

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

**DEVELOPMENT OF NATURAL FIBER REINFORCED COMPOSITES
WITH FLAME RETARDANT FILLERS**

M.Sc. THESIS

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Department of Textile Engineering

Innovative Technical Textiles Programme

23 JUNE, 2023

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**ALEV GECİKTİRİCİ KATKILAR İLE DOĞAL LİF TAKVİYELİ
KOMPOZİTLER GELİŞTİRİLMESİ**

YÜKSEK LİSANS TEZİ

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To all low waged farmers of BANGLADESH,

FOREWORD

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ABBREVIATIONS

APP	: Ammonium Polyphosphate
ATH	: Aluminum Trihydroxide
EHC	: Effective Heat of Combustion
FR	: Flame Retardant
FTIR	: Fourier-Transform Infrared
HRR	: Heat Release Rate
LOI	: Limiting Oxygen Index
MA-g-PP	: Maleic Anhydride Grafted Polypropylene
MARHE	: Maximum Average Rate of Heat Emission
MLR	: Mass Loss Rate
NC	: Not Classified
NFC	: Natural Fiber Composite
NFPCs	: Natural Fiber Polymer Composites
pHRR	: Peak Heat Release Rate
PP	: Polypropylene
SEA	: Specific Energy Area
SEM	: Scanning Electron Microscopy
SPR	: Smoke Production Rate
TFO	: Time to Flame Out
TSP	: Total Smoke Production
TSR	: Total Smoke Release
ZB	: Zinc Borate

SYMBOLS

A	: Cross-section area
ATHR	: Average heat release rate
CO	: Carbon mono-oxide
CO₂	: Carbon-dioxide
F_{max}	: Maximum tensile load
F_y	: Load at yield point
L	: Length
m	: Mass
TEHC	: Time of effect heat of combustion
TPHRR	: Time of peak heat release rate
T₁^a	: Sample burning time from first 10 seconds of flame application
T₂^a	: Sample burning time from second 10 seconds of flame application
ρ	: Density
Σ	: Stress
σ_u	: Ultimate tensile strength
σ_y	: Yield strength
E	: Strain
ε_f	: Strain at break
t_f	: Total flame time
ΔL	: Change of length
ΔL_f	: Change of length at failure

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DEVELOPMENT OF NATURAL FIBER REINFORCED COMPOSITES WITH FLAME RETARDANT FILLERS

SUMMARY

The global automotive industry is highly focused on application natural fibers with thermoplastic polymers due to its commercial applicability. Due to being environmentally friendly and having better advantages such as lightweight behavior, good strength to weight ratio and weather resistance, jute fiber has become a very suitable option to make composite material with thermoplastic materials such as polypropylene (PP). Application of jute fiber with PP matrix is growing day by day. In addition to that, flame retardant jute/PP composites have become a new set of trends of reliability. However, to achieve flame retardancy without compromising mechanical properties is a key concern to be noticed.

In this thesis, flame-retardant jute/PP composites based on ATH/ZB and APP/ZB at a ratio of 80:20 were produced via combination of extrusion process and hot press method. Zinc borate (ZB) was used for creating a synergetic effect with aluminium trihydride (ATH) and ammonium polyphosphate (APP). Before infusing flame retardant fillers to jute/PP composites, fiber loading levels were optimized with PP matrix at 20 wt.% of loading level based on long jute fiber (LJF) and short jute fiber (SJF) by the presence of 5 wt.% of maleic anhydride-grafted polypropylene (MA-g-PP). The results indicated that length of short jute fiber was having a positive impact over mechanical properties (tensile strength and flexural strength). A maximum tensile strength value of 37.32 ± 0.75 MPa and flexural strength value of 34.68 ± 1.70 MPa have been achieved. Tensile modulus was reached to 1811 ± 81 MPa and flexural modulus was reached to 1181 ± 95 MPa. Further investigations were carried out with 20 wt.% SJF by increasing fiber loading up to 60 wt.% since the 20 wt.% SJF had been found to performed better than the 20 wt.% of LJF one in the initial mechanical property experiments. It was evident that increasing jute fiber content decreases tensile strength as well as strain (%) value. A clear noticeable decrease of tensile strength value (19.50 ± 2.04 MPa) was found when 60 wt.% of SJF was infused within PP matrix. Until 40 wt.% of loading of short jute fiber flexural strength remains high (38.98 ± 3.45 MPa) and consistent compared to others. But, when 60 wt.% loading of SJF was infused flexural strength value was decreased to minimum of 37.77 ± 1.25 MPa was reached a tensile strength value of 19.50 ± 2.04 MPa. Morphological analysis was carried out via scanning electronic microscope (SEM). Better adhesion was noticed for 30 wt.% loading of SJF which ensured optimum mechanical properties (tensile properties: strength- 29.5 ± 2.74 MPa, strain- 4.97 ± 0.84 % and young modulus- 1517 ± 198 MPa; flexural properties: strength: 38.17 ± 1.9 MPa, strain- 7.18 ± 1.17 % and flexural modulus- 1488 ± 88 MPa) that was achieved. By keeping SJF loading level constant at 30 wt.%, as flame-retardant additives ATH/ZB and APP/ZB were

incorporated at a loading level of 30 wt.% and 40 wt.% to achieve better flame-retardant properties without compromising mechanical properties.

Char formation were greatly influenced via APP/ZB on jute/PP composites. To analyze flame-retardant behavior and thermal properties, UL-94 (Vertical burning and Horizontal burning), LOI, Cone calorimetry and TGA were carried out to investigate. An excellent flame retardancy was achieved at 40 wt.% loading of APP/ZB into jute/PP composites. V-0 classification level was achieved and 'no burning effect' was found which ensured self-extinguished (LOI- 28.8%) manner of flame-retardant jute/PP composite. A clear increment of LOI value was achieved up to 76.61% compared to unmodified jute/PP composite at a loading level of 30 wt.% of short jute fiber. Peak heat release rate (pHRR) was reached to 197.5 kW/m² with total smoke production (TSP) value of 10.5 m². Total heat release (THR) was 86.42 kW/m². Effective hear of combustion value was 23.47 MJ/kg. Tensile strength and flexural strength increased by 15.86% and 81.21%, respectively. However, impact properties were negatively affected due to the high loading of APP/ZB. 35.68% decrease was noticed. Density was increased 18.75% compared to unmodified short jute fiber at a constant loading level of 30 wt.%. Inclusion of flame-retardant additives increased wear rate as well as value of co-efficient of friction (COF) significantly. A maximum increase of 195.75% was observed due to high pin temperature with self-extinguished APP/ZB based jute/PP composites. COF value was moderate compared to ATH/ZB at a loading level of 40 wt.%. Thermogravimetric analysis (TGA) results showed the maximum degradation rate temperature of the composite structures containing 40 wt.% of APP/ZB to be 481.62 °C, which was approximately 16 °C higher than that of the unmodified jute/PP composite at a short jute fiber loading of 30 wt.%. Fourier-transform infrared (FTIR) spectroscopy results was showed no significant differences within chemical structures of APP/ZB or ATH/ZB compared to unmodified jute/PP composites which signifies flame retardants were acted just as a filler to improve flame retardancy without any sorts of drastic changes into produced composites.

ALEV GECİKTİRİCİ KATKILAR İLE DOĞAL LİF TAKVİYELİ KOMPOZİTLER GELİŞTİRİLMESİ

ÖZET

Kompozit malzemelerin/ürünlerin üretiminde kullanılan polimerlerin çoğunluğu petrol esaslıdır. Petrol kaynaklarının sonlu olması, depolama ve üretim sırasında çok fazla atık oluşturması büyük çevre problemlerine yol açmaktadır. Ayrıca, petrol esaslı polimerlerin geri dönüşüm seçeneklerinin çok sınırlı olması da sürdürülebilirlik konusunda endişeler oluşturmaktadır. Bu nedenle, petrol esaslı polimerlerin kullanımını mümkün olduğunca azaltmak, onların yerine biyolojik olarak parçalanabilir veya geri dönüştürülebilir kaynakların/çözümlerin araştırılması ön plana çıkmıştır. Geri dönüştürülebilirlik bakımından daha avantajlı olan termoplastik polimer matrisler ve doğal elyaf takviyeler ile oluşturulacak kompozit malzemeler sürdürülebilirlik konusunda birçok sektör için uygun bir çözüm olarak karşımıza çıkmaktadır. Bu malzemeler, ayrıca, hem maliyet etkinliği hem de yüksek verimlilik sunmaktadırlar.

Küresel otomotive endüstrisi de ticari uygulanabilirliği ve sürdürülebilirliği sebebiyle, doğal lif takviyeli termoplastik polimer matrisli kompozit malzemelere oldukça fazla ilgi göstermektedir. Geliştirilen bu doğal elyaf takviyeli termoplastik kompozitler pek çok marka araçların kapı panellerinde, gürültü izolasyon panellerinde, konsolda, tamponlarda, bagajda, koltuklarda vs. yaygın olarak kullanılmaktadır. Çevre dostu olması, hafifliği, ağırlığına oranla mukavemetinin yüksek olması ve iklim şartlarına dayanıklı olması gibi avantajları sebebiyle jüt lifi, polipropilen (PP) gibi termoplastik matrisli kompozit malzeme üretimi için çok uygun bir seçenek haline gelmiştir ve PP matrisi ile kullanımı gün geçtikçe artmaktadır. Ayrıca, alev geciktiricili malzemelerle katkılanmış jüt/PP kompozitler, oldukça güvenilir yeni ürünler olarak trend oluşturmaktadırlar. Fakat mekanik özelliklerden ödün vermeden alev geciktirici özellik elde etme konusu büyük önem taşımaktadır.

Genel olarak, polimer matrisin liflerle takviye edilmesi, elde edilen kompozit malzemelerin mukavemetini büyük ölçüde artırmaktadır ama selüloz esaslı lifler kullanıldığında selülozun artışı polimerin alev dayanımında negatif etkiye yol açmaktadır. Lignin ise alev dayanımını artırma etkisi göstermektedir. Lignin özellikle yanma esnasında duman absorblama özelliği göstermektedir. Selüloz esaslı bir lif olan jütün maksimum yükleme seviyesi sağlandıktan sonra jüt/PP kompozitlerine uygun katkı maddelerinin eklenmesi ile malzemeye alev geciktirici özellikler sağlanabilir. Bu tezde matris olarak PP, takviye olarak jüt lifi ve alev geciktirici katkı olarak halojen içermeyen malzemeler (Alüminyum trihidrid (ATH) ve amonyum polifosfate (APP) ile sinerjik bir etki oluşturmak için çinko borat (ZB)) kullanılarak kompozit plakalar üretilmiş ve bu plakalar şu test ve karakterizasyon metodlarıyla incelenmişlerdir: Çekme Testi, Üç-Nokta-Eğilme Testi, Izod Darbe Testi, Aşınma Testi, Yoğunluk Ölçümü, Taramalı Elektron Mikroskobu (SEM) görüntüleri, Fourier Dönüşümlü Kızılötesi Spektrometresi (FTIR) Analizi, UL-94 (Düşey

yanma ve Yatay yanma), LOI (Limit Oksijen İndeks) Testi, Koni Kalorimetre Testi ve TGA (termogravimetrik analiz).

