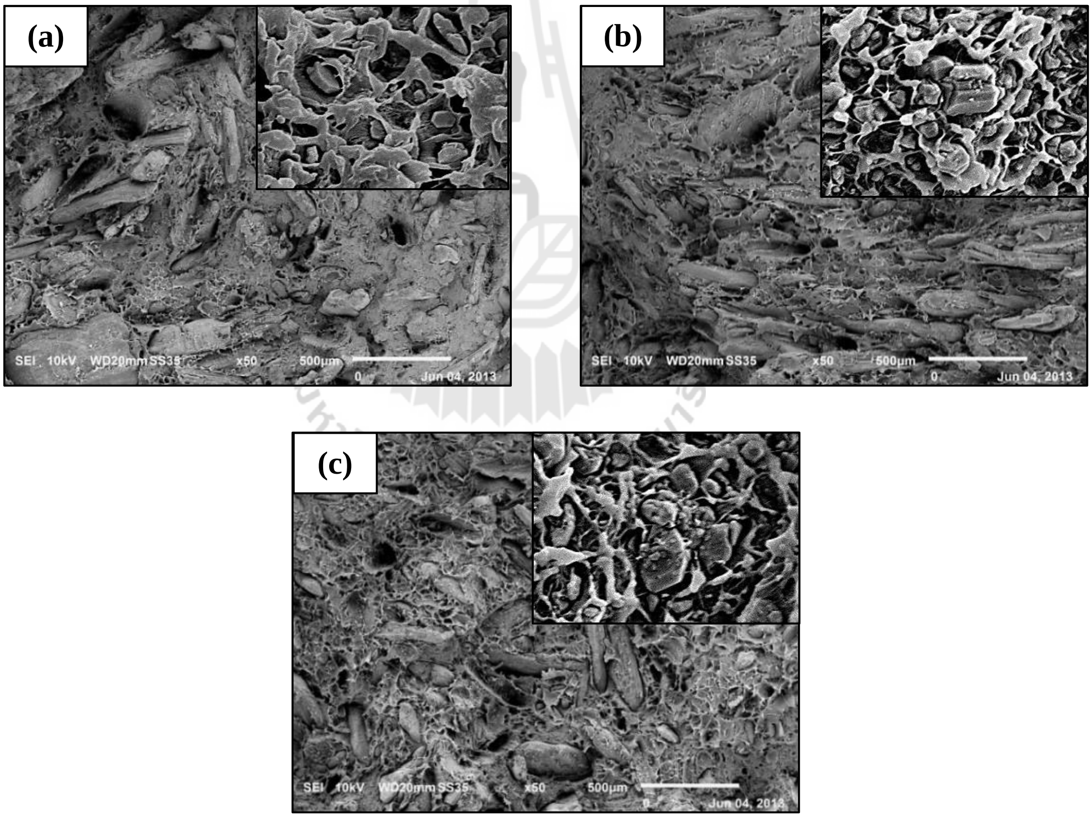


Figure 4.17 SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with different ATH contents at magnification 50x (a) 10ATH, (b) 20ATH and (c) 30ATH.

From the mechanical properties, flammability and thermal properties, the composite containing 30ATH was chosen to study the effect of combination of flame retardants on properties of alkali treated sawdust/rHDPE composites.

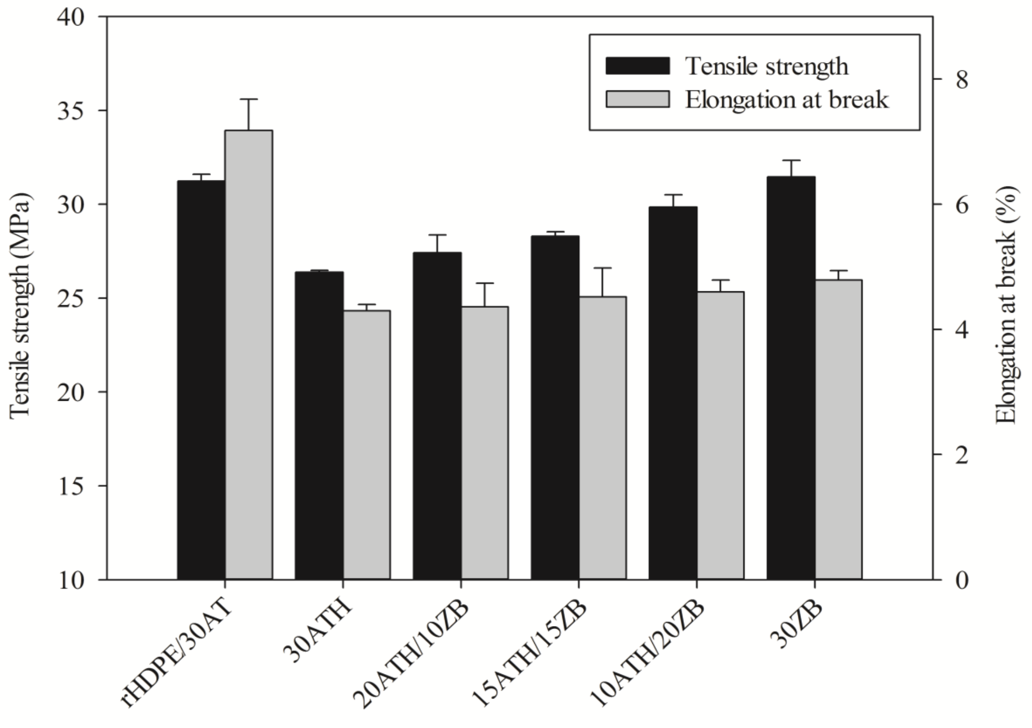
4.4 Effect of the combination of flame retardants on physical properties of alkali treated sawdust/rHDPE composites

Flame retardants are generally used to improve the flame retardancy of lignicellulosic fillers/polymer composites. However, the addition of flame retardant for enhancing flame retardancy of the composites causes the reduction of mechanical properties. The effectiveness of flame retardants can be improved by synergistic effect. The combination of flame retardants shows the enhancement of the flame retardantcy of wood fillers/polymer composites without the deterioration of mechanical properties (Garcia et al., 2009; Kurt and Mengeloglu, 2009; Bai et al., 2014).

4.4.1 Mechanical properties

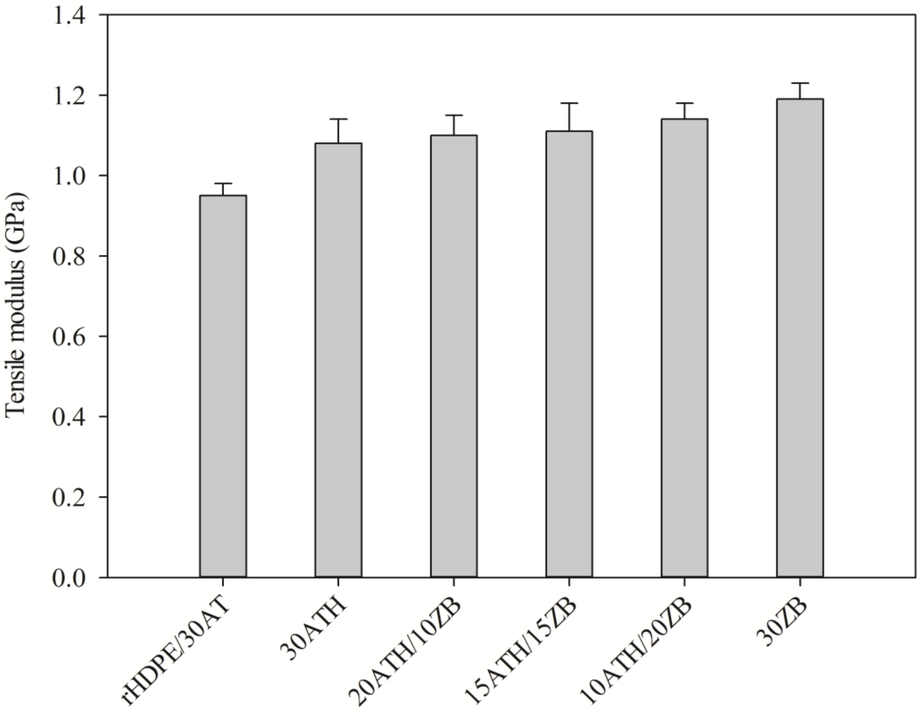
Tensile properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants are presented in Table 4.10. With the incorporation of ATH into the alkali treated sawdust/rHDPE composites, tensile strength and elongation at break of the composites were decreased as illustrated in Figure 4.18 (a). This was due to poor interfacial adhesion between ATH and rHDPE matrix (Wang et al., 2002). On the other hand, tensile modulus of the alkali treated sawdust/rHDPE composites was increased when ATH was added as shown in Figure 4.18 (b). This was because ATH was a rigid particle leading to enhanced stiffness of the composites (Tai et al., 2000).

The incorporation of the combination of ATH and ZB, i.e. 20ATH/10ZB, 15ATH/15ZB and 10ATH/20ZB into the alkali treated sawdust/rHDPE composites slightly increased tensile properties of the composites comparing to the composite containing ATH. Moreover, tensile properties of the composites increased with increasing ZB content in the mixture of ATH and ZB. The composite containing ZB exhitbited the highest tensile properties. This was because ZB had rougher surface than ATH as displayed in Figure 4.23. This may promote mechanical interlocking between ZB and polymer matrix. (Jeecham et al., 2010). Ramazani et al. (2007) also reported that the addition of combination of ATH and ZB into polypropylene composites significantly increased tensile properties of the composites containing ATH.



(a)

Figure 4.18 Tensile properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants (a) tensile strength and elongation at break and (b) tensile modulus.



(b)

Figure 4.18 Tensile properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants (a) tensile strength and elongation at break and (b) tensile modulus (continued).

Flexural properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants are shown in Figure 4.19. Flexural strength of alkali treated sawdust/rHDPE composites decreased with the incorporation of ATH while flexural modulus increased. With the addition of the combination ATH and ZB into the alkali treated sawdust/rHDPE composites, flexural strength and flexural modulus were slightly improved when compared to the composite containing ATH. Flexural properties of the composites containing the combination of ATH and ZB were increased with increasing ZB content in the mixture of ATH and ZB. The composite containing 30ZB composite exhibited the highest flexural properties.

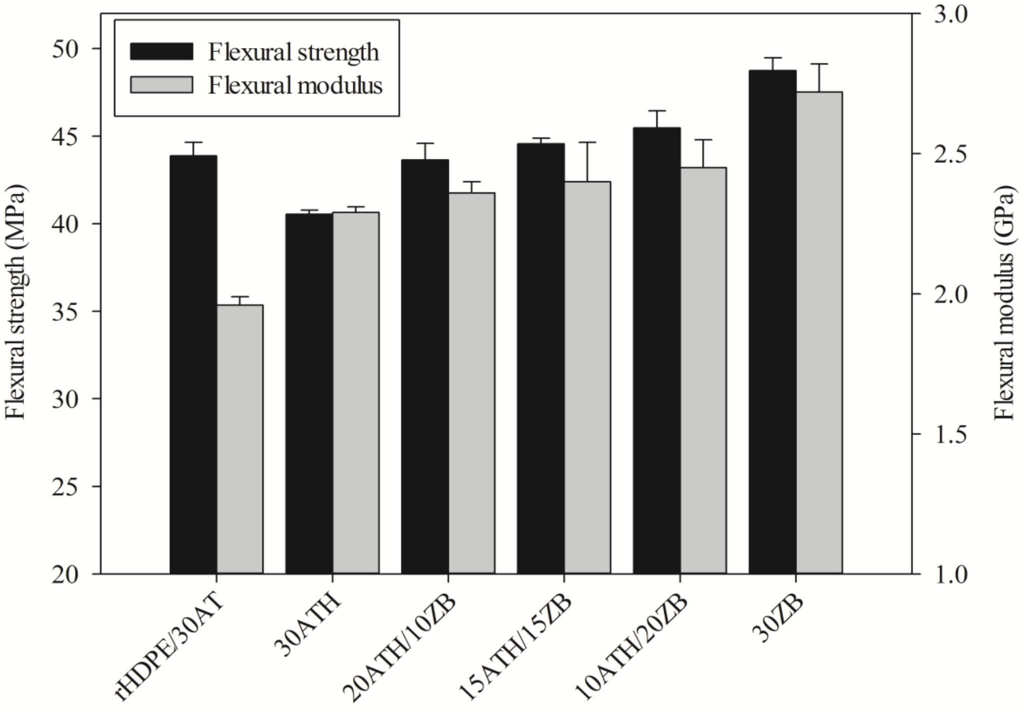


Figure 4.19 Flexural properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Impact strength of alkali treated sawdust/rHDPE composites with the combination of flame retardants is shown in Figure 4.20. The reduction of impact strength of the alkali treated sawdust/rHDPE composites was found when flame retardants were added. Moreover, insignificant difference in impact strength of the composites was observed with the addition of ATH, ZB or the combination of ATH and ZB.