Bu tezde, ekstrüzyon ve müteakip sıcak-pres işlemleri ile 80:20 oranında (ağırlıkça) %30 ve %40 ATH/ZB ve APP/ZB içeren alev geciktiricili jüt/PP kompozitler üretilmiştir. Üretimde önce jüt lifleri uygun boyutlarda kesilmiş ve öte yandan PP matrisi öğütülmüştür.

Arkasından, PP, jüt lifi ve alev geciktirici katkıları belli oranlarda karıştırılmış ve akabinde çift-vidalı ekstrüdere beslenmiştir.

Ekstrüderden çıktı olarak elde edilen homojen karışım daha sonra granül hale getirilmiş ve en sonunda granüller 8.5 ton ile 200 C’de 20 dakika boyunca sıcak-pres ile basılarak kompozit plakalar elde edilmiştir. Beslenen jüt lifinin mukavemeti düşürmesine sebep olmadan hem alev geciktirici özelliğinin artırılması hem de homojen bir şekilde fiberlerin dağıtılmasına çalışılmıştır.

Jüt/PP kompozitlerine alev geciktirici katkı maddeleri katmadan önce, farklı lif uzunluklarının jüt/PP kompozitin üzerine etkileri incelenmiştir. Bunun için %5 maleik anhidrit-aşılınmış polipropilen (PP) matrisi (MA-g-PP) içine ağırlıkça % 20 oranında uzun jüt lifi (LJF) ve kısa jüt lifi (SJF) eklenerek numuneler üretilmiş ve kısa lifli kompozitlerin mekanik özellikler (çekme ve eğilme mukavemetleri) bakımından uzun lifli kompozitlerden daha iyi bir performans gösterdiği tespit edilmiştir. Kısa lif numunelerinin maksimum çekme ve eğilme mukavemetleri, sırasıyla, $37,32 \pm 0,75$ MPa ve $34,68 \pm 1,70$ MPa olarak bulunmuştur. Çekme ve eğilme modülleri ise 1811 ± 81 MPa ve 1181 ± 95 MPa olarak ölçülmüştür. İlk mekanik özellik denemelerinde, ağırlıkça %20 lif içeren kısa lifli kompozitlerin uzun lifli olanlardan daha iyi performans gösterdiği sonucuna ulaşıldığı için sonraki denemeler %20 kısa lif içeren kompozitlerle ve lif yükleme oranlarını %60’a çıkararak yapılmıştır. Mekanik analiz sonuçlarına göre jüt oranı arttıkça çekme mukavemeti ve çekme geriminin azaldığı gözlemlenmiştir. PP matrisine ağırlıkça % 60 kısa jüt lifi yüklendiğinde çekme mukavemetinde büyük ölçüde bir düşüş ($19,50 \pm 2,04$ MPa) kaydedilmiştir. Diğerleri ile kıyaslandığında, %40’lık kısa lif yüklemesine kadar olan yüklemelerde eğilme mukavemeti yüksek değerini hep korumuş ($38,98 \pm 3,45$ MPa) ancak %60’lık kısa lif yüklemesinde eğilme mukavemeti en düşük değeri ($37,77 \pm 1,25$ MPa) vermiş, çekme mukavemeti ise $19,50 \pm 2,04$ MPa’ya ulaşmıştır. SEM cihazı ile yapılan morfolojik analiz sonuçlarına göre ağırlıkça %30’luk kısa lif yüklemesi durumunda en iyi matris-elyaf yapışması gerçekleştiği görülmüş ve bu numuneye ait elde edilen optimum mekanik özelliklerin (Çekme Mukavemeti: $29,5 \pm 2,74$ MPa, Çekme Gerinimi: $4,97 \pm 0,84\%$, Young Modülü: 1517 ± 198 MPa, Eğilme Mukavemeti: $38,17 \pm 1,9$ MPa, Eğilme Gerinimi: $7,18 \pm 1,17\%$ ve Eğilme Modülü: 1488 ± 88 MPa) bu sayede elde edildiği kanaatine varılmıştır.

Mekanik özelliklerden ödün vermeden alev geciktirici özellikler kazandırmak için, optimum olarak tespit edilen ağırlıkça %30 lif yükleme değerini sabit tutarak yine ağırlıkça %30 ve %40 oranlarında ATH/ZB ve APP/ZB alev geciktirici katkıları kullanılarak kompozit numuneler üretilmiştir. Mekanik analiz sonuçlarına göre alev geciktiriciler, maksimum gerilim ve gerinim değerlerinde düşüşe neden olurken Young Modülü değerlerinde iyileştirme sağlamıştır. Diğer taraftan alev geciktiricilik sonuçlarına göre APP/ZB katkısı daha olumlu etki göstermiştir. Yanma testlerinde APP/ZB’nin, jüt/PP kompoziti üzerindeki kömür oluşumunu önemli ölçüde etkilediği gözlemlenmiştir. Jüt/PP kompoziti içine ağırlıkça %40’lık APP/ZB yüklemesi yapıldığında mükemmel alev geciktiricilik elde edilmiştir. V-0 sınıflandırmasında “hiç

yanma etkisi yok” olarak tabir edilen seviyeye ulaşılmıştır ki bu kendini-söndüren (LOI: %28,8) alev geciktiricili özelliğe sahip bir jüt/PP kompozit malzemesi üretmiş olmak demektir. Modifiye edilmemiş jüt/PP kompoziti ile kıyaslandığında, %30’luk kısa lif yüklemesi yapıldığında LOI değerinde %76,61 gibi büyük bir artış sağlanmıştır. Maksimum ısı salımı (pHRR) 197,5 kW/m², toplam duman üretimi (TSP) 10,5 m², toplam ısı salımı (THR) 86,42 kW/m² ve efektif yanma ısısı 23,47 MJ/kg olarak bulunmuştur. Kopma gerinimi 15,86%, eğilme gerinimi ise 81,21% olarak yükselmiştir. Ne var ki, darbe özellikleri yüksek orandaki APP/ZB yüklemesinden olumsuz olarak etkilenmiştir (35,68%’lik bir azalma meydana gelmiştir). Modifiye edilmemiş kısa lif ile kıyaslandığında, %30’luk sabit yüklemeye yoğunluk 18,75% artmıştır. Alev geciktiricilerin eklenmesi ile aşınma oranı sürtünme katsayısı önemli ölçüde artmıştır. Kendini-söndüren APP/ZB esaslı jüt/PP kompozitte, yüksek pim sıcaklığı sebebiyle, 195,75%’lik maksimum bir artış gözlenmiştir.

Ağırlıkça %40’lık yüklemeye ATH/ZB ile kıyaslandığında sürtünme katsayısı (COF) değeri orta seviyelerde çıkmıştır. Termogravimetric Analiz TGA sonuçlarına göre, %40 APP/ZB içeren kompozitin maksimum bozulma oran sıcaklığı 481,62 C° olup, ağırlıkça %30 kısa lif yüklü modifiye edilmemiş jüt/PP kompozitininkinden yaklaşık 16 C° daha yüksektir. FTIR spektroskopisi incelemelerinde, APP/ZB veya ATH/ZB’nin kimyasal yapılarında modifiye edilmemiş jüt/PP kompozitlerle kıyaslandığında kayda değer bir fark bulunmamıştır ki bu da alev geciktiricilerin, kompozitler üzerinde fazla bir değişiklik yapmaksızın sadece bir dolgu malzemesi görevi görerek alev geciktiricilik özelliğini iyileştirdiğini göstermektedir.

1. INTRODUCTION

1.1 General

Nowadays, the rate of consumption of natural fiber based thermoplastic polymer composites is in the peak due to its diverse field of applications depending on high efficiency along with cost effectiveness. A sheer number of fiber-based composites rely on carbon and glass fibers. However, natural fiber has been brought an efficient way due to its applicability specially in automotive sectors with thermoplastic and thermoset materials. Using polymer materials alone, is a great matter of concern for environment. A big question mark comes forward when it comes to the subject of 'Sustainability'. The reason is simple. The prime source of these polymer materials is 'petroleum oil' and it is hazardous due to enormous waste generation in landfills and having lack of recycling options. Tones of microplastics have been produced from these polymer materials which have been making our daily lives more vulnerable.

To ensure sustainability, commercial industries should follow the path of selecting alternatives like bio-degradable and recyclable sources from both end: polymers and fibers. To ease the way of commercial industries, researchers have been greatly focused on eco-friendly solutions based on numerous natural fiber composite materials without compromising mechanical and physical properties. Flame retardant based natural fiber composites have been also introduced into the commercial industry from the concern of environment and safety issues. However, there should be a threshold limit of infusing natural fibers and flame retardant fillers within polymer materials to minimize the cost of advanced commercial end product without compromising mechanical, physical and flame retardant properties.

In the second chapter of this thesis, gives an overview of literature reviews on the natural fibers, jute and their surface modification methods for polymer matrixes. Then, market demand and supply of natural fibers have been discussed briefly. Later on, applications of polymer materials in natural fiber composites have been presented.

To understand flame retardancy of jute/PP composites, chemical structure of polypropylene and thermal stability of cellulose have been introduced thoroughly. Under the section of flame retardancy, numerous flame retardant additives have been discussed just after paying a special attention towards flame growth, flame retardant mechanism and synergism. It is important to understand the combustion and burning behavior of thermoplastic materials especially polypropylene. All possible factors and parameters regarding combustion and burning behavior have been presented and discussed accordingly. Moreover, flame retardant mechanism of ATH/ZB and APP/ZB have been put under the radar of explanations. At the very last part of the second chapter, flame retardancy of natural fiber composites, flame retardancy of thermoplastic based natural fiber composites and flame retardancy of different formulations related to only jute/PP composites have been explained.

In the third chapter of this thesis, gives the entire road map of producing flame retardant fillers-based jute/PP composites. Two different set of formulations have been followed to reach towards final conclusion. Fourth chapter emphasizes on results achieved from morphological analysis, mechanical tests, TGA, FTIR and flame retardant tests. Effect of LJF and SJF have been discussed mutually before ensuring maximum level of jute fiber loading. Then, after having a discussion over mechanical tests, flame retardant fillers-based jute/PP formulations have been established and their morphological analysis, mechanical test results, TGA, FTIR and flame retardancy results and discussions have been presented.

1.2 Problem Statements

Within the scope of this thesis, several issues have been taken care of as follows:

- Which type of jute fiber (long or short) is more suitable for compounding via twin extruder?
- How do the mechanical and physical property's function while compounding with high amount of jute fiber?
- What happens to flame retardancy and mechanical and physical properties while increasing fiber amount with the combination of flame retardant fillers and synergists at similar manner?

- What are the effects of zinc borate (ZB) as a synergist in combination with aluminium trihydrate (ATH) and ammonium polyphosphate (APP) individually?
- Is it possible to use 60 wt.% fillers along with polypropylene without compromising mechanical properties, physical properties & flame retardant properties?

1.3 Aim of Thesis

The aim of this thesis was to achieve an optimum formulation for the development of natural fiber (Jute) reinforced composites with different flame retardant fillers (Aluminium Trihydrate /Zinc Borate, Ammonium Polyphosphate/Zinc Borate) in the presence of Polypropylene matrix without compromising mechanical and physical properties. In order to reach the goal, following issues have been focused on:

- To produce highly efficient flame retardant composites based on jute fiber and polypropylene matrix from the aspects of mechanical flame retardant properties.
- To investigate the mechanical performance of short jute fiber (SJF) and long jute fiber (LJF) along with polypropylene (PP) based on tensile and flexural tests.
- To ensure interfacial bonding between polypropylene and jute fibers in the presence of maleic anhydride grafted polypropylene (MA-g-PP).
- To find the maximum limit of short jute fiber (SJF) into polypropylene matrix from the mechanical aspects (tensile and flexural strength values).
- To investigate furthermore about mechanical performances, impact strength values, density and wear rate values have been discussed.
- To find the maximum limit of two different flame retardant filler combinations (Alumina Trihydrate /Zinc Borate, Ammonium Polyphosphate/Zinc Borate) in the presence of short jute fiber (SJF) and polypropylene matrix to lessen the usage of polypropylene.
- Synergic effect of zinc borate (ZB) into jute fiber reinforced polypropylene composites in the presence of aluminium trihydrate (ATH) and ammonium polyphosphate (APP).

- To investigate surface morphology, SEM (Scanning Electronic Microscope) have been carried out.
- To investigate thermal stability of jute/pp composites along with flame retardant fillers, TGA (Thermal Gravimetric Analysis) have been carried out.
- To investigate adhesion behavior and interfacial bonding behavior of jute/PP, flame retardants-based jute/PP composites, FTIR (Fourier Transform Infrared) have been carried out.
- To reduce the ratio of polypropylene by increasing the ratio of jute and flame retardant fillers.

The hypothesis of this study is to find an optimum fiber loading balance within jute/PP composites and to achieve optimum flame retardancy without compromising mechanical properties.

2. LITERATURE BACKGROUND

2.1 Natural Fibers and Jute

Current industries are shifting towards sustainability with its great innovation power coming from science and technology [1] to replace nonrenewable resources which is a bigger threat for our environment. As a replacement of nonrenewable resources, different types of natural fibers are taking place with growing interest. Applications of natural fibers can be done within three key ways as follows:

- i. Textiles, paper and fabrics
- ii. Biofuel &
- iii. Reinforcer for composites

Classification of natural fibers depending on lignocellulosic fibers, animal fibers and mineral fibers (Figure 2.1). Lignocellulosic fibers are mainly based on wood fibers (hardwood and softwood) and non-wood fibers (cotton, kapok, loofah, sisal, banana, pineapple, abaca, henequen, flax, ramie, hemp, jute, kenaf, coir, oil palm, rice, barley, wheat and maize) which are having major market share around the world. Animal fibers are being also used in purpose. Especially silk fibers have been used thoroughly besides wool, human hair, feathers, horse hair and alpaca hair. Moreover, there are mineral fibers (amosite, crocidolite, tremolite, actinolite, anthophyllite and chrysolite) which are in use. Lignocellulose fibers are in great demand as a reinforcement material with thermoset and thermoplastic polymers for industrial applications due to its economic and high-end mechanical properties like strength and stiffness [2].

There are noticeable advantages of using natural fibers as composites. Such as:

- Low density
- Good wear properties
- Biodegradable
- Renewable

- Low cost
- Design compatibility
- Good alternative to glass fibers and carbon fibers
- Less energy consumption
- Good thermal and insulation &
- Lower toxicity level

However, some disadvantages are present as well. Such as:

- Hard to replace synthetic fibers
- Extreme moisture absorption
- Extreme wettability
- Bond creation is tough in between fiber and matrix
- Fiber dimension is critical
- Low fire resistance &
- Poor thermal resistance [2,3]

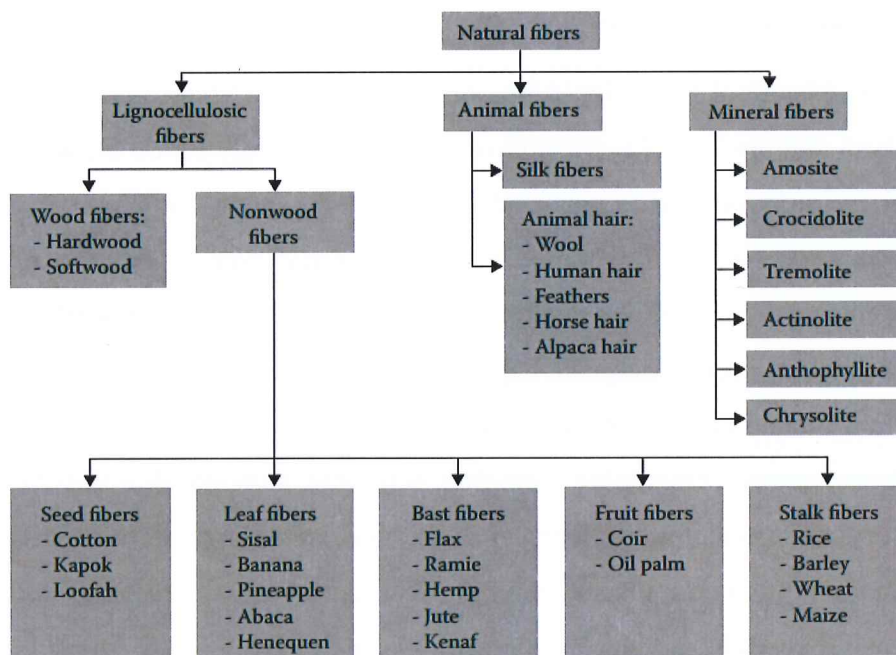


Figure 2.1: Classification of natural fibers [2].

If adhesion is not so significant within fibers, its length becomes a critical parameter which is directly related to composite strength. Natural fibers are very flexible with polymers like synthetic or inorganic in terms of low density, strength & stiffness [2].

Jute fiber is one of the key members of natural fiber segments due to its great applicability within polymer matrixes.

It has been used into automotive industry from 1940 since it has been always a potential candidate to take the position of glass fibers. It is having similar advantages of basic natural fibers [4]. Such as annually renewable fiber, cost effective, possible full utilization, fully environmental, during incineration, causes very minimum impact over surroundings, good amount of stability, fracture resistant, abrasion resistant, lightweight, non-significant elongation, higher CO₂ absorption [5], good insulation and anti-static properties and low amount of thermal conductivity [4].

Jute fiber is having significant disadvantages as well. Such as:

- Moisture absorption ability
- Insignificant drapability & crease resistance
- Brittle nature
- Decreasing mechanical strength due to wetness [4]
- Degradation level is close to 200°C [5]
- Poor mechanical properties regarding jute composites [6].

Natural fibers are classified within three core sections: lignocellulose fibers, animal fibers and mineral fibers. Lignocellulose fibers are also named as plant fibers. Lignocellulose fibers or plant fibers are having a combination of cellulose fibrils and lignin matrix. Plant fibers are generally two types. One is primary plant (Jute, hemp, kenaf etc.) and the other is secondary plant (coir, pineapple, oil palm etc.). The key issue of primary fibers is their 'fiber content'. Secondary plant fibers come forward as a 'By product' with different initial processes. Natural fibers are being obtained from plants that constitutes of cellulose, hemicellulose, lignin, pectin and wax. Figure 2.2 represents the basic structure of natural fibers. Crystalline cellulose is having 80% crystalline regions. Hemicellulose of natural fibers are made of polysaccharides which are highly branched connected with cellulose within structure of natural fibers. Lignin is basically an amorphous that makes cell wall stiffer to protect as a shield mainly for cellulose. Chemical structure of plant-based fibers is very close to each other but degree of polymerization varies within cellulose of fibers [7]. Table 2.1 and Table 2.2 represents mechanical properties and chemical composition of different types of common lignocellulosic fibers.

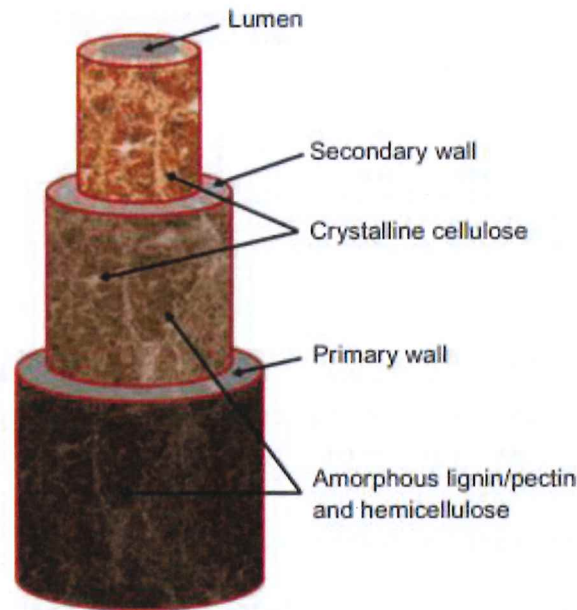


Figure 2.2: Basic structure of natural fiber [7].

From Table 2.2, It is very clear to see that percentage of cellulose is higher compared to hemicellulose, lignin and waxes [2]. But the key issue of cellulose is its ‘thermal resistance behavior’. It is very poor. Overall properties of natural fibers are depending on numerous constraints such as:

- Structure
- Chemical Composition
- Angle of microfibrils
- Dimension of cells &
- Defect within fibers

Before choosing natural fibers to produce composites, it is important to know about each property and their growing resources [7].

Origin of jute fiber is lignocellulosic jute plant. Multiple cellular structures exist within bast type of jute fibers. Within the cellular structure of jute fibers, there are multiple compositions available such as lignin, cellulosic component, oils, waxes and a small amount of fat. Existence of a strong network can be found due to these components within stem portion named as ‘strand’. A formation of mesh type network is visible to create long type of jute fibers. However, growth formation of jute fibers differs to country to country due to weather conditioning and fiber types.

Table 2.1: Mechanical properties of natural fibers [2].

Fiber	Density (g/cm ³)	Length (mm)	Diameter (μm)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Specific Modulus (Approx.)	Elongation (%)
Abaca	1.5	-	-	400-980	6.2-20	9	1-10
Bamboo	0.6-1.2	1.5-4	25-40	140-800	11-32	25	2.5-3.7
Banana	1.35	300-900	12-30	500	12	9	1.5-9
Coir	1.15-1.46	20-150	10-460	95-230	2.8-6	4	15-51.4
Cotton	1.5-1.6	10-60	10-45	287-800	5.5-12.6	6	3-10
Flax	1.4-1.5	5-900	12-600	343-2000	27.6-103	45	1.2-3.3
Hemp	1.4-1.5	5-55	25-500	270-900	23.5-90	40	1-3.5
Jute	1.3-1.49	1.5-120	20-200	320-800	8-78	30	1-1.8
Kenaf	1.4	-	-	223-930	14.5-53	24	1.5-2.7
Oil Palm	0.7-1.55	-	150-500	80-248	0.5-3.2	2	17-25
Palf	0.8-1.6	900-1500	20-80	180-1627	1.44-82.5	35	1.6-14.5
Ramie	1.0-1.55	900-1200	20-80	400-1000	24.5-128	60	1.2-4.0
Sisal	1.33-1.5	900	8-200	363-700	9.0-38	17	2.0-7.0

Table 2.2: Chemical properties of natural fibers [2].