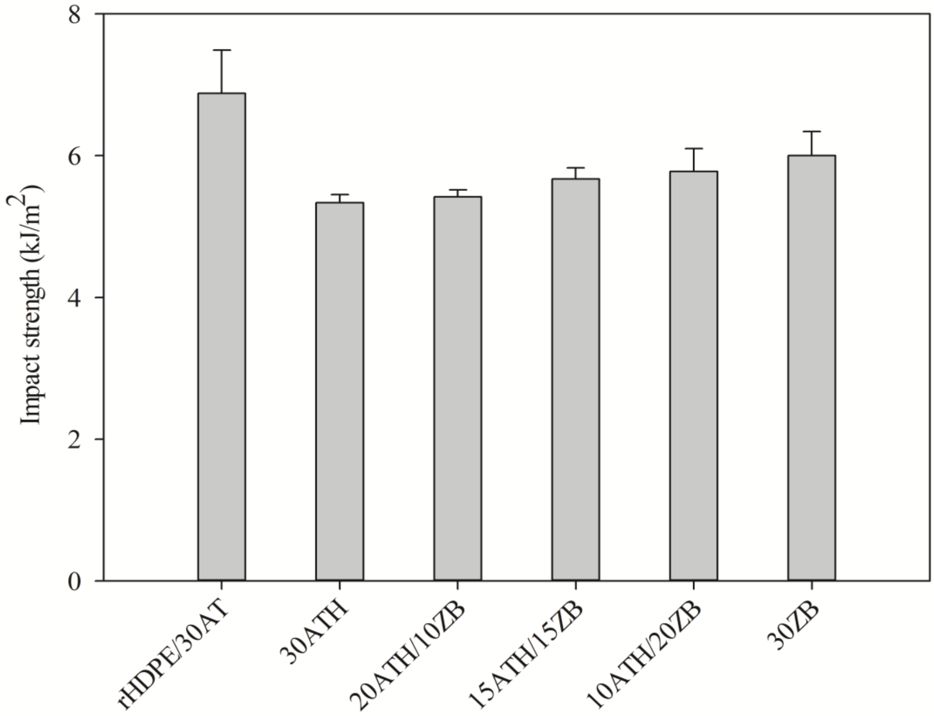


Figure 4.20 Impact strength of alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Table 4.10 Mechanical properties of alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Sample name	**Tensile strength (MPa)**	**Tensile modulus (GPa)**	**Elongation at break (%)**	**Flexural strength (MPa)**	**Flexural modulus (GPa)**	**Impact strength (kJ/m^2)**
rHDPE/30AT	31.24±0.36	0.95±0.03	7.18±0.55	43.88±0.76	1.96.±0.03	6.88±0.61
30ATH	26.39±0.54	1.08±0.06	4.30±0.10	40.54±0.24	2.29±0.02	5.34±0.11
20ATH/10ZB	27.41±0.96	1.10±0.05	4.36±0.38	43.64±0.95	2.36±0.04	5.42±0.10
15ATH/15ZB	28.29±0.24	1.11±0.07	4.49±0.46	44.56±0.32	2.40±0.14	5.67±0.16
10ATH/20ZB	29.85±0.66	1.14±0.04	4.56±0.19	45.47±0.98	2.45±0.10	5.78±0.32
30ZB	31.45±0.89	1.19±0.04	4.79±0.15	48.74±0.73	2.72±0.10	6.00±0.34

4.4.2 Flammability

UL-94 test results and horizontal burning rate of alkali treated sawdust/rHDPE composites with the combination of flame retardants are shown in Table 4.11. From UL-94 test, all of composites were not classified in UL-94 classification.

Adding the combination of ATH and ZB at 20ATH/10ZB and 15ATH/15ZB into the composites resulted in a decrease in burning rate when compared to the composite containing 30ATH as shown in Figure 4.21. ATH decomposed at the temperature range of 220-240°C. During the decomposition process of ATH, water was released and thermal insulating protective layer was formed on the polymer surface separating its form heat and oxygen. At the temperature range of 290-450°C, zinc borate was decomposed. Water was released and char protective layer was formed which separated the composites from heat and oxygen (Formicola et al., 2009). Ibibiko (2013) also reported that the combination of ATH and ZB showed synergistic effect in enhancing flame retardancy of LDPE composites.

However, the composite containing 10ATH/20ZB had higher burning rate that the composites containing 20ATH/10ZB and 15ATH/15ZB. Ibibiko (2013) also reported that flame retardancy of the LDPE composites was slightly reduced when ZB content in the mixture of ATH and ZB was increased. In addition, the composite containing 30ZB exhibited the highest burning rate. This suggested that ZB was poor flame retardant for the composites.

From the results, it can be seen that the combination of ATH and ZB showed synergistic effect on the flame retardancy of alkali treated sawdust/rHDPE composites at ATH:ZB ratio of 2:1 and 1:1. Moreover, the composite containing 20ATH/10ZB showed the lowest burning rate.

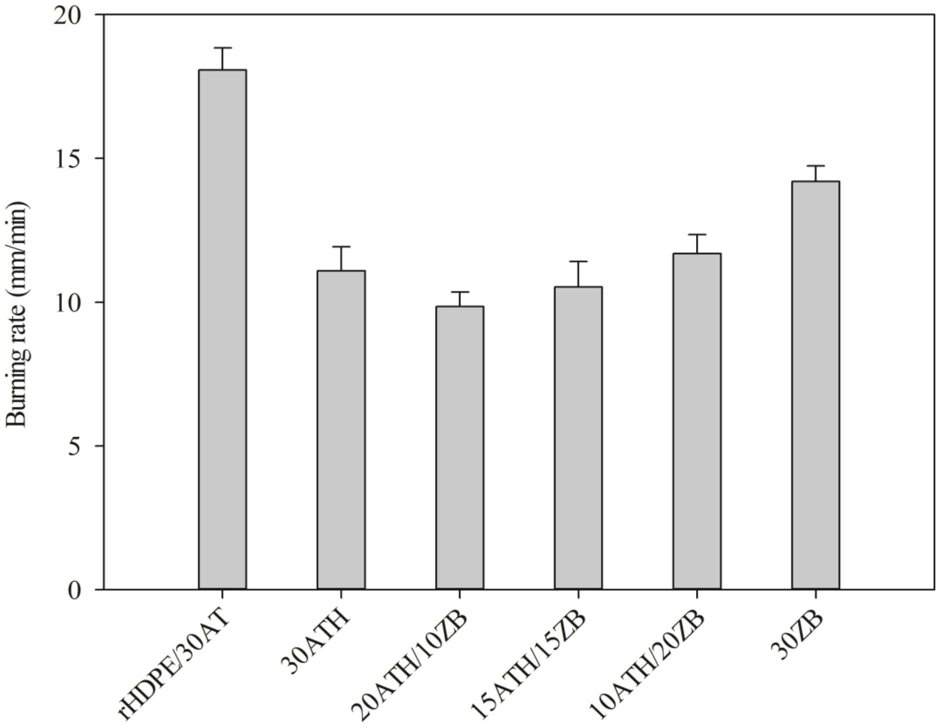


Figure 4.21 Horizontal burning rate of alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Table 4.11 UL-94 classification and horizontal burning rate of alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Sample name	**UL-94 classification**	**Horizontal burning rate (mm/min)**
rHDPE/30AT	NC	18.07±0.77
30ATH	NC	11.09±0.84
20ATH/10ZB	NC	9.85±0.50
15ATH/15ZB	NC	10.53±0.88
10ATH/20ZB	NC	11.69±0.66
30ZB	NC	14.20±0.54

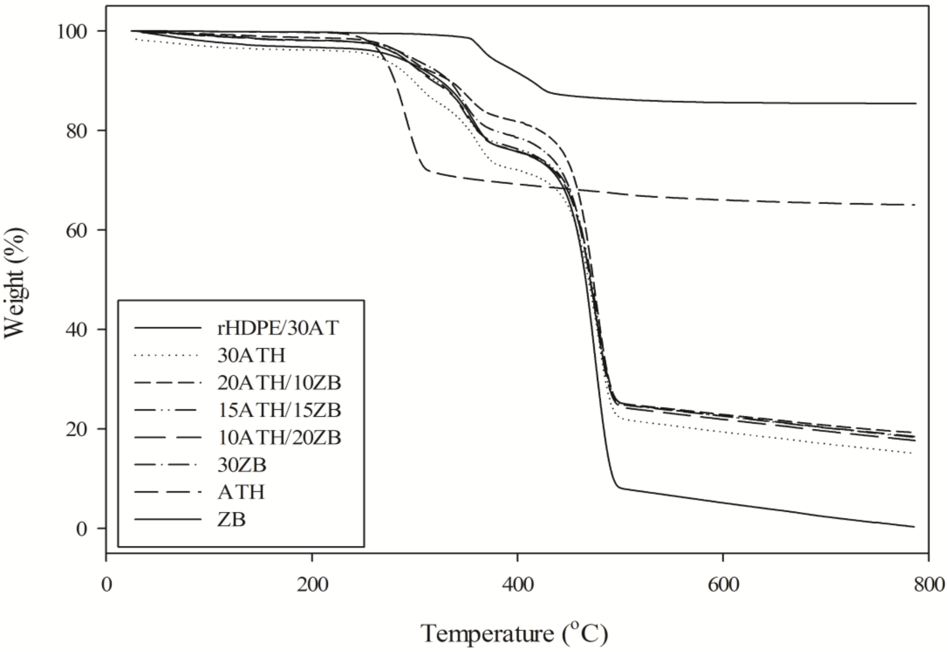
4.4.3 Thermal properties

TGA and DTA thermograms of alkali treated sawdust/rHDPE composites with the combination of flame retardants are shown in Figure 4.22 (a)-(b). rHDPE/30AT composite showed two weight loss steps due to the decomposition of sawdust and rHDPE. With the addition of ATH, the composites exhibited three weight loss steps due to the decomposition of ATH, sawdust and rHDPE. The composite containing ZB had two weight loss steps because the thermal decomposition temperature of ZB was overlapped with alkali treated sawdust. ZB was decomposed in the temperature range of 366.64-421.00°C. During the decomposition of ZB, water was released and char residue was produced at about 85% as shown in Figure 4.22 (a).

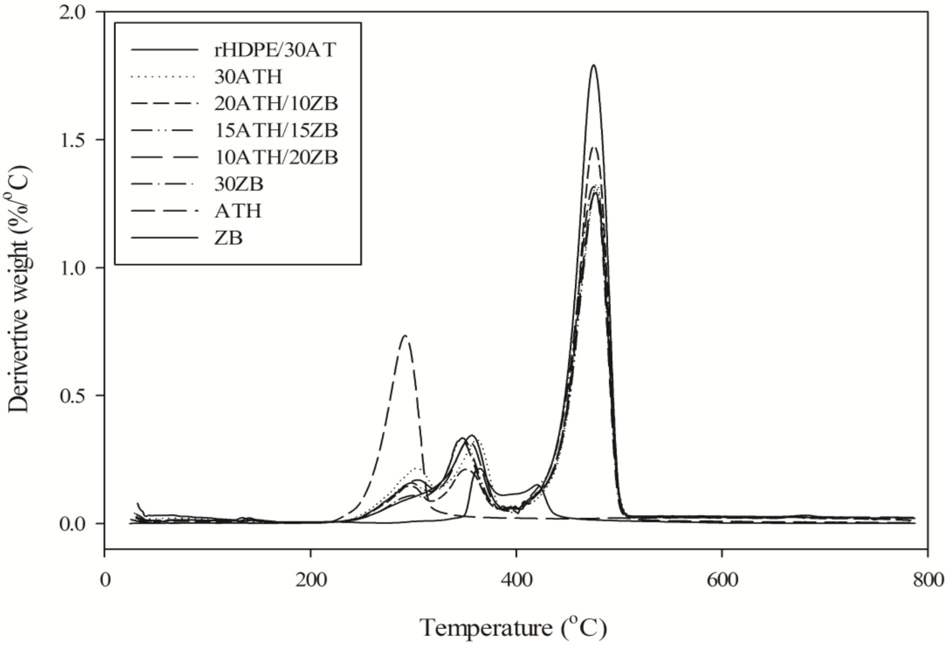
When certain content of ATH was replaced with 10 phr of ZB, the thermal decomposition temperatures of composite were slightly increased when compared to the composite containing ATH as shown in Table 4.12. In addition, the decomposition temperatures of the composites were increased with increasing ZB content in the mixture of ATH and ZB. ATH released water and produced char protective layer during the decomposition process resulting in the retardation of thermal decomposition process. ZB was decomposed at about 360°C and released water. Moreover, char protective layer was formed which resulted in more effective char protective layer suppressing thermal decomposition process (Formicola et al., 2009). The composites containing the combination of ATH and ZB exhibited higher char residue than the composite with ATH. However, char residue of the composites was insignificantly changed with increasing ZB content in the mixture of ATH and ZB.

Table 4.12 Thermal decomposition characteristics of ATH, ZB and alkali treated sawdust/rHDPE composites with the combination of flame retardants.

Sample name	**1st weight loss temperature (°C)**	**2nd weight loss temperature (°C)**	**3rd weight loss temperature (°C)**	**Residue (%)**
rHDPE/30AT	-	353.83	476.33	6.71
30ATH	292.03	360.40	478.66	15.07
20ATH/10ZB	293.62	360.95	479.23	17.25
15ATH/15ZB	294.16	363.95	479.80	17.63
10ATH/20ZB	297.58	364.16	480.07	18.28
30ZB	-	365.14	480.52	18.46
ATH	291.67	-	-	65.21
ZB	-	366.64	421.00	85.40



(a)



(b)

Figure 4.22 Thermal decomposition characteristics of ATH, ZB and alkali treated sawdust/rHDPE composites with the combination of flame retardants (a) TGA and (b) DTA.

4.4.4 Morphological properties

SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with the combination of flame retardants are shown Figure 4.23 (a)-(e). From Figure 4.23 (a), gap between ATH and rHDPE matrix was observed due to the poor interfacial adhesion between ATH and rHDPE matrix. Howover, from Figure 4.23 (b)-(e), it can be seen that ZB had better adhesion with rHDPE matrix than ATH. This was corresponded well with mechanical properties that the addition of ZB into the composites containing ATH enhanced the mechanical properties of the composites.

In this section, the combination of flame retardants showed the improvement of mechanical properties and thermal stability of the composites. The synergistic effect in enhancing flame retardancy of the composites was observed when 20ATH/10ZB and 15ATH/15ZB was added. The composite filled with 20ATH/10ZB showed the highest flame retardancy. Therefore, this composite was chosen to study the effect of compatibilizer on the properties of sawdust/rHDPE composites containing flame retardant.

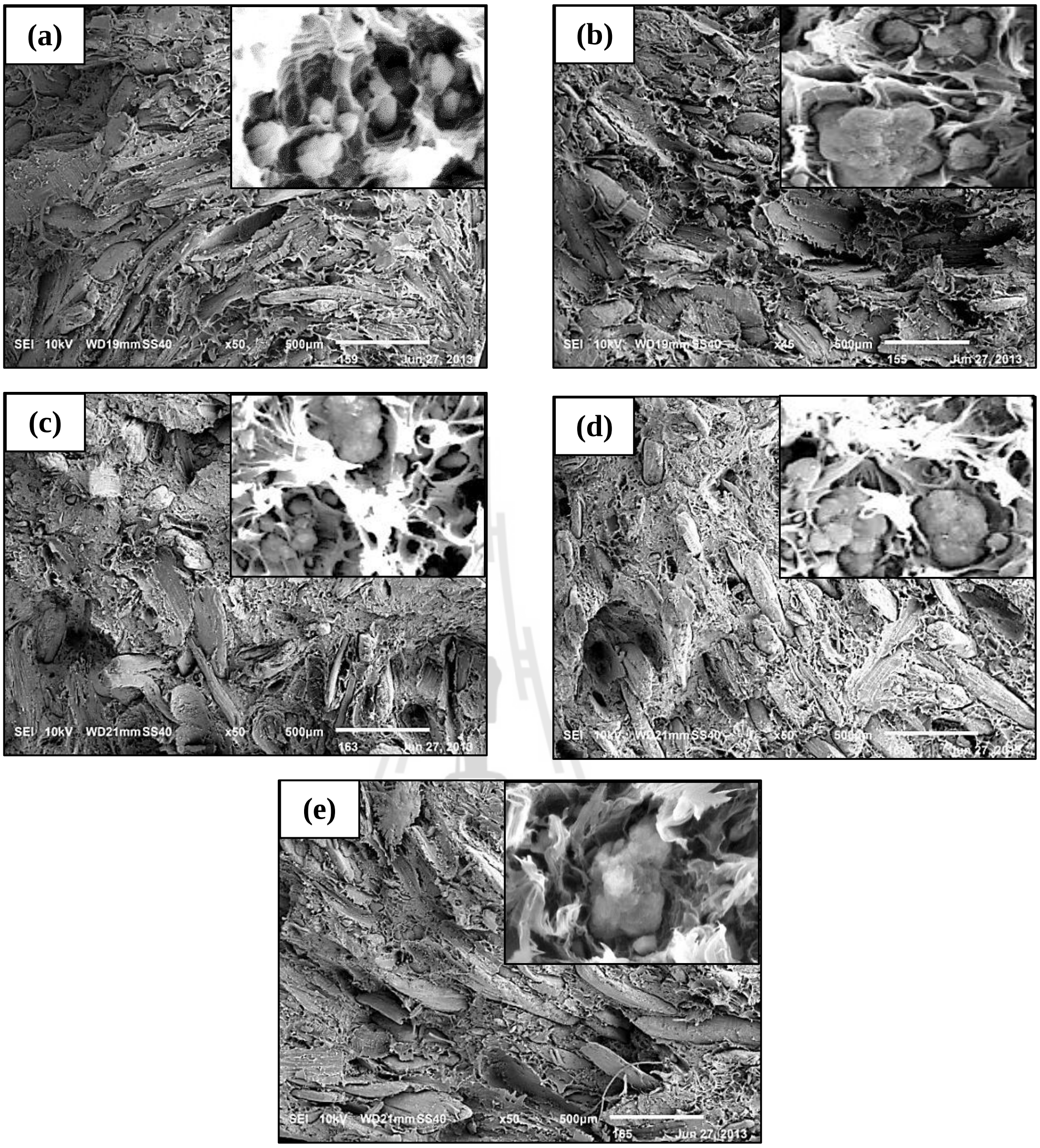


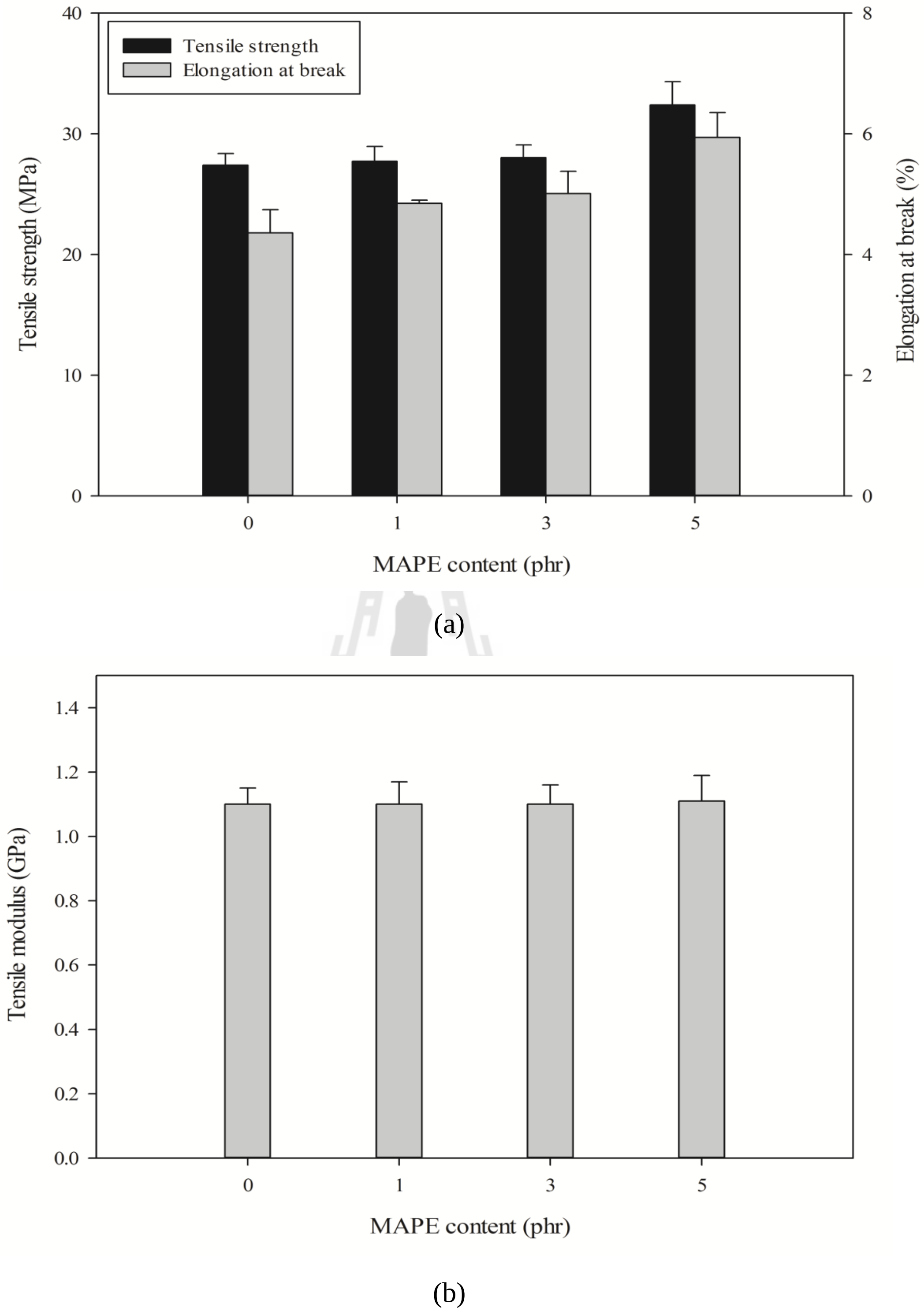
Figure 4.23 SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with the combination of flame retardants at magnification 50x (a) 30ATH, (b) 20ATH/10ZB, (c) 15ATH/15ZB, (d) 10ATH/20ZB and (e) 30ZB.

4.5 Effect of compatibilizer on physical properties of alkali treated sawdust/rHDPE composites containing flame retardants

4.5.1 Mechanical properties

Tensile properties of the composites containing 20ATH/10ZB at various maleic anhydride grafted polyethylene (MAPE) contents are illustrated in Table 4.13. From Figure 4.24 (a)-(b), tensile strength and elongation at break of the composites increased with increasing MAPE content while tensile modulus was relatively unchanged. This may be due to the improvement of interfacial adhesion between flame retardants and rHDPE matrix. The chemical bond was formed between OH groups of the flame retardants and anhydride groups of MAPE while PE chains of MAPE diffused into the PE matrix. Therefore, the surface adhesion between flame retardants and rHDPE can be improved by use of MAPE (Ayrilmis, 2013; Ridzuan et al., 2013). Moreover, the addition of MAPE can also improve the compatibility between sawdust and rHDPE matrix. The polar part of MAPE interacted with hydroxyl groups of sawdust and MAPE anchors its nonpolar PE part to the rHDPE matrix (Tai and Li, 2001) resulting in the improvement of tensile properties.

Flexural properties of the composites containing 20ATH/10ZB at various MAPE contents are illustrated in Figure 4.25 and listed in Table 4.13. Flexural strength of the composites was increased with increasing MAPE content whereas flexural modulus was insignificantly changed. Jeencham (2010) also observed that flexural strength of sisal fiber/PP composites containing ammonium polyphosphate (APP) increased with increasing MAPP content.



(a)

(b)

Figure 4.24 Tensile properties of alkali treated sawdust/rHDPE composites with different MAPE contents (a) tensile strength and elongation at break (b) tensile modulus.