Fiber	Cellulose (wt.%)	Hemicellulose (wt.%)	Lignin (wt.%)	Pectin (wt.%)	Waxes (wt.%)	Micro Fibriller Angle (Degrees)	Moisture Content (wt.%)
Abaca	56-63	20-25	7-13	1	3	-	5-10
Bamboo	26-65	30	5-31	-	-	-	-
Banana	63-67.6	10-19	5	-	-	-	8.7-12
Coir	32-43.8	0.15-20	40-45	3-4	-	30-49	8
Cotton	82.7-90	5.7	<2	0-1	0.6	-	7.85-8.5
Flax	62-72	18.6-20.6	2-5	2.3	1.5-1.7	5-10	8-12
Hemp	68-74.4	15-22.4	3.7-10	0.9	0.8	2-6.2	6.2-12
Jute	59-71.5	13.6-20.4	11.8-13	0.2-0.4	0.5	8.0	12.5-13.7
Kenaf	31-72	20.3-21.5	8-19	3-5	-	-	-
Oil Palm	60-65	-	11-29	-	-	2-46	-
Palf	70-83	-	5-12.7	-	-	14.0	11.8
Ramie	68.6-85	13-16.7	0.5-0.7	1.9	0.3	7.5	7.5-17
Sisal	60-78	10.0-14.2	8.0-14	10.0	2.0	10-22	10-22

Several key factors are affecting directly not only to the structure of jute fiber but also to the length of jute fiber and chemical components of jute fiber. These factors are as follows:

- ✓ Available environment
- ✓ Conditioning weather
- ✓ Available defect types of fiber
- ✓ Age of plants
- ✓ Fiber extraction process
- ✓ Fiber modification process

Generally, density of jute fibers is low. The prime reason behind it 'existence of lumen'. Lumen is like a cavity or hole within cellular structure of jute fibers. All unit cells are linked to other unit cells of jute fiber through middle lamella. Within the composition of jute fiber, there are many types of polysaccharides and lignin. In the composition of polysaccharides, two different types of cellulose available. One is hemi-cellulose and another one is alpha cellulose. Chemical composition of jute fiber is largely dependent on cellulose. There are glucose rings within cellulose structure. Polymerization occurs linearly through these covalent bond-based glucose rings and creation of cell wall happens which is actually 'jute fiber'. Basically, determination of physical and mechanical properties is largely dependent on these covalent based glucose rings. Available hydroxyl groups of glucose and chain of cellulose bonds altogether with water molecules via a chemical interaction with the aim of creating strong hydrogen bonds. Jute fiber is not only hydrophilic but also becomes crystallized in a unorganized manner with 3D structure because of the hydrogen bonds. Water absorption is a basic phenomenon of jute fiber because of this hydrophilic nature of jute fiber which is highly responsible for swelling. Moreover, acidic behavior of jute fiber can be easily integrated because of hemicellulose and poly-uronic acid. Micro-fibril zone from the cross section of jute fiber is basically largely ordered zone and greatly packed (completely crystallized). However, unpacked or easily packed zones are also available within same microfibril zone. Those are named as 'fringed fibril zone' (completely amorphous). Angular position of these microfibril decides mechanical and physical properties of jute fibers.

From cross-section of jute fiber, it is clearly visible that two different types of cells of cellulose are available for jute fiber such as primary wall and secondary wall (Figure 2.3). Formation of microfibril is basically the bridge of these two walls [8].

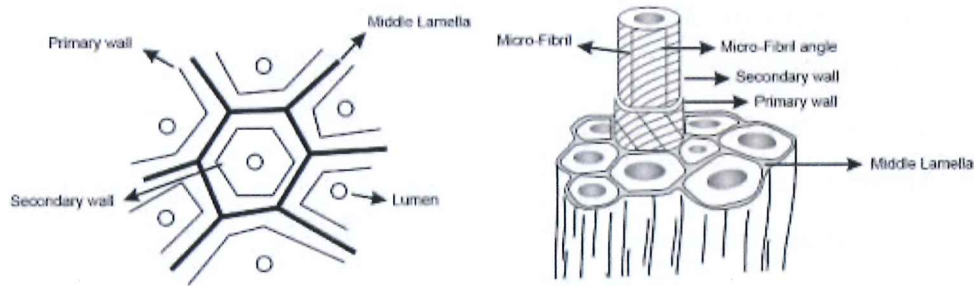


Figure 2.3: Cross-section of jute fiber [8].

2.2 Surface Modifications of Natural Fiber Polymer Composites (NFPCs)

In the way of improving mechanical properties of natural fiber polymer composites, absorption of moisture is a key factor which needs to be improved. Due to the moisture absorption behavior of natural fibers, losing of stability becomes very dominant which causes biodegradation of fibers and narrow down the range of applications of natural fibers industrially. To overcome this issue, with time and technology several surface modifications processes have been innovated with specific purposes specially to improve compatibility between different type of natural fibers and matrixes with compressive adhesion properties. Table 2.3 represents different types of surface modification processes along with its positive aspects [9].

Several studies have been carried out on physical surface modification methods to improve fiber/matrix compatibility with the aim of improving mechanical properties. DBD plasma treatment is one of the processes by which a very common natural fiber ‘Banana’ can be utilized regarding to increase mechanical properties very effectively with proper adhesion. Hydrophilic behavior of banana fiber has been in a positive node with this surface modification process [10]. ‘Jute’ fiber is another common type of natural fiber which can be go through plasma type surface treatment along with numerous thermoplastic polymer matrix like PLA (Polylactic acid) to have better mechanical properties. Alkali treatment is also very much applicable for the same purpose with improved adhesion.

Surface modification of jute fiber can be done with low temperature oxygen plasma treatment as well for HDPE (High density polyethylene) matrix. Properties like interlaminar shear strength have been improved significantly up to 47%. Not only that but also, flexural strength (45.6 MPa) and flexural modulus (1244.5 MPa) properties have been increased significantly by this method for jute/HDPE composites. Moreover, 'Hemp' fiber have been found fruitful with PP (Polypropylene) matrix to improve mechanical properties like tensile strength, young modulus and flexural strength with corona plasma treatment.

Table 2.3: Different types of surface modification processes and their advantages for NFPCs [9].

Modification Method	Type	Advantages
Plasma Treatment	Physical	Flame retardancy
Silane Treatment	Chemical	Tensile strength
Corona Treatment	Physical	Hydrophilic fiber/Hydrophobic polymer
Alkali Treatment	Chemical	Adhesion, Roughness
MA	Chemical	Interfacial bonding
Acetylation	Chemical	Hydrophobic behavior
Benzoylation	Chemical	Thermal stability

Various types of chemical-based surface modifications have been carried out to improve fiber/matrix compatibility apart from physical methods. Alkali treatment is one of them. Hemp/PP/MA-g-PP composite have been prepared with alkali treatment to increase interfacial shear strength properties. Flax/PLA composites have shown lean manufacturing process through different chemical treatments like corona, alkali and silane. Each of these treatments have been proven as a prominent way to improve adhesion and fiber/matrix compatibility as well as mechanical properties (Tensile strength, flexural strength and surface roughness).

Alkali treatment, benzoylation treatment and silane treatment have been proved as a fruitful one for banana/PP composites regarding to improve thermal properties like conductivity and some other thermophysical properties.

In addition to that, there are enough proofs of using pineapple leaves and kenaf fibers to prepare composite materials by using alkali, silane, alkali-silane treatment for the purpose of increasing mechanical properties. Kenaf fibers are having good opportunity cost to use acetylation, isocyanate, MA, potassium permanganate treatment for the same purpose with PU (Polyurethane). Only MA did not show good improvement from all of them. Fiber surface can be improved with purity via alkali treatment easily from all other options discussed above. Because this process modifies the entire structure of natural fibers to improve water resistance behavior. Separation of cellulose with better adhesion in between fiber and matrix, alkali influences most. Reason is ‘removal of impurities from fiber’. Chemical treatments improve adhesion and adhesion is directly linked to synergetic effect of produced composites. This is another key reason to have chemical treatment before producing composites. Figure 2.4 shows effect of different chemical treatment on sisal fiber [9].

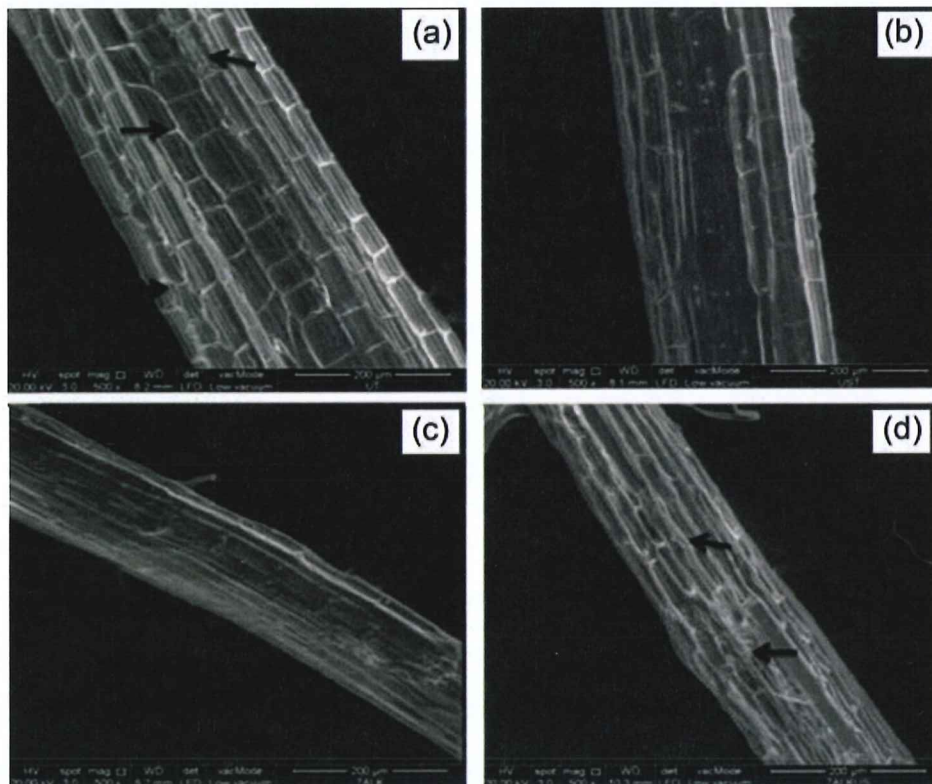


Figure 2.4: Chemical treatment of sisal fibers-(a) untreated, (b) alkali-treated, (c) ultrasound treated and (d) alkali-ultrasound treated [9].

2.3 Market Research and Applications of Natural Fibers and Jute Fibers

Based on application areas, major shares of natural fibers belonging towards sugarcane bagasse, bamboo and jute fibers (Table 2.4).

Table 2.4: Total production of commercial natural fibers [11].

Fiber Type	World Production (10 ³ ton)
Sugarcane bagasse	75000
Bamboo	30000
Jute	2300
Kenaf	970
Flax	830
Grass	700
Sisal	378

There is a great demand of commercial natural fibers within industries. However, that demand is to meet towards consumers via four critical stages as follows:

- i. **Growth of the plant:** It is greatly depending on selection of correct plant species, cultivation of plant, fiber placement within plant, cultivation area and its climate.
- ii. **Cropping:** It is totally depending on fiber ripeness which is largely depending on internal fiber structures like cell wall thickness, adhesion of fibers etc.
- iii. **Separation of fibers:** It is done via decortication method and retting method
- iv. **Supply chain management:** Depending on fiber age, storage and transportation system involves towards end customers [11].

Current industries are facing a great challenge to ensure usage of renewable sources to make our environment more ecofriendly. Natural fiber composite (NFC) materials are having a great contribution in this case.

Entire natural fiber composite market is categorized by four segments as follows:

- i. **Fiber Segment:** Fiber segment is in combination of two types of composite materials such as wood and non-wood. Non-wood composite materials are having different type of natural fibers including jute, kenaf, sisal, hemp, flex and so on. Compared to wood materials, demand of wood composites is way higher in current market due to easy processability in molding, trimming and framing for construction and automotive sectors.
- ii. **Polymer Segment:** Polymer segment is classified in two sections including with thermosets and thermoplastics. From application aspects, thermoset is largely sharing its position compared to thermoplastic. To make lightweight composite materials with high mechanical properties, thermoset and thermoplastic polymers are playing a major role when it comes to use natural fibers specially in sporting goods, automotive and wind energy sections.
- iii. **End user Segment:** Automotive industry is the key end user of polymer based natural fiber composites. Major shares in automotive industries are holding by wood (37%), Cotton (24%), Flax (19%), Kenaf (8%), Hemp (5%) and others (7%) in 2021. There are several reasons available such as: environmental issues, reduction of CO₂ emission, ensuring lightweight manners, ensuring high mechanical properties, developing fuel economy, lessen the production cost by 20%.
- iv. **Geography Segment:** North America (USA, Canada, Mexico) is the largest consumer of natural fiber composite materials specially in construction materials. Manufacturing industries are turning into green units. For that reason, North American countries (USA, Canada, Mexico) along with European countries (Germany, UK, Italy, France, Spain) are bringing new laws to implement green materials. Germany is a leading producer of cars. This is one of the concerns for European zone. Asian countries like Japan are also following the path of Germany to ensure the usage of recyclable materials to make automotive parts for fuel efficiency.
From Asian Pacific zone (China, Japan, Australia, India, Indonesia, South Korea), China is one of the largest consumers of wood-based composites. A significant portion of the world (Latin America, Africa, Middle East) is also using natural fiber composite materials like others [12,13].

Total production of natural fiber was around 33 million tons during 2013 with the economy value of US\$60 billion. Cotton had been dominant as usual (26 million tons-US\$45 billion). Production of jute fiber was around 3.3 million tons (US\$ 2 billion) and wools had been manufactured around 1.2 million tons (US\$ 8 to 9 billion). The least production was for coir fiber (900,000 tons). Manufacturing capacity of other natural fibers had been around 1.6 million tons (US\$ 3 billion)[14]. From 2016 to 2017 economical value had decreased (US\$ 40 billion) due to limitation of production capacity [15]. However, after covid growth of production of natural fibers and its economic value have been increased significantly up to US\$ 66 billion. For 2023, economic projection value has been US\$ 70.13 billion at a CAGR (compound annual growth rate) of 5.3%. US\$ 85 billion have been projected for 2027 based on current production rate and economic value [16]. Natural fiber based composite market have been around 4.4 billion during 2022 and it is expected to have an exponential growth at a CAGR of 7.7% in between 2022-2028 [17].

Application range of natural fiber-based composites materials can be segmented by four major sections such as-automotive, electrical and electronics, sports components and construction components. Key applications are mainly based on some core manufacturing processes including injection molding, compression molding and extrusion or melt compounding (Figure 2.5) [18-19]. Major fibers for the application of these areas (Table 2.4) are based on flax, kenaf and jute fibers. Composite materials based on glass and carbon fibers are segmented by market and values as follows:

- ✓ Automotive (\$2.7 Billion)
- ✓ Marine (\$0.5 Billion)
- ✓ Aerospace ((\$2.5 Billion)
- ✓ Construction piping and tank (\$5.2 Billion)
- ✓ Energy sector (\$2.0 Billion)
- ✓ Miscellaneous consumer goods (\$1.1 Billion)

Within all of these markets, natural fiber based composite materials are being introduced remarkably. Lightweight behavior and strength of composite parts are important. Such that possibility of composite penetration is larger in marine sector (68%) and energy sector (38%). However, scenario of transportation or automotive industry is gradually changing.

Automotive industry is the major market of natural fiber-based polymer composites. There are many major companies are having keen interest in application of natural fibers due its ability to replace glass fibers.

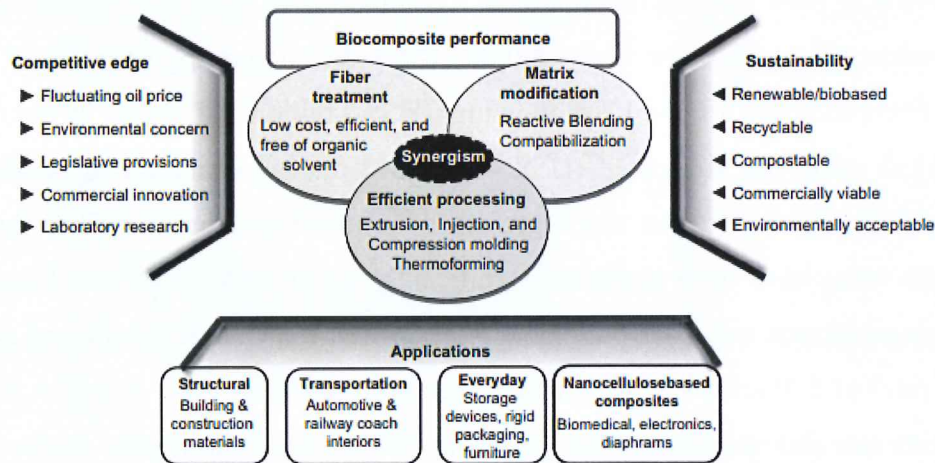


Figure 2.5: Overview of bio-composite [19].

Audi, BMW, Porsche, Renault, Ford, Rover, Toyota, Mercedes, Volkswagen, Volvo and so many others are making automotive parts including both interior and exterior parts based on natural fibers because of its flexibility, lightweight and cheap manner. Table 2.5 represents application areas based on natural fiber polymer composites focused by key automotive manufacturers [9].

Production capacity of jute fiber is around 3.2 million tons [20]. From Table 2.6, It is evident that- From last 10 years, Bangladesh, India and China are largest producers of jute fiber due to its weather conditioning. Due to global change in weather day by day production environment of India is largely improving compared to Bangladesh. However, concentration on producing jute fiber becoming less by China. There are some other countries involving to produce jute fiber at low level of capacity such as Uzbekistan, Nepal, Myanmar, Thailand, Sudan, Egypt and Vietnam. In terms of export value (Table 2.7) Bangladesh is in significant growth compared to others [21].

Application range of jute fibers are largely depending on usage. For traditional usage, jute fibers are being mainly as for packaging, roofing, tenting and so on. For modern usage, lot of consuming goods are being produced via jute fibers. Such as- school bags, shoe, toys etc. Modern day composite materials are largely based on jute fibers especially automotive sectors, medical sectors (personal protective equipment).

As a high-tech use, geotextile materials have been in trend nowadays which is replacing non-biodegradable, non-ionic, on-photodegradable geotextile materials [21].

Table 2.5: Application areas of NFCs by leading automotive manufactures [9].

Manufacturer	Model	Application Areas
Audi	A2, A3, A4, A6, A8	Seat backs, back door panel
BMW	I series	Door panels, door trim
Volvo	C70, V70	Cargo floor tray
Renault	Clio, Twingo	Rear parcel shelf
Ford	Freestar	Door panels
Rover	2000	Insulation
Toyota	Brevis, Harrier, Raum	Door panel
Mercedes Benz	Mercedes A	Bumper, insulation
Volkswagen	Golf, Passat, Bora	Door panel, seat back

Table 2.6: Production growth of jute fiber in between 2010-2019 [21].

Year	Jute Production Quantity (Tone)			
	World	Bangladesh	India	China
2010	2,828,732	923,464	1,799,100	40,000
2011	3,582,789	1,523,315	1,960,380	43,500
2012	3,453,600	1,452,044	1,912,000	39,400
2013	3,418,366	1,391,000	1,944,000	35,500
2014	3,395,972	1,349,000	1,968,000	32,480
2015	3,260,996	1,399,000	1,789,200	30,740
2016	3,264,150	1,344,000	1,850,510	30,750
2017	3,442,372	1,496,216	1,877,760	30,000
2018	3,404,261	1,613,762	1,726,380	27,110
2019	3,375,884	1,600,474	1,709,460	29,934

Table 2.7: Export data of jute fiber in between 2010-2019 [21].

Year	Export Value (in thousand USD)			
	World	Bangladesh	India	China
2010	261,179	191,351	33,457	232
2011	290,736	248,147	17,729	184
2012	236,668	194,000	10,163	121
2013	187,009	134,247	9143	249
2014	166,954	107,416	15,811	135
2015	190,046	123,473	10,558	158
2016	234,527	182,087	11,660	417
2017	188,261	137,524	8154	513
2018	154,330	119,840	5573	116
2019	171,084	107,247	12,854	101

2.4 Polymers in Natural Fiber Composites (NFCs)

Environmental pollution has become an invisible threat to the world with visible manners due to the continuous application of polymers, synthetic fibers and additives (8300 million tons) from the period of 1950 to 2015 and it is still ongoing. Many approaches like incineration, landfilling, recycling have been taken to reduce the amount of these materials. More than half of plastics (4600 million tons) have been thrown to landfills, 7% (700 million tons) of these plastics have been incinerated & the number of recycled plastics is 500 million (6%). Consumption of these plastics have been encountered by packaging (146 million tons), building & construction, textiles (65 million tons), consumer & institutional products (59 million tons), transportation (27 million tons), electrical & electronic (18 million tons), industrial machinery (3 million tons) and other sectors (47 million tons) [22]. These plastics are composed of numerous polymers.

Within the industry Polypropylene (PP), Low-density polyethylene (LDPE), High-density polyethylene (HDPE), Polyester, Polyamide & acrylic fibers, Polyvinyl chloride (PVC), Polyethylene terephthalate (PET), Polyurethanes (PU), Polystyrene (PS) & additives are being produced where majority portions are being captured by PP (68 million tons), LDPE (64 million tons), Polyester- Polyamide & acrylic fibers (59 million) & HDPE (52 million tons) [23]. Majority waste generations (194 million tons) have been done by these four types within China, United States, Germany, Egypt & India due to mismanagement [22,24]. Mean product lifetime of these plastics is in between 0.5 years to 35 years depending on consumption sectors [25]. Prime reasons of using these polymeric materials are their attractive properties including lightweight, easy processibility & cost effectiveness [26]. To reduce the usage of these polymeric materials several attempts have been taken care of by many industries including construction, automotive sector, biomedical and packaging by introducing green composites based on natural fibers for ensuring sustainable environment for global interest [27]. Natural fiber composites (NFCs) are having competitive advantages over glass and carbon fibers in terms of cost and energy. There is a huge window opportunity to incorporate natural these fibers as reinforcers within matrix material like thermoset or thermoplastics or any kind of natural or bio-based polymers to make natural fiber polymer composites to achieve low production cost in terms of available raw materials and to achieve better mechanical properties compared to glass or carbon fibers [28]. Table 2.8 represents information about some commercial biobased polymers to incorporate with natural fibers to make green composites [29].

There is no doubt of acknowledging importance of green composites based on polymer matrix and natural fibers. Natural oil-based resin materials are a good replacement of petrochemical based thermoset resins. However, there is an extreme lack of production capacity of compatible bio-based thermoset resins. Currently, growing interest have been noticed in the usage of ‘Soyabin-oil’ as a bio-based resin.