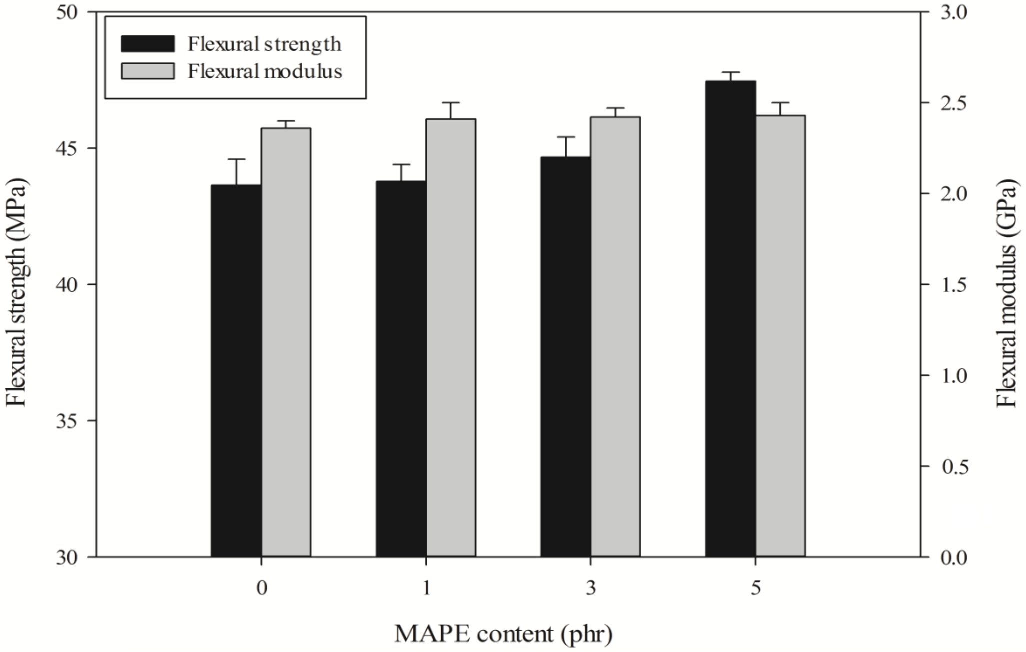


Figure 4.25 Flexural properties of alkali treated sawdust/rHDPE composites with different MAPE contents.

Impact strength of the composites containing 20ATH/10ZB at various MAPE contents is shown in Figure 4.26. Impact strength of the composites was continuously enhanced when MAPE content was increased because of the improvement of the interfacial adhesion between fillers and rHDPE resulting in more efficient stress transfer from the polymer matrix to the fillers. Therefore, the composites were able to absorb higher amount of energy to stop crack propagation. (Yang et al., 2007; Ayrilmis, 2013; Ridzuan et al., 2013).

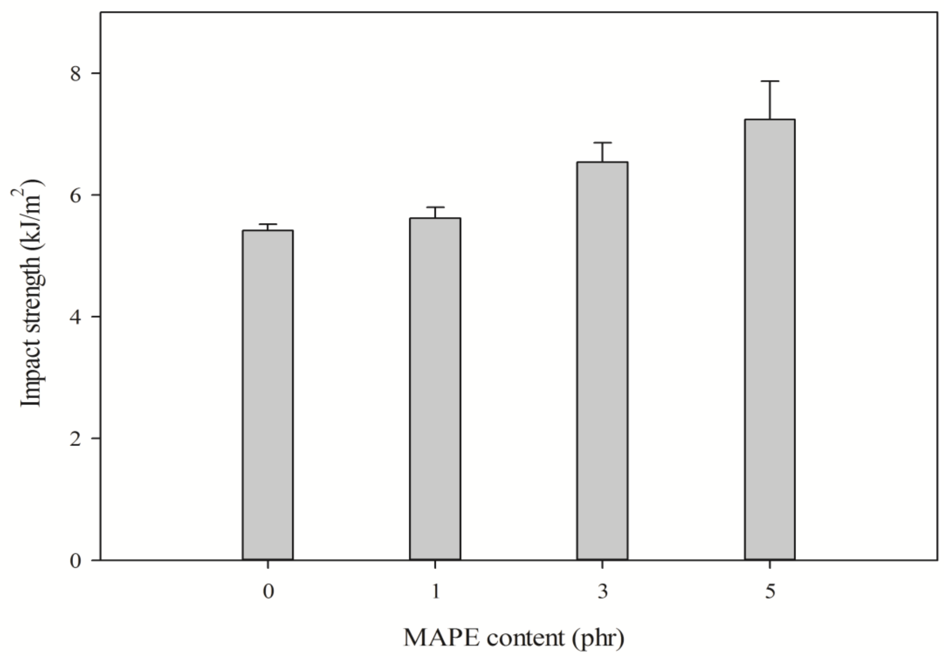


Figure 4.26 Impact strength of alkali treated sawdust/rHDPE composites with different MAPE contents.

Table 4.13 Mechanical properties of alkali treated sawdust/rHDPE composites with different MAPE contents.

Sample name	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (kJ/m^2)
20ATH/10ZB	27.41±0.96	1.10±0.05	4.36±0.38	43.64±0.95	2.36±0.04	5.42±0.10
1MAPE	27.72±1.23	1.10±0.07	4.85±0.05	43.78±0.62	2.41±0.09	5.62±0.18
3MAPE	28.02±1.06	1.10±0.06	5.01±0.37	44.67±0.74	2.42±0.05	6.54±0.32
5MAPE	32.39±1.93	1.11±0.08	5.94±0.41	47.45±0.34	2.43±0.07	7.24±0.63

4.5.2 Flammability

UL-94 test results and horizontal burning rate of the composites containing 20ATH/10ZB at various MAPE contents are shown in Table 4.14. It can be seen that all of the composites were not classified in UL-94 classification.

On the other hand, the burning rate of the composite containing 20ATH/10ZB was reduced with increasing MAPE content as illustrated in Figure 4.27 due to good distribution of flame retardants in rHDPE matrix (Zhang et al., 2005). This can be confirmed by SEM micrographs (Figure 4.29) that flame retardants were well distributed in rHPDE matrix with the addition of MAPE. Li and He, (2003) also reported that flame retardancy of wood fibers/LLDPE composite containing APP was improved with increasing MAPE content.

Table 4.14 UL-94 classification and horizontal burning rate of alkali treated sawdust/rHDPE composites with different MAPE contents.

Sample name	**UL-94 classification**	**Horizontal burning rate (mm/min)**
20ATH/10ZB	NC	9.85±0.50
1MAPE	NC	8.94±0.56
3MAPE	NC	8.52±0.78
5MAPE	NC	8.37±0.65

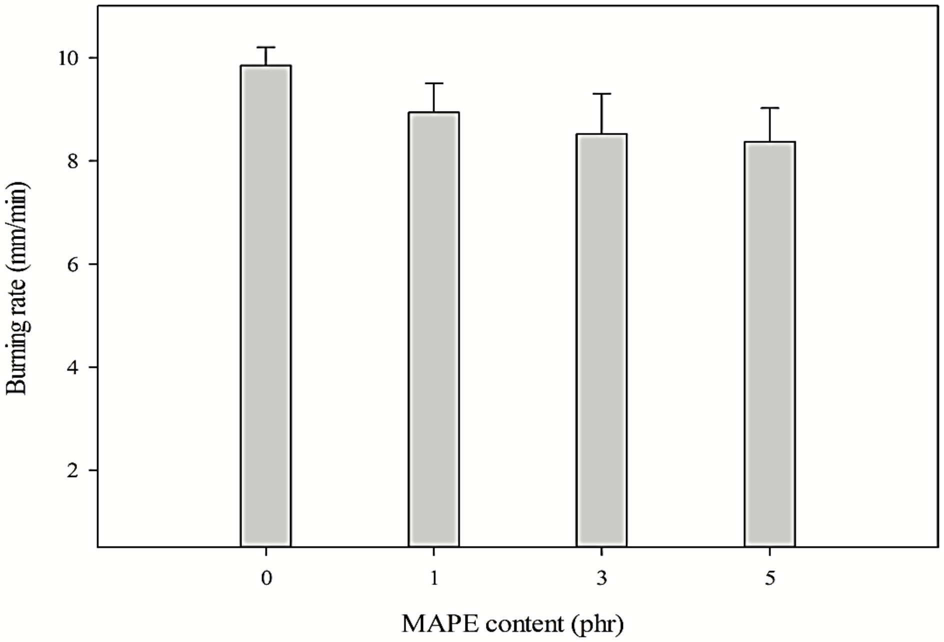


Figure 4.27 Horizontal burning rate of alkali treated sawdust/rHDPE composites with different MAPE contents.

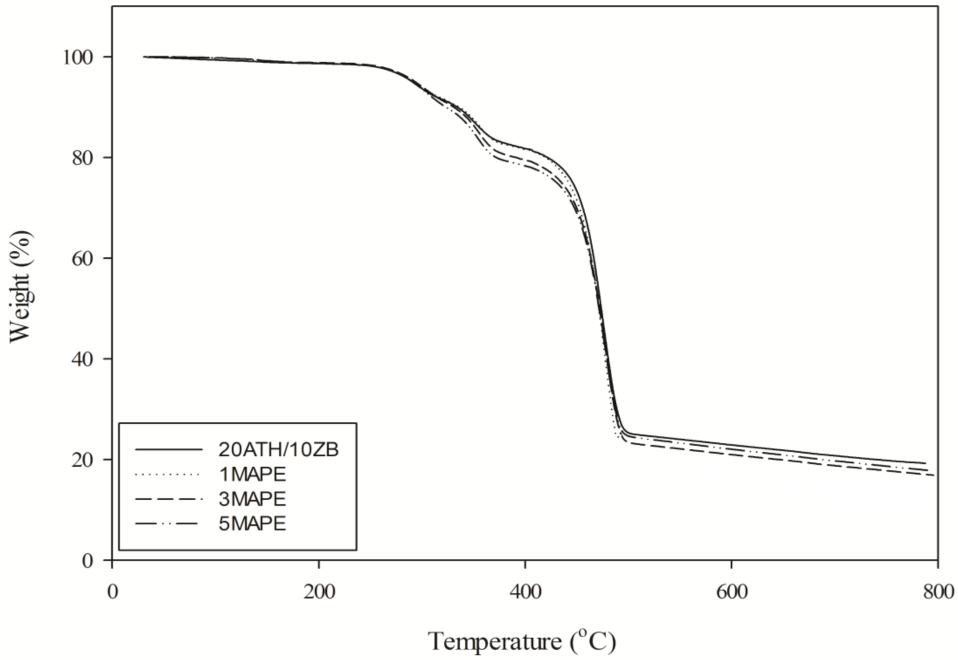
4.5.3 Thermal properties

TGA and DTA thermograms of the composites containing 20ATH/10ZB at various MAPE contents are shown in Figure 4.28 (a)-(b). The composite containing 20ATH/10ZB exhibited three weight loss steps. The 1st decomposition temperature was due to the decomposition of ATH. The 2nd decomposition temperature was assigned to the decomposition of alkali treated sawdust and ZB. The 3rd decomposition temperature was attributed to the decomposition of rHDPE matrix. The 1st, 2nd and 3rd thermal decomposition temperature of the composites slightly increased with the incorporation of MAPE into the composites due to the improvement of interfacial adhesion between flame retardants and rHDPE matrix. Ridzuan et al. (2013) also found that thermal stability of oil palm empty fruit bunch fiber/PP composites containing flame retardant increased with the addition of MAPP. Li and He (2003) also reported that thermal stability of wood fiber/LLDPE composite containing APP was enhanced with the incorporation of MAPE.

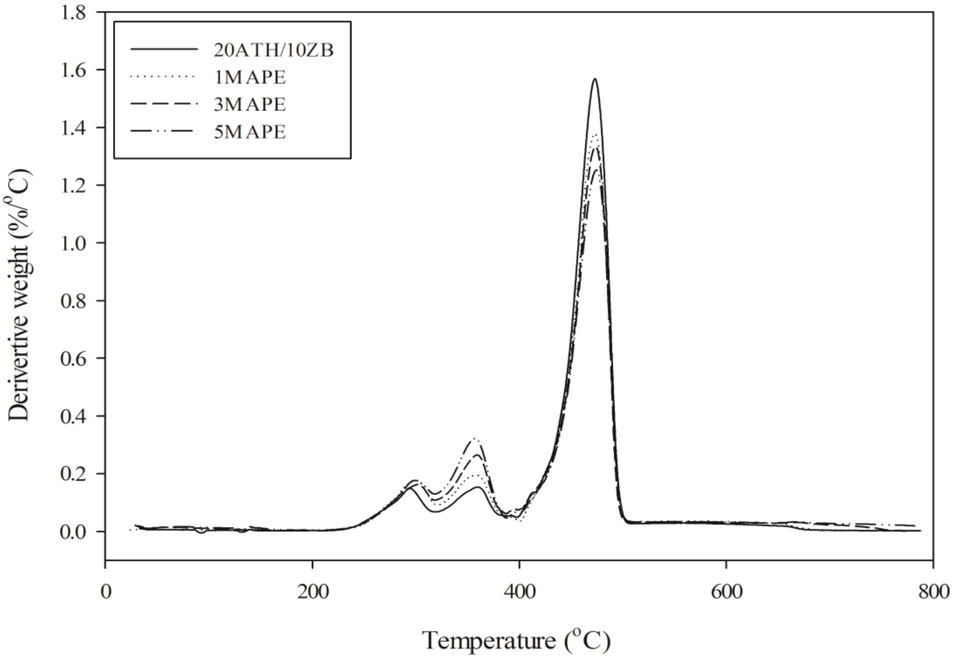
In addition, the insignificant change of 1st, 2nd and 3rd thermal decomposition temperature of the composites was observed with increasing MAPE content. Jeencham (2010) also observed the insignificant change in thermal stability of sisal fibers/PP composites containing APP with increasing MAPP content. Moreover, char residue observed at 800ºC of the composites was relatively unchanged with increasing MAPE content.