Table 2.9 represents a comprehensive list of bio-based resins which can be used for natural fibers to make green composites by different manufacturing methods (Vacuum-assisted resin transfer molding, resin transfer molding, sheet molding and compression molding).

Table 2.8: List of commercial bio-based polymers [29].

Polymer	Biobased Polymer Fraction	Biobased Content (%) ^a
Polylactic Acid (PLA)	L-Lactic acid	Up to 100
Polytrimethylene terephthalate (PET)	1,3 Propanediol	30
Polyacrylic acid	3-Hydroxypropionic acid	100
Polyethylene (PE)	Ethanol	100
Polybutylene Succinate (PBS)	Succinic acid	~54
Polyvinylchloride (PVC)	Ethanol	100
Polyamide 11 (PA 11)	Ricinoleic acid	100
Cellulosics	Cellulose	Up to 100
Polyurethane (PU)	Fatty acids	Up to 70
Polyamide 6/10	Ricinoleic acid	66

Table 2.9: List of bio-based resins for green composites [29].

Fiber	Matrix	Fiber weight content (%)
Chicken feather	Acrylated epoxidized soybean oil + styrene	5-20
Chicken feather	Soybean oil pentacrythritol glyceride maleates	5-20
Hemp	Castor oil + PCL + diphenylmethane diisocyanate	5
Jute	Polyurethane and epoxy resins from Messua Ferrea seed oil	50
Flax	Epoxidized soybean oil	0-15
Kenaf	Epoxidized soybean oil + maleic anhydride	10
Coconut, sisal	Castor oil + diphenylmethane diisocyanate	14-30

Figure 2.6 represents classification of biodegradability of polymers.

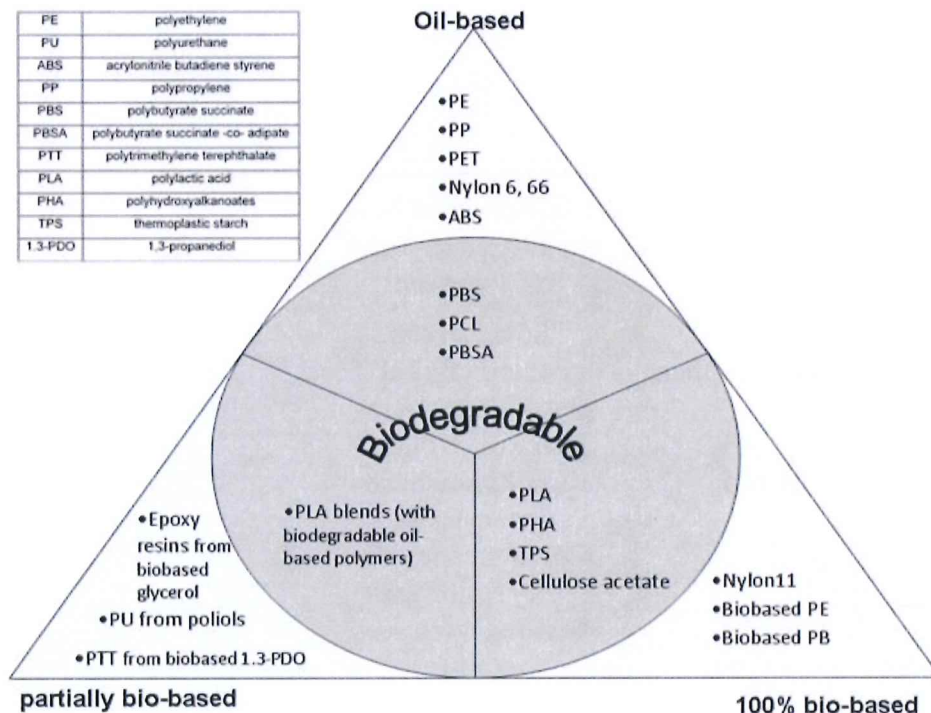


Figure 2.6: Biodegradability of polymers based on classifications [29].

To make green composites based on bio-based thermoplastic resins, polyhydroxyalkanoates (PHAs) have been investigated at a large extent (Table 2.10). If we compare fiber volume percentage of both thermoset and thermoplastic bio-based polymers we will notice that fiber loading capacity is quite limited until 50 wt.% because of manufacturing processes that are available currently to produce natural fiber composite materials. Within these techniques, dispersion of fibers with high viscosity thermoplastic polymers becomes tough. But injection molding and melt compounding have becoming a promising process day by day because of the capability to handle short length of natural fibers. For long length of fibers, compression molding is to be preferred for desired result. Automotive industry is already focusing on compression molding to work with long natural fibers. Key players of this industry are keenly having deep interest to have the best outcome of natural fibers. PLA have been used more by these players along with flax/hemp fiber combination (Green Line-Germany), Wood/PLA have been used to produce commercial compound named as 'FIBROLON 1'. A company from Finland named as 'Kareline Oy' have been produced one of their products tagged as 'Kareline1 PLMS'.

Moreover, ‘GreenGran BV’ from Germany have been manufactured their composite product from PLA/Wood compound. Compound market of Austria and France have been also followed by companies like ‘FASAL WOOD KG’ and ‘FuturaMat’ [29].

Table 2.10: List of bio-based resins for green composites based on fiber mass proportion [29].

Fiber	Matrix	Fiber mass proportion
Flax	PHB, thermoplastic starch, PLA	20-30 v/v%
Wood flour	PHB	0-30 w/w%
Pineapple leaf	PHBV	0-30 w/w%
Wood	PHBV	10-40 w/w%
Wood	Thermoplastic starch	10-30 w/w%
Flax	PHB, PHBV	8-40 w/w%
Straw	PHB	0-50 w/w%
Wool	Cellulose acetate	5-20 w/w%
Hemp	Cellulose acetate	0-30 /w%

2.5 Structure of Polypropylene (PP)

Polypropylene (C_nH_{2n}) is one of the heavily used thermoplastic polymer around the world due to its versatile application ranges. Most of the commercial application is mainly based on injection molding, extrusion or melt compounding and blow molding. Commercial polypropylene (PP) is having a combination of isotactic and atactic structures within its molecular orientation of solid phase.

Percentage of isotactic structures is 75% and atactic is 25%. Figure 2.7 represent chemical structure of PP [30]. It is having a molecular weight of 10k-40k g/mol. Within the chemical structure of PP, long carbon chain of backbone is dominant.

To avoid any sorts of oxidation from environment, stabilizers and some antioxidants are generally being preferred to use for its synthesis process [31]. PP is having extensive demand due its properties that supporting to produce commercial goods. Properties of PP are listed below:

- ✓ Having resistance behavior towards acids, alkalis and other type of solvents
- ✓ Lightweight due to its low density
- ✓ Good stiffness properties
- ✓ Provides impact strength
- ✓ Provides rigidity
- ✓ Resistant to heat flow
- ✓ Completely transparent [30].

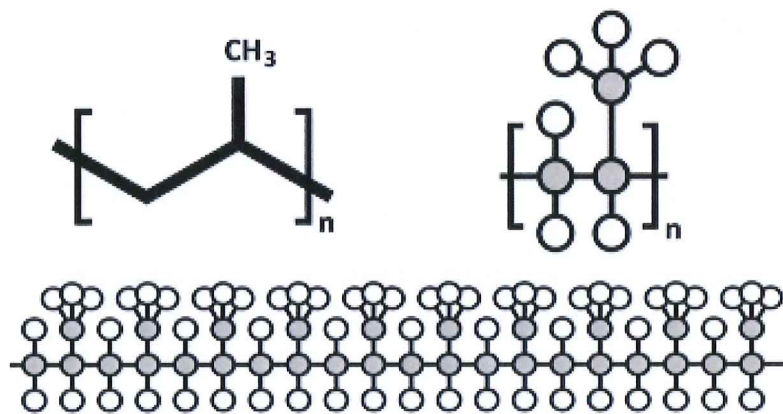


Figure 2.7: Chemical structure of PP [30].

2.6 Thermal Stability of Cellulose

Natural fibers are mainly based on combination of several organic materials. Due to this reason, if natural fiber undergoes through any sort of thermal treatment a drastic change can happen both physically and chemically with the structure of natural fibers. Behavior of thermal stability have been well understood through TGA (thermogravimetric analysis) method. Figure 2.8 represent a basic TGA graph of jute powder where thermal degradation can be noticed at 240⁰C.

Generally, thermal degradation of jute fibers occurs within 2 roots. At the very first root takes its place by degrading hemicellulose when temperature is in between 220⁰C to 280⁰C (low level). Second root takes place by degrading lignin of natural fibers in between 280⁰C to 300⁰C (high level) [32] .

Apart from jute fibers, thermal degradation process of other cellulose fibers also follows the same path way but in a different manner (dehydration and depolymerization) depending on type of fibers (Figure 2.9). Chemical reaction of dehydration takes place within temperature range of 300⁰C to 400⁰ C where anhydrocellulose and water molecule both remains in a uniformed manner. Oxidation takes place and aromatic char can be seen because of immediate release of carbon mono-oxide (CO) and carbon di-oxide (CO₂). Temperature limits to 800⁰C for thermal stability of the aromatic char. In addition to that- depolymerization gets active for cellulose material within both regions: crystalline and amorphous. While having thermal degradation of natural fibers, obtaining low molecular weight of components is directly connected to levoglucosan formation [33].

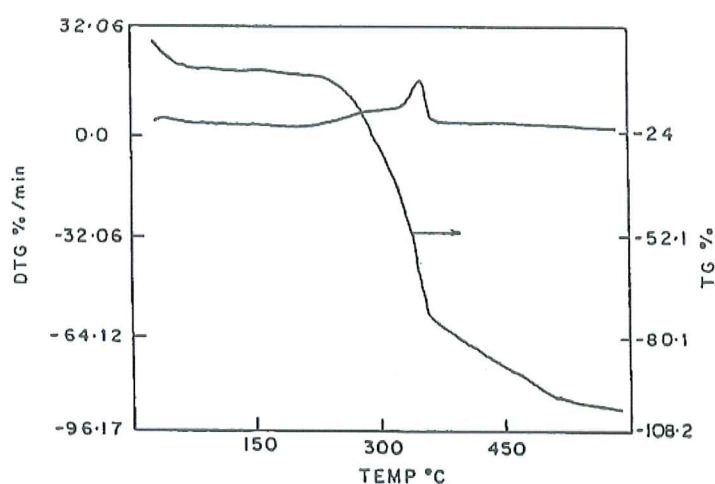


Figure 2.8: A basic TGA graph of cellulose of jute fiber [32].

For these two roots, it is very important to focus on temperature related to thermal degradation while manufacturing natural fiber-based polymer composites (thermoset and thermoplastic). Several initiatives have been carried out to enhance the behavior of thermal stability. Coating of natural fibers or polymer grafting is a fruitful idea to increase thermal stability to avoid thermal degradation. Lignin of natural fibers strongly support chemical reaction with monomers so that thermal degradation temperature can be increased by grafting method.

For example, with acrylonitrile thermal degradation temperature of jute fibers, sisal fibers can be increased marginally to increase thermal stability at a very first step. Acrylonitrile lessens the level of thermal degradation and percentage of weight loss of natural fibers.

There is an immense impact of thermal stability on mechanical properties of natural fiber-based composites. Due to thermal degradation, chance of causing deterioration of mechanical properties of composite materials increases. Possible reason might be 'change within surface' of natural fibers. In addition to that, if we exceed above 200°C while manufacturing composites with natural fibers, there is a high chance of having low-density and porous polymer with poor mechanical properties [32].

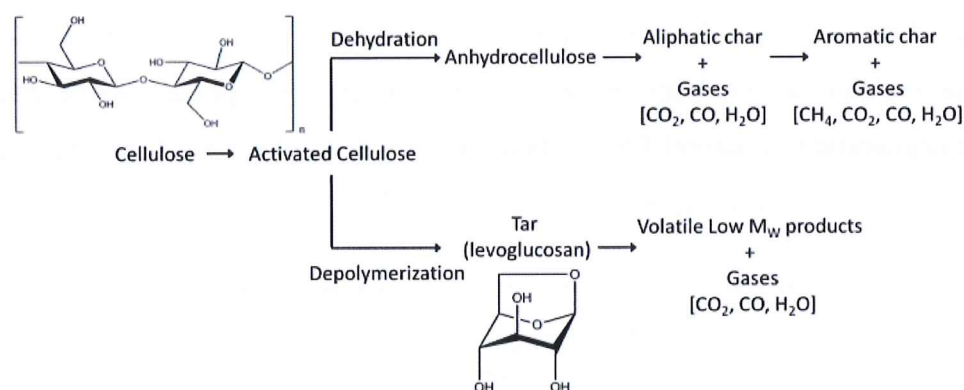


Figure 2.9: Schematic of formation of thermal and thermally oxidative degradation regarding cellulose fibers [33].

2.7 Flame Retardancy

2.7.1 Concept of flame and growth

Fire hazard is a common phenomenon related to flame and its growth. Controlling fire hazards is largely dependent on selected materials and its composition. Flame-retardant additives or fillers can have a direct impact on the behavior of flame and its initial growth. In most cases, flame retardants will help to control dripping behavior otherwise fire hazard can be evident. Growth of flame is interrelated to flammability of a material and incorporation of large amount of heat flux. Increasing oxygen concentration can increase flammability of a material as well. Flammability can be controlled in two general ways- one is by increasing ventilation and another is by forming chars which will induce growth of flame.

Concept of fire triangle have been represented in Figure 2.10. Figure 2.11 explains the entire relation of growth of flame and time. Initiation of growth of flame starts with a period of induction prior to flame ignition. Temperature increase is noticeable time to time.

After a sudden increase of temperature at a level of 800°C to 1000°C , flame continues in a steady state manner. This increase directly depending on polymeric material. Then, ignition of flame decays gradually with enough of consumption of fuel. Stabilization of flame growth at high temperature occurs such that breakage of chemical bonds can be dominant which creates volatile contents remaining over the surface of polymer materials. When amount of these volatile contents is reasonably staying around even with highly flammable polymers stabilization establishes. Condition of ignition differs in between outer source of flame and surface temperature of polymer (200°C). Two different types of heat will be significant. One is convection type and another is radiation type.

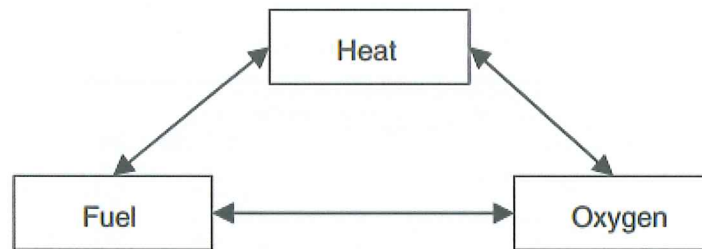


Figure 2.10: Concept of fire tri-angle [34].

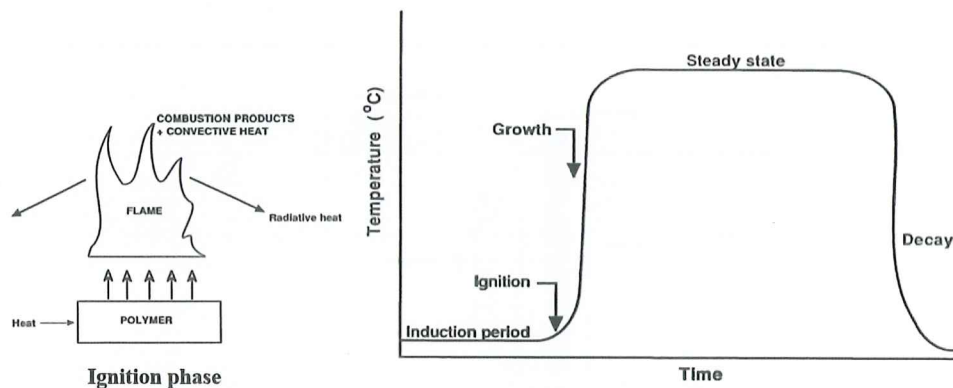


Figure 2.11: Growth of flame and ignition [34].

Flame growth initiation and heat transfer after ignition is completely different in between horizontal and vertical burning method (Figure 2.12 and Figure 2.13). Horizontal burning reflects on burning at a slow rate gradually. The key reason is heat conduction occurs only by gas-phase and heat radiations are at downward direction. On the other hand, vertical burning reflects on burning at a very fast rate instantly because of conductive, convective and radiative heat transfer altogether.

Growth of flame becomes larger immediately and radiative heat becomes more evident which causes decomposition of materials. Large amount of heat flux due to radiation have been produced. Area around the material becomes more flammable and smokier due to pyrolyze. Later on, flashover will occur. When flashover occurs, flame spreads rapidly with the aim of burning. Burning will only stop when fuel consumption completes extremely [34].

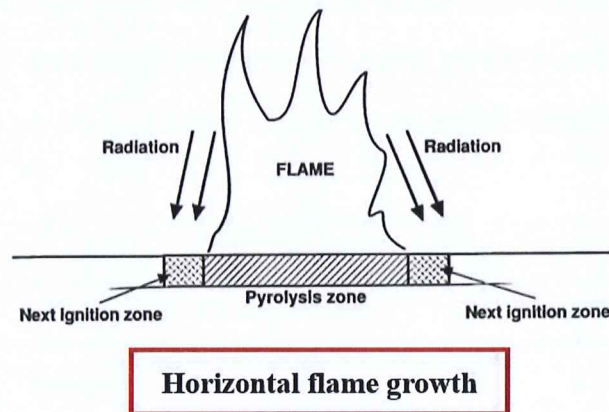


Figure 2.12: Flame growth (horizontal burning) [34].

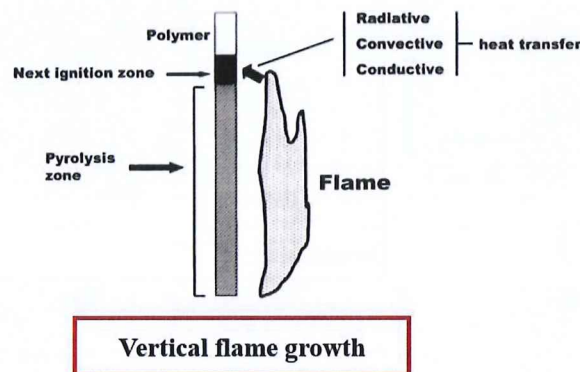


Figure 2.13: Flame growth (vertical burning) [34].

2.7.2 Flame-retardant mechanism

Flame-retardant mechanism depends on 2 modes of actions. One is 'physical action' and another one is 'chemical action'. Both mode of actions has been discussed below:

- I. **Physical action:** Physical actions have been dependent on cooling, char forming layer and dilution. Cooling action starts with an endothermic reaction where temperature lies below the level of combustion limit. Char forming layer gives protection to polymeric materials from excessive oxygen or heat penetration. Lastly, dilution of flame radicals occurs with water vapour or CO₂ release. Such that- flow of flame reduces. Flame-retardant additive like aluminum hydroxide behaves the same. It initiates water vapour release through an endothermic reaction. Not only that but also, a char forming layer forms by the residue of alumina.
- II. **Chemical action:** Chemical actions have been dependent on reactions related 2 states. One is 'solid phase' and another one is 'gas phase'. At solid phase, commercial flame-retardant additives can create a protective layer to increase thermal stability by protecting from degradation of selected polymer materials. This protective layer is a carbonaceous layer caused by dehydration of flame-retardant additives, behaves like an insulation way out to improve flame retardancy. On the other side, within gas phase exothermic reaction does not take place such that flame suppression occurs which reduce the temperature of materials while flame tends to propagate. Flame suppression can only be possible due to suppression of gases that are burnable. Intumescent flame-retardant fillers are more beneficial into commercial industry for smoke suppression [34].

2.7.3 Concept of synergism

Concept of synergism largely interconnected with flammability, polymer substrate and fire testing conditions. A carbonaceous layer or char layer forms after some chemical reactions at condensed phase. When inclusion of single or multiple flame retardant additives takes its space along with polymers there might be three different possible effects. First one is additive, second one is synergistic and third one is antagonistic. Inclusion of multiple flame-retardant additives means every effect related to each flame-retardant additive will take its place separately one by one.

A complete independent additive effect will be created. But in synergism concept, end value of addition of multiple flame-retardant fillers is much more beneficial compared to adding value of flame-retardant fillers separately. If we predict the result by an additive approach and result of flame-retardancy comes out less than our prediction value then antagonistic effect borns. There is still a lot to research about a complete synergism effect for a polymer [35].

2.7.4 Flame-retardant additives

2.7.4.1 Mineral based flame-retardant additives

Mineral based flame-retardant fillers are also known as ‘halogen free’ flame retardants. To improve flame-retardancy of polymers several mineral based fillers are available in industry (Table 2.11).

Table 2.11: List of key mineral based flame-retardant fillers [37].

Filler	Formula	T _{decomposition} /°C	ΔH _{decomposition} /kJ g ⁻¹
Aluminium hydroxide	Al ₂ O ₃ .3H ₂ O	180-200	1300
Magnesium hydroxide	Mg(OH) ₂	300-320	1450
Calcium hydroxide	Ca(OH) ₂	430-450	1150
Nesquehonite	MgCO ₃ .3H ₂ O	70-100	1750
Hydromagnesite	Mg ₅ (CO ₃) ₄ (OH) ₂ .4H ₂ O	220-240	1300
Huntite	Mg ₃ Ca(CO ₃) ₄	400	980
Ultracarb	Hydromagnesite/Huntite 60/40	220-400	1172
Boehmite	AlO(OH)	340-350	560

Such as- Aluminium hydroxide (Al₂O₃.3H₂O), Magnesium hydroxide (Mg(OH)₂), Calcium hydroxide (Ca(OH)₂), Nesquehonite (MgCO₃.3H₂O), Hydromagnesite (Mg₅(CO₃)₄(OH)₂.4H₂O), Huntite (Mg₃(CO₃)₄), Ultracarb (combination of hydromagnesite and huntite) and Boehmite (AlO(OH)) are key mineral based flame retardant fillers.