Table 4.15 Thermal decomposition characteristic of alkali treated sawdust/rHDPE composites with different MAPE contents.

Sample name	**1st weight loss temperature (°C)**	**2nd weight loss temperature (°C)**	**3rd weight loss temperature (°C)**	**Residue (%)**
20ATH/10ZB	293.62	360.95	479.23	17.25
1MAPE	295.52	361.12	480.08	16.54
3MAPE	296.03	361.79	480.78	15.64
5MAPE	296.73	362.86	481.33	15.53



(a)



(b)

Figure 4.28 Thermal decomposition characteristics of alkali treated sawdust/rHDPE composites with different MAPE contents (a) TGA and (b) DTA.

4.5.4 Morphological properties

SEM micrographs of tensile fracture surface of the composites containing 20ATH/10ZB at various MAPE contents are shown in Figure 4.29. Poor interfacial adhesion between flame retardants and rHDPE matrix was observed in Figure 4.29 (a). However, the addition of MAPE enhanced the interfacial adhesion between flame retardants and rHDPE matrix as illustrated in Figure 4.29 (b)-(d). Flame retardants also well distributed in rHDPE matrix. This was corresponded well with mechanical properties of the composites. In addition, the improvement of interfacial adhesion between sawdust and rHDPE matrix was also observed when MAPE content added in to the composites as illustrated in Figure 4.30 (a)-(d).

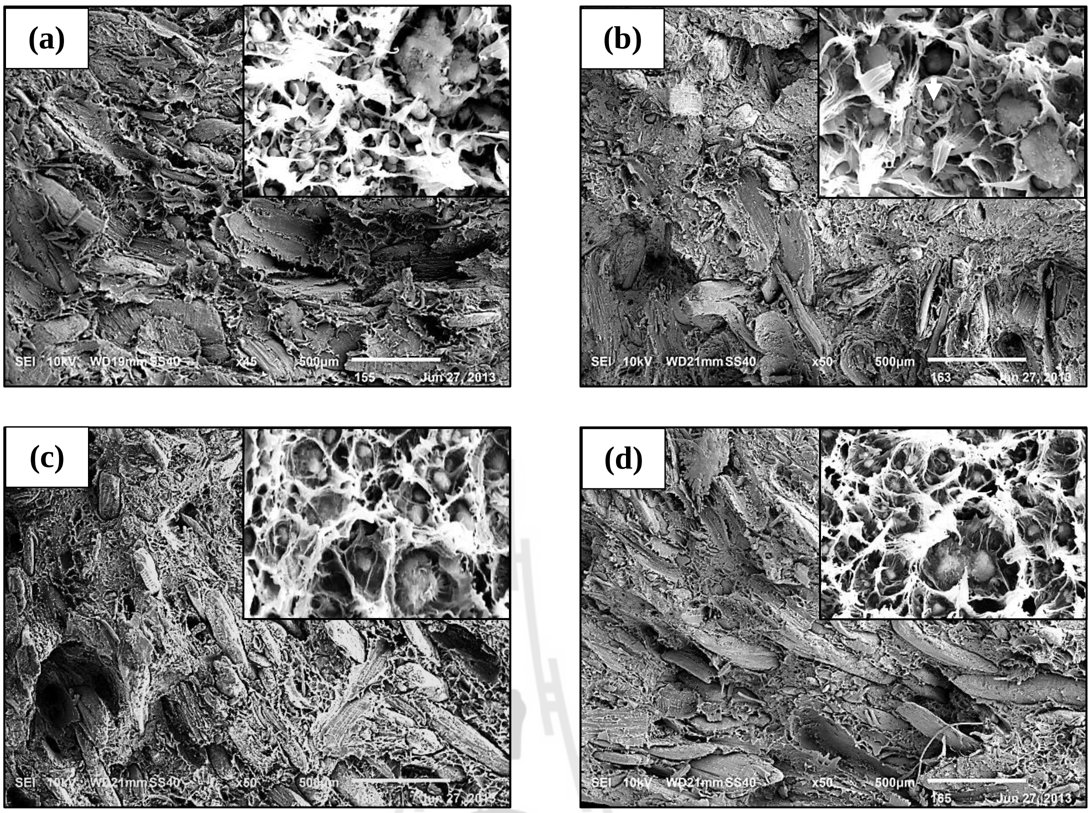


Figure 4.29 SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with different MAPE contents at magnification 50x (a) 20ATH/10ZB, (b) 1MAPE, (c) 3MAPE and (d) 5MAPE.

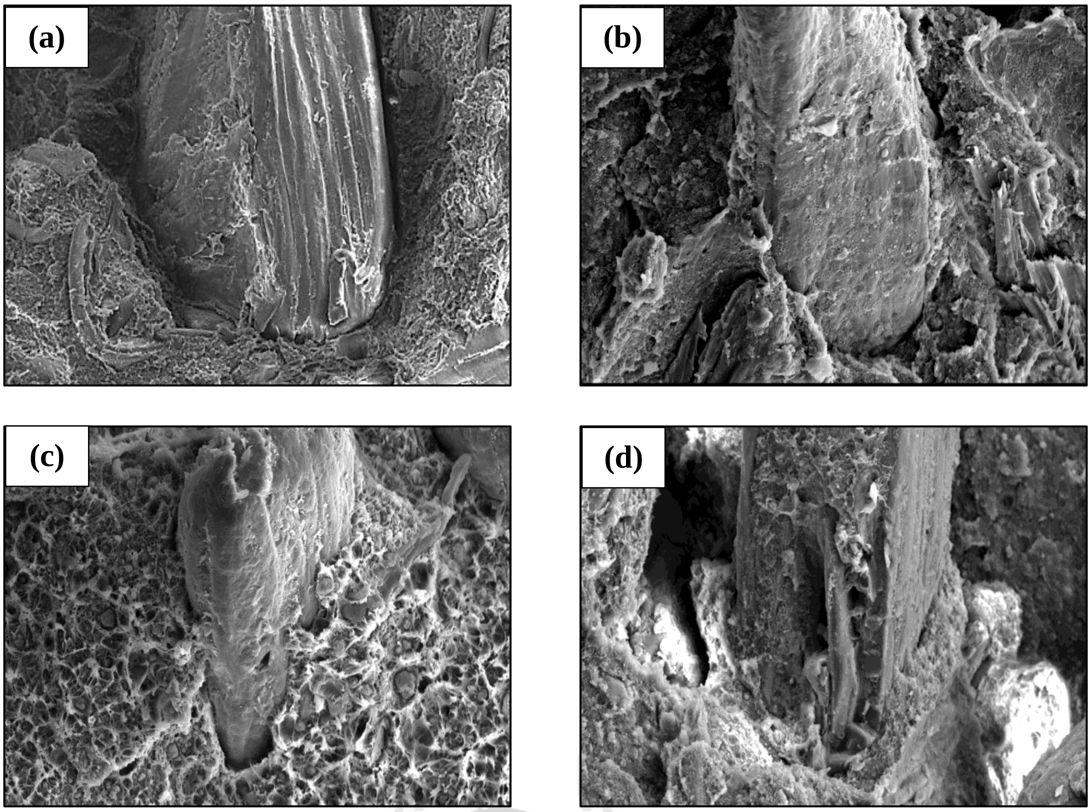


Figure 4.30 SEM micrographs of tensile fracture surface of alkali treated sawdust/rHDPE composites with different MAPE contents at magnification 350x (a) 20ATH/10ZB, (b) 1MAPE, (c) 3MAPE and (d) 5MAPE.

CHAPTER V

CONCLUSIONS

Hemicellulose and lignin contents of sawdust were decreased whereas cellulose content was increased after alkali treatment. FTIR results showed a decrease in the peak intensity at 3339, 1732, 1600, 1505, 1332 and 1231 cm^{-1} due to a removal of hemicellulose and lignin. Alkali treatment improved thermal stability of sawdust because the low thermal stability constituents i.e. impurities, wax and hemicellulose containing in sawdust were removed during alkali treatment. SEM micrographs revealed that surface of alkali treated sawdust was cleaner and rougher than that of untreated sawdust. The optimum alkali treatment condition was 2% w/v NaOH for 30 min.

Tensile strength, elongation at break, flexural strength and impact strength of sawdust/rHDPE composites were reduced with increasing sawdust content whereas tensile modulus and flexural modulus were increased. Thermal stability and flame retardancy of the composites were decreased when sawdust content was increased. SEM micrographs showed the agglomeration of sawdust in rHDPE and the poor interfacial adhesion between sawdust and rHDPE. Alkali treatment enhanced the compatibility between sawdust and rHDPE resulting in the improvement of mechanical properties of the composites at all sawdust contents. Thermal stability of alkali treated sawdust/rHDPE composites exhibited higher than that of untreated sawdust/rHDPE composites. SEM micrographs revealed the improvement of interfacial adhesion between alkali treated sawdust and rHDPE matrix. Among sawdust/rHDPE composites, alkali treated sawdust/rHDPE composite containing 30 wt% of sawdust showed the highest mechanical properties, flame retardancy and thermal stability.

ATH improved flame retardancy and thermal stability of alkali treated sawdust/rHDPE composites because water and char residue which formed during the thermal decomposition of ATH delayed thermal decomposition of the composites. With increasing ATH content, flame retardancy and thermal stability of the composites were improved while mechanical properties were not much affected. The composite containing 30 phr of ATH showed the highest flame retardancy and thermal stability. SEM micrographs showed poor dispersion of ATH in rHDPE matrix.

The incorporation of the combination of ATH and ZB into the alkali treated sawdust/rHDPE composites slightly increased mechanical properties of the composites comparing to the composite containing ATH because ZB had rougher surface than ATH resulting in better mechanical interlocking between ZB and polymer matrix. Thermal stability of the composites increased with increasing ZB content in the mixture of ATH and ZB. The composite containing 30ZB exhibited the highest mechanical properties and thermal stability but the lowest flame retardancy. The combination of ATH and ZB at ATH:ZB ratios of 2:1 and 1:1 exhibited synergistic effect in enhancing flame retardancy of the composites. Moreover, the composite containing ATH/ZB at the ratio of 2:1 showed the highest flame retardancy. SEM micrographs displayed that ZB had better adhesion with rHDPE matrix than ATH.

Mechanical properties of the composites were significantly enhanced whereas flame retardancy and thermal stability were slightly increased with increasing MAPE content. SEM micrographs revealed that the interfacial adhesion between fillers and rHDPE matrix was improved with increasing MAPE content. Moreover, the good distribution of flame retardants and sawdust in rHDPE matrix was also observed.

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APPENDIX A

PUBLICATIONS

List of Publications

Jarapanyacheep, R. and Jarukumjorn, K. (2013). Mechanical properties and flammability of sawdust/recycled high density polyethylene composites. In **Proceedings of Pure and Applied Chemistry International Conference 2013 (PACCON)** (pp 665-668). Chonbiri, Thailand.

Jarapanyacheep, R. and Jarukumjorn, K. (2013). Effect of flame retardant on mechanical properties and flammability of sawdust/recycled high density polyethylene composites. In **abstract of The 4th Research Symposium on Petrochemical and Materials Technology and The 19th PPC Symposium on Petroleum, Petrochemicals and Polymers** (pp 93). Bangkok, Thailand.

Jarapanyacheep, R. and Jarukumjorn, K. (2013). Effects of sawdust content and alkali treatment on mechanical and flame retarding properties of sawdust/recycled high density polyethylene composites. **Adv. Mater. Res.** 970: 79-83.

MECHANICAL PROPERTIES AND FLAMMABILITY OF SAWDUST/RECYCLED HIGH DENSITY POLYETHYLENE COMPOSITES

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Abstract: Sawdust/recycled high density polyethylene (rHDPE) composites were prepared at sawdust contents of 30, 40, and 50 wt%. The composites were prepared using a twin screw extrusion and test specimens were molded by a compression molding machine. Mechanical properties, flammability, and morphology of the composites were investigated. With increasing sawdust content, tensile modulus of the composites increased while tensile strength and elongation at break decreased. Burning rate of the composites increased with increasing sawdust content. In order to enhance the flame retardancy of the composites, aluminum trihydrate (ATH) was used at a content of 10 phr. ATH improved the flame retardancy of the composites at all sawdust contents without deterioration of the mechanical properties. Moreover, maleic anhydride grafted polyethylene (MAPE) was added at 5 phr to improve the compatibility of the composites. The mechanical and flame retarding properties of the composites at all saw dust contents were enhanced with incorporating MAPE.

1. Introduction

Lignocellulosic filler/Polymer composites have attracted much attention from a number of researchers and manufactures in recent years. The addition of the lignocellulosic filler in polymer aims to produce unique characteristics of light weight and recyclability. In comparison with other fillers such as traditional glass fibers and inorganic mineral fillers, lignocellulosic fillers have received much attention for being used as reinforcing materials because of their biodegradability and low cost. Sawdust (SWD) is the one of lignocellulosic filler obtaining from wood industry. The most utilization of this material is used as fuel or made the particles board [1]. The use of sawdust in wood polymer composites is an alternative method to add value to sawdust. Basically, sawdust is light, cheap and stiffness, thus it can be added to commodity matrix in certain loading level hence offering the best solutions for the utilization of waste wood with good mechanical properties and low cost [2].

Nowadays, plastics are widely used as packaging, furniture, electronic parts, and housewares. This has caused an increasing concern regarding the environment and problem of plastic waste disposal. Alternative methods for handling plastic waste include burial, incineration, depolymerization and recycling. Plastic recycling is a convenient way to solve the problem of the waste management. High density polyethylene (HDPE) is thermoplastics commonly used as packaging (bottles, films, etc.). HDPE is chosen to produce the composites since it is a major portion of the post-consumer household wastes. In addition, HDPE possesses outstanding properties such as high toughness, good impact resistance and good chemical resistance.

Wood/thermoplastic composites (WRPCs) have been known for many years. WRPCs containing recycled plastics and wood fibers offer interesting combinations of cost and properties. The utilization of recycled plastic for the manufacture of WRPCs has been studied [3-4]. Najafi [5] found that the wood flour/rHDPE showed similar mechanical properties but were much cheaper when compared to wood flour/virgin HDPE composites.

However, there are two major problems facing the production of SWD/rHDPE composites. The first one is the flammability of the composites rHDPE and SWD are flammable materials. This problem limits the application of the composites in many areas. Therefore, the improvement of flame retardancy of the composite must be considered. Generally, the method to improve fire resistance of flammable materials is directly incorporate flame retardants into the materials. Flame retardants are often preferred to provide low flammability to polymeric materials because the use of flame retardants is an acceptable compromise between cost and properties [6]. Aliminum trihydrate (ATH) is a wildly used flame retardant for plastics. The particular benefits of ATH include low health hazard, cost effectiveness and smoke suppressant [7]. ATH decomposes in an environmental friendly mechanism known as dehydration. This reaction results in the formation of aluminum oxide, which forms an inert residue acting as protective layer, and water which dilutes the smoke and combustible gases [8].

Another problem is the poor interfacial adhesion between hydrophobic PE and hydrophilic SWD resulting in the composites with poor mechanical properties [9]. The compatibility of the composites can be improved by various methods such as fiber surface treatment, matrix modification and addition of compatibilizer. Maleic anhydride grafted polyethylene (MAPE) is generally introduced as a compatibilizer to improve the compatibility between SWD and HDPE matrix. The PE segments of MAPE form compatible blends with HDPE. The anhydride groups of MAPE form hydrogen and chemical bonds with the hydroxyl groups of cellulose, which strongly anchored the reactive groups onto SWD surface [10]. Moreover, the addition of MAPE can also improve the compatibility and dispersion of ATH in HDPE matrix. The polar part of MAPE can interact with hydroxyl groups of ATH and MAPE anchors its nonpolar HDPE part to the HDPE matrix resulting in the better compatibility and the dispersion of ATH and rHDPE matrix [11].

In this study, the effects of SWD content and ATH on mechanical properties and flame retardantcy of SWD/rHDPE composites were studied. In addition, the influence of MAPE as a compatibilizer on the properties of the composites was investigated.

2. Materials and Methods

2.1 Materials

Recycled high density polyethylene (rHDPE) was purchased from a local plastic recycling plant. Indian Walnut sawdust *(Albizia lebbeck Benth)* with particle sizes of 425-600 μm was obtained from Huathalae saw mill, Nakhon Ratchasima. Maleic anhydride grafted polyethylene (MAPE, Fusabond® MB100D, DuPont) was used as a compatibilizer. Aluminum trihydrate (ATH, APYRAL® 40CD, Nabaltec), was used as a flame retardant.

Table 1: Composition and their designation of rHDPE and rHDPE composites

Name	rHDPE (wt%)	SWD (wt%)	ATH (phr)	MAPE (phr)
rHDPE	100	-	-	-
30SWD	70	30	-	-
40SWD	60	40	-	-
50SWD	50	50	-	-
30SWD/ 10ATH	70	30	10	-
40SWD/ 10ATH	60	40	10	-
50SWD/ 10ATH	50	50	10	-
30SWD/ 10ATH/ 5MAPE	70	30	10	5
40SWD/ 10ATH/ 5MAPE	60	40	10	5
50SWD/ 10ATH/ 5MAPE	50	50	10	5

2.2 Composite preparation

Composition of rHDPE composites and their designation used in this study are shown in Table 1. rHDPE composites were prepared using a twin screw extruder (Brabender, 35/17D). The temperatures of the four processing zones were 175, 180, 185 and 190°C. The screw speed was 20 rpm. The test specimens were processed by a compression molding machine (Labtech, LP20-B) at 190°C.

2.3 Composite characterization

Mechanical properties: Tensile tests were examined according to ASTM D638 using a universal testing machine (Instron, 5565) with a load cell of 5 kN and a crosshead speed of 50 mm/min. Five samples were investigated.

Flammability: Flammability of rHDPE and rHDPE composites was examined by a horizontal burning test according to ASTM D635. The specimen was held horizontally and a flame was applied to one end of the specimen. A burning time from the first reference mark, i.e. 25 mm from the end, to the second reference mark, i.e. 100 mm from the end, was recorded. Three specimens from each composite were tested. Then, burning rates of the composites were calculated.

Morphological properties: Tensile fracture surfaces of rHDPE composites were examined using a scanning electron microscope (SEM, NeoScope JCM-6010). The fracture surface of the specimens was coated with gold before analysis.

Table 2: Tensile strength, tensile modulus and elongation at break of rHDPE and rHDPE composites.

Name	Tensile strength (MPa)	Tensile Modulus (GPa)	Elongation at break (%)
rHDPE	25.81±6.90	0.60±0.02	109.27±3.15
30SWD	20.29±0.23	0.88±0.10	4.48±0.13
40SWD	19.97±0.14	0.96±0.01	4.26±0.50
50SWD	16.05±2.79	1.05±0.03	2.12±0.13
30SWD/ 10ATH	22.42±0.19	0.93±0.08	4.45±0.21
40SWD/ 10ATH	21.95±2.25	1.05±0.01	4.25±0.10
50SWD/ 10ATH	19.39±3.14	1.07±0.04	2.10±0.31
30SWD/ 10ATH/ 5MAPE	29.13±0.78	0.94±0.03	6.10±0.33
40SWD/ 10ATH/ 5MAPE	27.63±0.43	1.04±0.01	5.40±0.42
50SWD/ 10ATH/ 5MAPE	21.58±1.87	1.05±0.01	2.69±0.23

3. Results and Discussions

3.1 Mechanical properties

Tensile properties of rHDPE and rHDPE composites are illustrated in Table 2. Tensile strength of the composites decreased with adding SWD. Moreover, tensile strength of the composites decreased continuously with increasing SWD content. This indicated the poor compatibility between polar filler SWD and nonpolar rHDPE matrix. When ATH was added, tensile strength of rHDPE composites increased. The results suggested that ATH acted as reinforcing filler in this system. Tensile strength of the compatibilized rHDPE composites was higher than the rHDPE composites. This was probably due to improved interfacial adhesion between SWD and HDPE matrix. The chemical (ester bond) can be formed between hydroxyl groups of SWD and anhydride groups of MAPE. The PE segment of MAPE formed compatible blends with HDPE matrix. Therefore, the surface adhesion between SWD and HDPE matrix enhanced with incorporating MAPE compatibilizer [10]. Moreover, the addition of MAPE also improved the compatibility and dispersion of ATH in rHDPE matrix resulting in better tensile strength [11].

Table 3: Burning rate of rHDPE and rHDPE composites

Name	Burning rate (mm/min)
rHDPE	21.48±0.60
30SWD	30.61±0.35
40SWD	31.51±2.44
50SWD	32.83±2.75
30SWD/10ATH	24.51±2.14
40SWD/10ATH	29.65±0.33
50SWD/10ATH	31.30±1.84
30SWD/10ATH/5MAPE	18.99±1.38
40SWD/10ATH/5MAPE	22.22±0.23
50SWD/10ATH/5MAPE	22.04±1.46

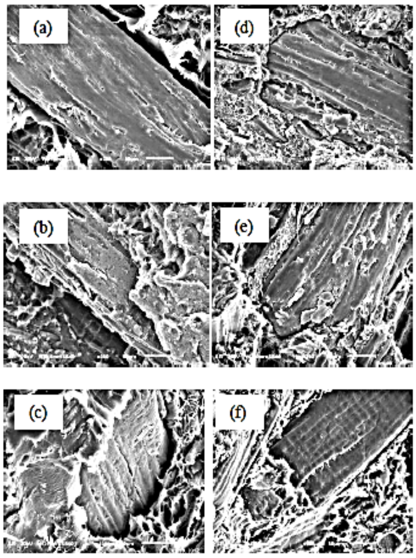


Figure 1. SEM micrographs of fracture surfaces of composites at 350x (a) 30SWD, (b) 40SWD and (c) 50SWD, (d) 30SWD/10ATH/5MAPE, (e) 40SWD/10ATH/5MAPE and (f) 50SWD/10ATH/5MAPE

Tensile modulus of rHDPE composites increased with increasing SWD content. This indicated the ability of SWD to impart greater stiffness to the composites. With the addition of ATH, the tensile modulus of the composites was increased. However, the tensile modulus of the composites was insignificantly changed with the presence of MAPE.

Elongation at break of the composites decreased with increasing SWD content. This was because the presence of SWD caused matrix to lose its ability against elastic deformation. Hence, the composites broke at a lower elastic deformation. When ATH was added, elongation at break of rHDPE composites was slightly decreased. Moreover, MAPE marginally improved the elongation at break of the composites.

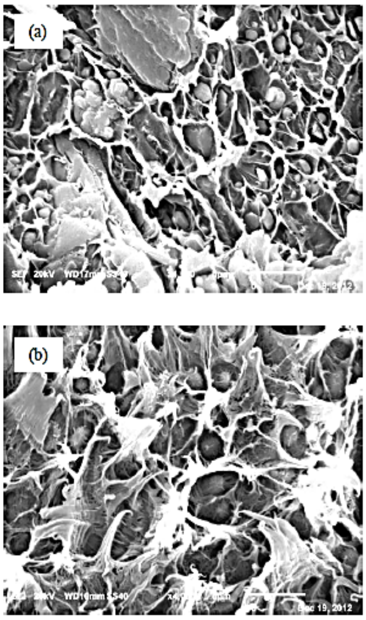
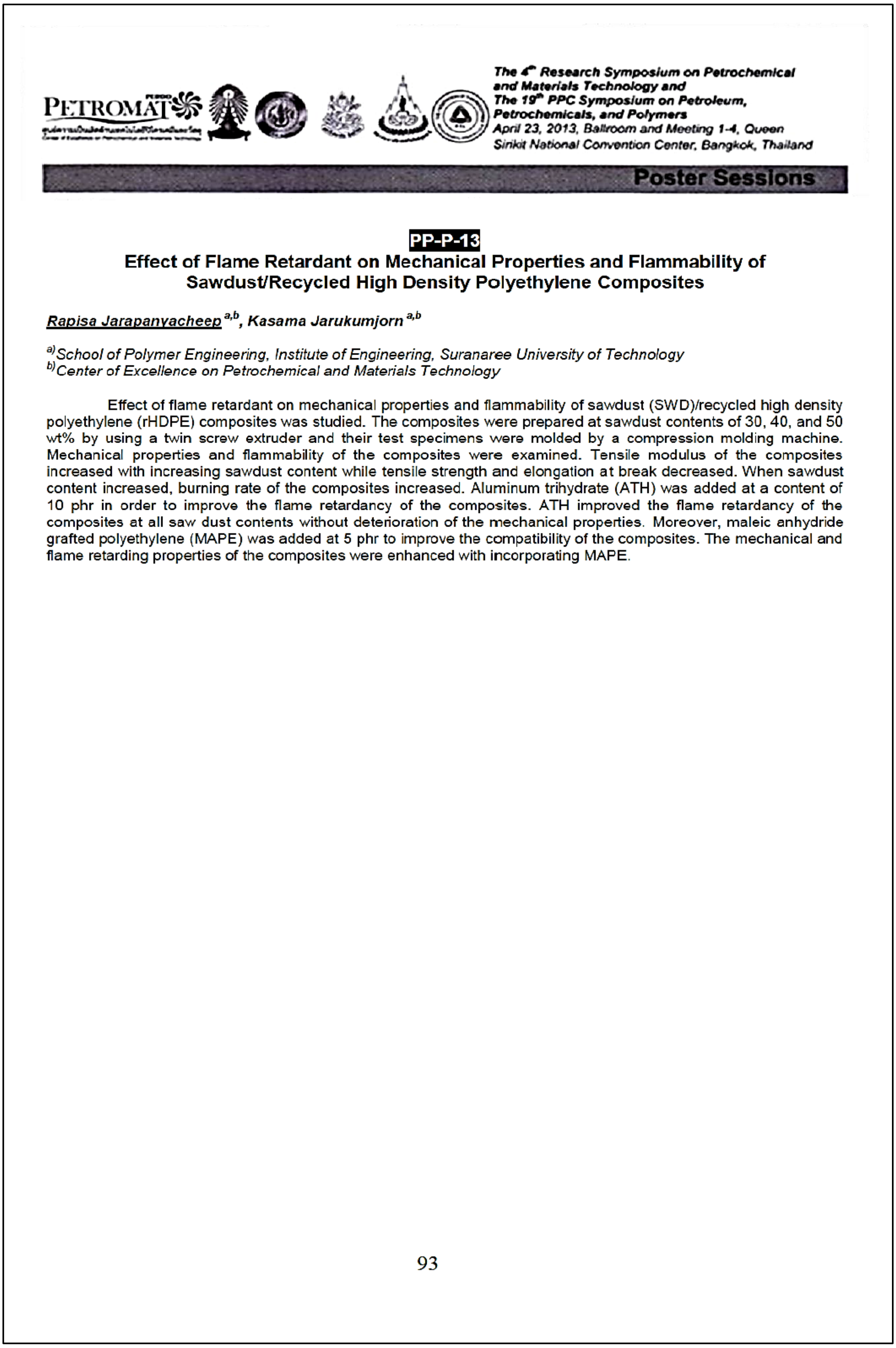


Figure 2. SEM micrographs of fracture surfaces of composites at 4000x (a) 50SWD and (b) 50SWD/ATH/MAPE

3.2 Flammability

Burning rates of rHDPE and rHDPE composites measured by horizontal burning test are shown in Table 3. rHDPE composite showed higher burning rate than rHDPE and the burning rate increased with increasing sawdust content. This indicated that sawdust filled rHDPE composite had high sensitivity to flame. Li and He [12] reported that the thermal degradation temperature of LLDPE in wood fiber/LLDPE composites was lower than neat LLDPE due to the low thermal stability of wood fiber.



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Poster Sessions

PP-P-13

Effect of Flame Retardant on Mechanical Properties and Flammability of Sawdust/Recycled High Density Polyethylene Composites

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Effect of flame retardant on mechanical properties and flammability of sawdust (SWD)/recycled high density polyethylene (rHDPE) composites was studied. The composites were prepared at sawdust contents of 30, 40, and 50 wt% by using a twin screw extruder and their test specimens were molded by a compression molding machine. Mechanical properties and flammability of the composites were examined. Tensile modulus of the composites increased with increasing sawdust content while tensile strength and elongation at break decreased. When sawdust content increased, burning rate of the composites increased. Aluminum trihydrate (ATH) was added at a content of 10 phr in order to improve the flame retardancy of the composites. ATH improved the flame retardancy of the composites at all saw dust contents without deterioration of the mechanical properties. Moreover, maleic anhydride grafted polyethylene (MAPE) was added at 5 phr to improve the compatibility of the composites. The mechanical and flame retarding properties of the composites were enhanced with incorporating MAPE.

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Effects of Sawdust Content and Alkali Treatment on Mechanical and Flame Retarding Properties of Sawdust/Recycled High Density Polyethylene Composites

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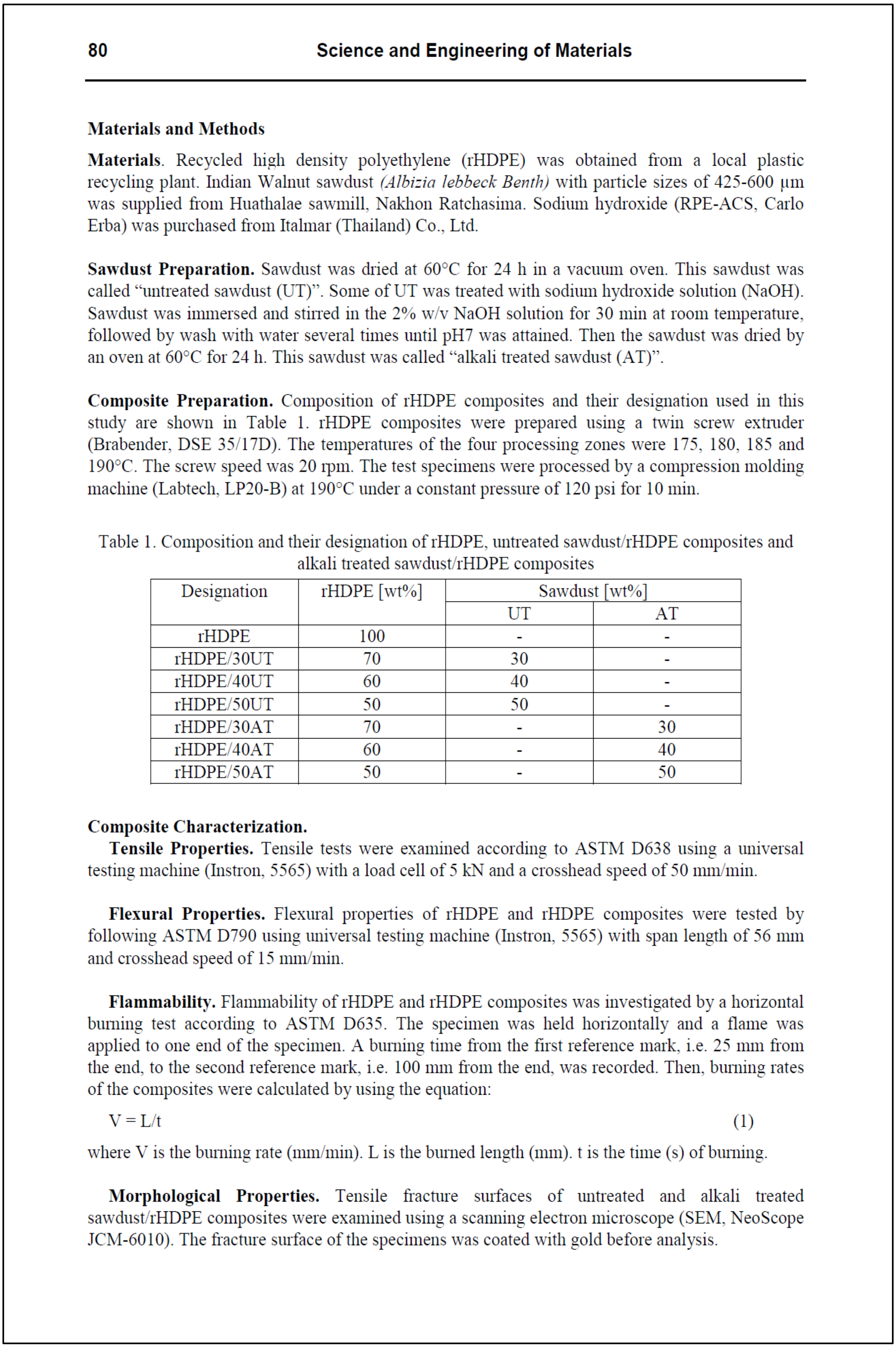
Keywords: Recycled high density polyethylene, Sawdust, Alkali treatment, Mechanical properties, Flammability.

Abstract. Sawdust/recycled high density polyethylene (rHDPE) composites were prepared and their mechanical properties, flammability and morphology were investigated. Sawdust was used at contents of 30, 40 and 50 wt%. With increasing sawdust content, tensile strength and elongation at break of the composites decreased whereas tensile modulus increased. Flexural properties showed the same trend as tensile properties. Flammability of the composites enhanced with increasing sawdust content. Mechanical properties of alkali treated sawdust/rHDPE composites were higher than those of untreated sawdust/rHDPE composites at all sawdust contents. Moreover, alkali treatment improved flame retardancy of the composites. SEM micrographs showed that alkali treatment enhanced the interfacial adhesion between sawdust and rHDPE matrix.

Introduction

Wood/polymer composites (WPCs), made of virgin or recycled polymer and wood filler, have gained much attention both in the research area and the industry. The addition of wood filler into polymers improves stiffness and strength of the composites. The advantages of the wood filler include high modulus, high strength, low cost, low density, renewable nature, biodegradability and nonabrasiveness. Sawdust, by-product obtaining from the wood industry, is one the most used wood filler for the manufacture of WPCs. The use of sawdust in producing WPCs benefits to the enviroment and also adds value to sawdust. Many researchers have prepared WPCs from recycled thermoplastics and sawdust [1,2,3]. They reported that the composites, made of recycled high density polyethylene (rHDPE) and sawdust, showed similar mechanical properties but were much cheaper than the composites made from virgin HDPE [2,3]. However, the main drawbak of the WPCs is the incompatibility between hydrophilic sawdust and hydrophobic rHDPE matrix, leading to poor mechanical properties of the composites [4,5,6]. Many strategies, i.e. fiber surface treatment, matrix modification, adding compatibilizer, have been developed to improve the compatibility of the composites [7,8,9]. Alkali treatment, one of the most use chemical surface treatments, increases the fiber surface roughness by removal of waxes, pectin, lignin and hemicellulose on fiber surface, resulting in better mechanical interlocking between sawdust and rHDPE matrix [10,11,12]. This results in the improvement of the mechanical properties of the composites. Tensile and flexural properties of alkali treated piassava fiber/rHDPE composites were increased when compared to untreated piassava fiber/rHDPE composites due to improved adhesion between piassava fiber and rHDPE matrix [12]. In addition, alkali treatment increased char residue of rice husk fiber reinforced recycled low density polyethylene composites [13]. This led to the enhancement of flame retardancy of the composites because the char residue formed the protective layer and separated the underlying material from heat and oxygen [14]. The aim of this study is to investigate the effects of sawdust content and alkali treatment on mechanical and flammability of sawdust/rHDPE composites.



Materials and Methods

Materials. Recycled high density polyethylene (rHDPE) was obtained from a local plastic recycling plant. Indian Walnut sawdust *(Albizia lebbeck Benth)* with particle sizes of 425-600 µm was supplied from Huathalae sawmill, Nakhon Ratchasima. Sodium hydroxide (RPE-ACS, Carlo Erba) was purchased from Italmar (Thailand) Co., Ltd.

Sawdust Preparation. Sawdust was dried at 60°C for 24 h in a vacuum oven. This sawdust was called "untreated sawdust (UT)". Some of UT was treated with sodium hydroxide solution (NaOH). Sawdust was immersed and stirred in the 2% w/v NaOH solution for 30 min at room temperature, followed by wash with water several times until pH7 was attained. Then the sawdust was dried by an oven at 60°C for 24 h. This sawdust was called "alkali treated sawdust (AT)".

Composite Preparation. Composition of rHDPE composites and their designation used in this study are shown in Table 1. rHDPE composites were prepared using a twin screw extruder (Brabender, DSE 35/17D). The temperatures of the four processing zones were 175, 180, 185 and 190°C. The screw speed was 20 rpm. The test specimens were processed by a compression molding machine (Labtech, LP20-B) at 190°C under a constant pressure of 120 psi for 10 min.

Table 1. Composition and their designation of rHDPE, untreated sawdust/rHDPE composites and alkali treated sawdust/rHDPE composites

Designation	rHDPE [wt%]	Sawdust [wt%]	
		UT	AT
rHDPE	100	-	-
rHDPE/30UT	70	30	-
rHDPE/40UT	60	40	-
rHDPE/50UT	50	50	-
rHDPE/30AT	70	-	30
rHDPE/40AT	60	-	40
rHDPE/50AT	50	-	50

Composite Characterization.

Tensile Properties. Tensile tests were examined according to ASTM D638 using a universal testing machine (Instron, 5565) with a load cell of 5 kN and a crosshead speed of 50 mm/min.

Flexural Properties. Flexural properties of rHDPE and rHDPE composites were tested by following ASTM D790 using universal testing machine (Instron, 5565) with span length of 56 mm and crosshead speed of 15 mm/min.

Flammability. Flammability of rHDPE and rHDPE composites was investigated by a horizontal burning test according to ASTM D635. The specimen was held horizontally and a flame was applied to one end of the specimen. A burning time from the first reference mark, i.e. 25 mm from the end, to the second reference mark, i.e. 100 mm from the end, was recorded. Then, burning rates of the composites were calculated by using the equation:

$$V = L/t \qquad (1)$$

where V is the burning rate (mm/min). L is the burned length (mm). t is the time (s) of burning.

Morphological Properties. Tensile fracture surfaces of untreated and alkali treated sawdust/rHDPE composites were examined using a scanning electron microscope (SEM, NeoScope JCM-6010). The fracture surface of the specimens was coated with gold before analysis.

Results and Discussion

Mechanical Properties. Tensile strength, tensile modulus and elongation at break of rHDPE and rHDPE composites are shown in Fig. 1 (a)-(c). With the addition of sawdust, tensile strength and tensile modulus of rHDPE were improved but elongation at break was reduced. This result suggested that sawdust acted as reinforcing filler in the system [15]. With increasing sawdust content, tensile strength of the composites decreased as illustrated in Fig. 1 (a). This may be due to the poor dispersion of sawdust in rHDPE matrix and the incompatibility between sawdust and rHDPE matrix [6,7]. From Fig. 1 (b), tensile modulus of the composites enhanced with increasing sawdust content indicating the ability of sawdust to impart greater stiffness to the composites [6]. The reduction of elongation at break of the composites was found with increasing sawdust content as shown in Fig. 1 (c). This was because the presence of sawdust caused matrix to lose its ability against elastic deformation [7]. Hence, the composites broke at a lower elastic deformation. Tensile strength, tensile modulus and elongation at break of alkali treated sawdust/rHDPE composites were slightly higher than that of untreated sawdust/rHDPE composites at all sawdust contents. This was because alkali treatment improved the fiber surface roughness by removal of waxes, pectin, lignin and hemicellulose on fiber surface resulting in better mechanical interlocking between sawdust and rHDPE matrix [10,11,12]. Flexural strength and flexural modulus of rHDPE, untreated and alkali treated sawdust/rHDPE composites are shown in Fig. 2 (a) and (b), respectively. Flexural properties of rHDPE composites showed the same trend as tensile properties. Flexural strength and flexural modulus enhanced with incorporating sawdust. Flexural strength of the composites decreased whereas flexural modulus increased with increasing sawdust content as illustrated in Fig. 2 (b). After alkali treatement, flexural strength and flexural modulus of alkali treated sawdust/rHDPE composites were slightly higher than those of untreated composites at all sawdust contents due to enhanced adhesion between sawdust and rHDPE matrix. Elzubair et al. [12] also reported that alkali treatment improved flexural properties of piassava fiber/rHDPE composites.

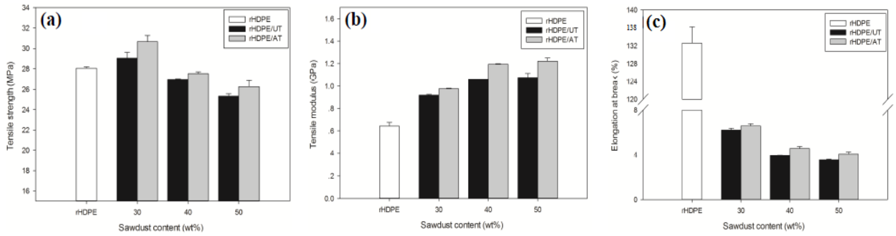


Figure 1. Tensile properties of rHDPE, untreated sawdust/rHDPE composites and alkali treated sawdust/rHDPE composites with different sawdust contents: (a) tensile strength, (b) tensile modulus and (c) elongation at break.

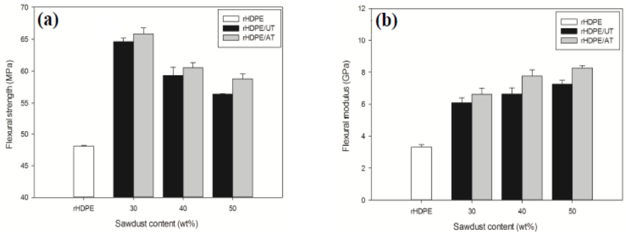


Figure 2. Flexural properties of rHDPE, untreated sawdust/rHDPE composites and alkali treated sawdust/rHDPE composites with different sawdust contents: (a) flexural strength and (b) flexural modulus.

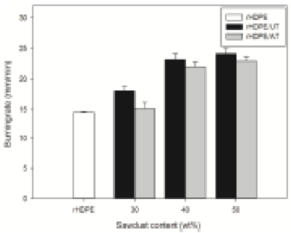


Figure 3. Burning rates of rHDPE, untreated sawdust/rHDPE composites and alkali treated sawdust/rHDPE composites with different sawdust contents.

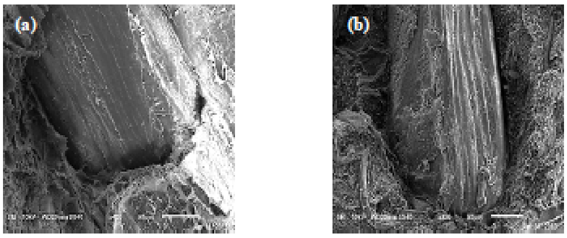


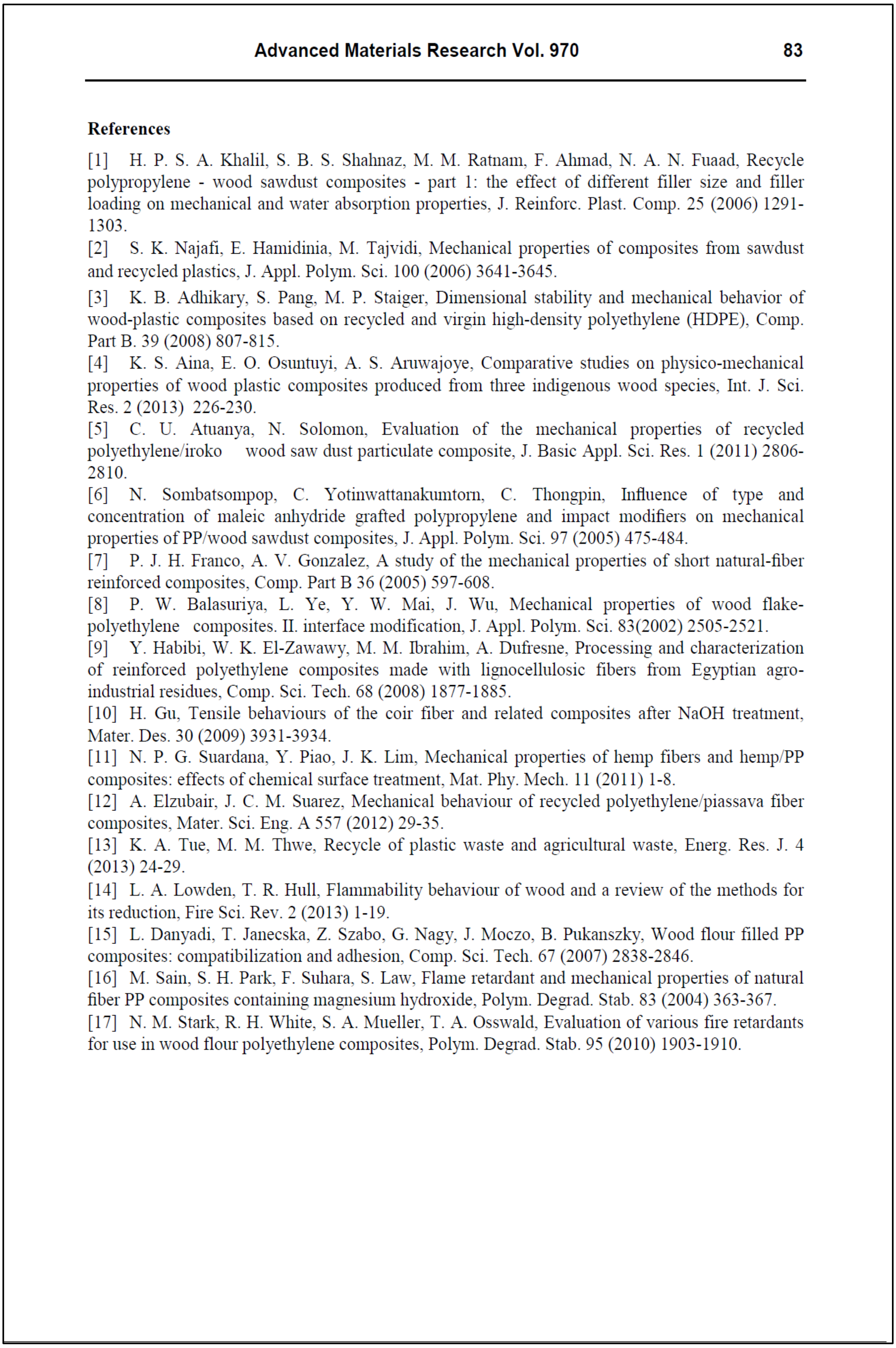
Figure 4. SEM micrographs of tensile fracture surface of the composites at 350x (a) rHDPE/30UT and (b) rHDPE/30AT.

Flammability. Horizontal burning rates of rHDPE, untreated and alkali treated sawdust/rHDPE composites are shown in Fig. 3. rHDPE composites had higher burning rate than rHDPE and the burning rate increased with increasing sawdust content. This indicated that sawdust/rHDPE composite had high sensitivity to flame [16]. Stark et al. [17] also found that flammability of wood flour/HDPE increased with increasing wood flour content. Alkali treated sawdust/rHDPE composites showed slightly lower burning rate than untreated sawdust/rHDPE composites. This may be due to an increase in char residue of the composites by alkali treatment [13]. As the char layer built up, it may separate the composites from heat and oxygen [14].

Morphological Properties. SEM micrographs of tensile fracture surfaces of untreated and alkali treated sawdust/rHDPE composites are shown in Fig. 4 (a) and (b). As shown in Fig. 4 (a), gap between rHDPE and sawdust was observed indicating the poor compatibility between sawdust and rHDPE matrix. This led to the reduction of mechanical properties of rHDPE composites. From Fig. 4 (b), alkali treatment enhanced the interfacial adhesion between sawdust and rHDPE matrix resulting in the improvement of the mechanical properties of the composites [11]. Elzubair et al. [12] also observed that alkali treatment improved the compatibility of piassava fiber/rHDPE composites.

Summary

Tensile strength and elongation at break of rHDPE reduced whereas tensile modulus improved with the incorporation of sawdust. With increasing sawdust content, tensile strength and elongation at break decreased but tensile modulus increased. Burning rate of rHDPE composites was higher than rHDPE and further enhanced with increasing sawdust content. Alkali treatment improved the interfacial adhesion between sawdust and rHDPE resulting in the improvement of mechanical properties at all sawdust contents. Furthermore, flame retardancy of alkali treated sawdust/rHDPE composites was higher than untreated sawdust/rHDPE composites. SEM micrographs revealed that alkali treatment improved the interfacial adhesion between sawdust and rHDPE matrix.



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BIOGRAPHY

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