There are some other mineral based flame-retardant fillers available which require minimum level of loading such as Zinc borate, Nanoclays and Zirconium phosphates. Working principle is same for all. All of these mineral based flame retardant fillers function at a phase where condensation takes place and resulting a solid type residue. Decomposition temperature is greatly influenced by these mineral based flame-retardant fillers. It must occur above the processing temperature or at the same level of processing temperature [36]. From all of these fillers, aluminum hydroxide (ATH) is a well-known commercial flame retardant filler that requires higher level of loading (>50%) due to its economic perspective. As a mineral filler, ATH tends to conduct heat that initiates ignition of fire as well. When decomposition of ATH occurs alumina (Al_2O_3) produces due to water release and an endothermic reaction takes place to form water vapor which helps dilution of flame. Leftover residues of alumina work as a protective shield. Chemical reaction of this process is as follows according to Equation 2.1 and Equation 2.2:



$$\Delta H = +1.3 \text{ Kj. kg}^{-1} \quad (2.2)$$

In some cases, magnesium hydroxide (MDH) is being preferred where decomposition level is concerned. Some polymers need high amount of thermal stability. In that case, calcium hydroxide can be a fruitful one to use because its decomposition temperature is higher (430°C) but usually it does not give an overall satisfactory result due to high stability of carbonate.

Hydromagnesite ($\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) and Huntite ($\text{Mg}_3(\text{CO}_3)_4$) are also very popular commercial mineral-based flame-retardant fillers due to its particle size. Energy efficiency is directly related to particle size of a mineral based fillers. While infusing flame retardant fillers, decomposition temperature limit to be followed strictly. Sometimes filler can be beneficial but having limited range of decomposition temperature then user value falls down. For example, Nesquehonite ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$) is having a low range of decomposition temperature in between 70°C - 100°C .

Instead of using nesquehonite, Hydromagnesite ($\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) can be used with elastomers due to its high range of thermal stability (220°C - 240°C). For good heat absorbing capacity, boehmite ($\text{AlO}(\text{OH})$) is a suitable candidate due to its endothermic decomposition phenomena [36].

2.7.4.2 Halogenated flame-retardant additives

Activation of halogenated flame-retardant fillers occurs in gas phase. While having a complete combustion, halogen radicals have become free to create hydrogen halides. Creation these hydrogen halides occur by presentation of hydrogen atom. Source of these hydrogen atom is basically from polymers which tending to have degradation. These hydrogen halides have been responsible for sustaining the entire duration of combustion. A proper dilution of combustible gas in a mixture formation occurs when incorporation of halogenated flame-retardant fillers exists with polymer. After dilution, flame propagation stops. A chemical reaction between hydrogen halides and hydrogen or hydroxyl radicals is noticeable from chemical reactions listed below (Equation 2.3 to Equation 2.6). It is possible to form char layer by using these types of fillers. Halogenated flame- retardant fillers are harmful for environment due to immediate release of toxic smoke even with good flame retardancy [33].



2.7.4.3 Phosphorus based flame retardants additives

Phosphorous-based flame-retardant additives are very effective for cellulose-based materials because chemical reaction takes place with polymer and phosphorus component and a covalent bond (P-O) produces that helps to create char formation within surface of polymers. Generally, organophosphorus flame-retardant fillers are preferred for cellulose fibers with the aim of achieving flame retardancy. An incident named 'phosphorylation' can be seen within cellulose material where hydroxyls exist which causes a barrier to the creation of levoglucosan and toxic gases through a pyrolysis process.

Nitrogen-based and phosphorus-based flame-retardant additives are a perfect combination to use for polymers to achieve enhanced flame-retardancy and charring effect. Char layer causes minimization of heat and mass transfer related to flame and polymer material. Simply an insulation layer forms which stops the diffusion process while combustion takes place [33].

2.7.4.4 Nitrogen based flame-retardant additives

There are two specified groups available related to nitrogen-based flame retardants (NFR) based on their action mode while incorporation with polymers. The major mode of action is based on endothermic type of decomposition where bulk amount of inflammable gas including nitrogen and ammonia releases. Key concern is to maintain thermal stability while selecting these fillers. NFR is basically holding its thermal stability at high level along with low volatile content. Second action mode is largely connected related to interaction between polymer and fillers where within condensed phase takes it to the one way of thermal decomposition of polymer or other way of formation of char. Formation of char is greatly influenced by polymer interactions with fillers.

A well-known nitrogen-based flame-retardant filler is ammonium polyphosphate (APP-NH₃PO₄). It is also known as 'intumescent flame retardant' that contains inorganic salt relates to polyphosphoric acid and ammonia itself. An endothermic decomposition takes place for ammonium polyphosphate to create char formation layer. Thermal stability of APP is greatly dependent on its number of phosphate unit. If number of phosphate unit is greater than 1000, it becomes more stable thermally. Thermal decomposition takes place around 300°C. Figure 2.14 explains intumescent behavior and char formation mechanism of APP with polymer matrix. Removal of heat occurs via an endothermic reaction. Dilution of combustible gases happen by the presence of ammonia and water. A chemical reaction takes place between produced polyphosphoric acid and hydroxyl group of selected polymer material. Result of this chemical reaction is 'phosphate ester'. Through a dehydration process formation of char takes place immediately. Char formation behaves like a protective shield. Flame retardancy of APP can be improved more by adding a suitable synergist.

Thermal stability of melamine polyphosphates (MPP) is greater than melamine phosphate and melamine pyrophosphate. Transformation of these phosphates are related with heat applications where water releases subsequently and creates an effect like heat sinking. Endothermic reaction of MPP takes place over temperature limit of 350°C.

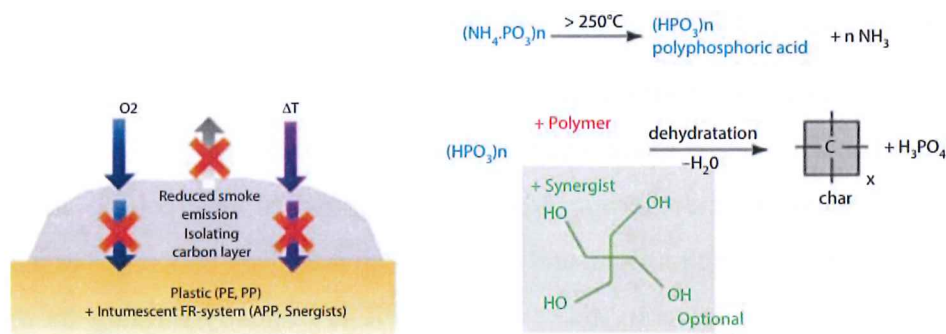


Figure 2.14: Intumescent and char formation mechanism of APP [38].

Char formation is heavily influenced by MPP. Concentration of oxygen gets lower within the surface of polymer with the effect of formed char. Due to its increased amount of thermal stability, application range with polymers also increases within commercial industry. Melamine cyanurate (MC) is also well known for its high thermal stability causes from its chemical structure. It is a combination of melamine and cyanuric acid. For high processing temperature of polymers ($>300^\circ\text{C}$), MC is a good choice. Endothermic decomposition occurs beyond 320°C . A heat sink type behavior can be observed from MC. While combustion takes place an inert gas takes an attempt to dilute existing oxygen and other type of gases related to fuel. But it is highly recommended to use P-based FRs along with MC to get desired result [38].

2.7.4.5 Silicon based flame-retardant additives

Nowadays, silicon-based flame-retardant additives are very promising candidates in terms of flame retardancy. In the market, there have been many silicon-based products which have been tested. Such as:

- ✓ Silicones
- ✓ Silicas
- ✓ Organosilanes
- ✓ Silsesquioxanes and
- ✓ Silicates

From the list of above, only silicon becomes more promising in terms of achieving desired result of flame-retardancy because of having high thermal stability and resistance behavior towards processing temperature without producing high level of toxic components.

Char formation is dominant in the presence of silicon by instant migration towards the polymer surface. A complete thermal insulation is possible to achieve via fumed silica and silica gel [38].

2.7.4.6 Boron based flame-retardant additives

To make halogen free flame-retardant compound, boron-based flame-retardant additives are very useful. There are several advantages to use these kind of additives as follows:

- It helps to create char form
- It stops dripping
- It increases suppression of afterglow, smoke and carbon monoxide
- It makes water release significant which ensures better flame-retardancy
- Improves heat flux behavior of polymers
- Effective with nitrogen-based, phosphorus based and silicon-based flame retardants
- Synergetic effect improves
- Improves crosslinking behavior with OH group to create char layer
- Stabilization of alpha protons is significant in chemical reaction with OH group

There are several commercial boron-based flame retardants around the world. Such as: Borax pentahydrate, borax decahydrate, boric acid, boric oxide, anhydrous borax, disodium octaborate tetrahydrate, calcium metaborate, zinc borate, ammonium pentaborate, melamine diborate, boron phosphate and magnesium borate [38].

2.7.5 Combustion and flame-retardant behavior of PP

Polypropylene (PP) is a very common type of thermoplastic polymers which is very linear. It is highly flammable and can produce high level of toxic fumes when causing generation of melt by ignition. Flammable behavior of PP is completely connected to chemical structure of itself. For that reason, it is important to understand the relation between chemical structure of PP and source of fire ignition and flame types. Large amount of oxidation occurs when PP exposes to heat.

Generally, combustion process takes place from an immediate chain reaction from the fuel source based on exothermic, luminescent methods. Combustion mechanism of PP starts with several processes.

Firstly, from a fire ignition source PP can ignite and heated up rapidly. It is completely depending on external behavior of heating source. If heat generation is higher, heating process gets influenced faster. Secondly, from fire ignition PP tends to go through a thermal degradation stage where chain of PP takes in all the possible energy to itself to cross out the bond energy coming from the most-weakest bonds within the molecule of PP. A lot of polymer fragmentation can be seen. Decomposition is coming into third place where produced fragments from the second phase will produce more smaller components along with fumes and toxic gases. These components can liquid or solid. Only liquid component can be helpful to prevent the burning of PP. On the other side, solid components tend to create chars or unwanted hazardous components. At fourth phase, when oxygen concentration gets enough limit within PP combustion of PP occurs and heat release is noticeable [39]. To improve flaming behavior of PP, there have been lot of flame-retardant fillers are available. But due to the limitation of PP (high crystallites), to make inflammable PP is tough to happen. Higher loading of fillers is required. But then mechanical properties start to decrease. However, lot of research is ongoing to improve the flame-retardant behavior of PP [40]. PP is having an aliphatic hydrocarbon structure. Such that flammability of PP increases. The value of heat combustion is generally higher compared to other type of polymers (40 kJ/g). A lot of research has been carried out with atactic PP and isotactic PP at a condition of non-isothermal where volatile components have been produced at over 420⁰C regarding atactic and for isotactic PP volatile components have been produced at 425⁰C. Process of recovery of carbon is made of with a composition of diene, alkanes and alkenes. Highly flammable behavior of PP is related to formation of fuel. It is related to hydrogen component that belongs to pyrolysis component produced via flash pyrolysis at an elevated temperature of 520⁰C. And an accumulation of some toxic compounds occurs from cooled combustion of flames [41].

2.7.6 Combustion and burning behavior of thermoplastic polymers

Thermoplastic polymer materials are having wide range of applications including home decorations and insulation of buildings. For that reason, safety is a great concern because of possible fire hazards. When ignition occurs, melt dripping of polymers come to the first place to cause excessive load of flames. When amount of flame increases, toxic gas increases and situation gets more complicated.

The entire process of expanding flames and gases is very tricky specially for thermoplastic polymers. Pyrolysis is not the only possible root cause which is responsible for high burning behavior of these polymers. There are some more reasons available (Figure 2.15).

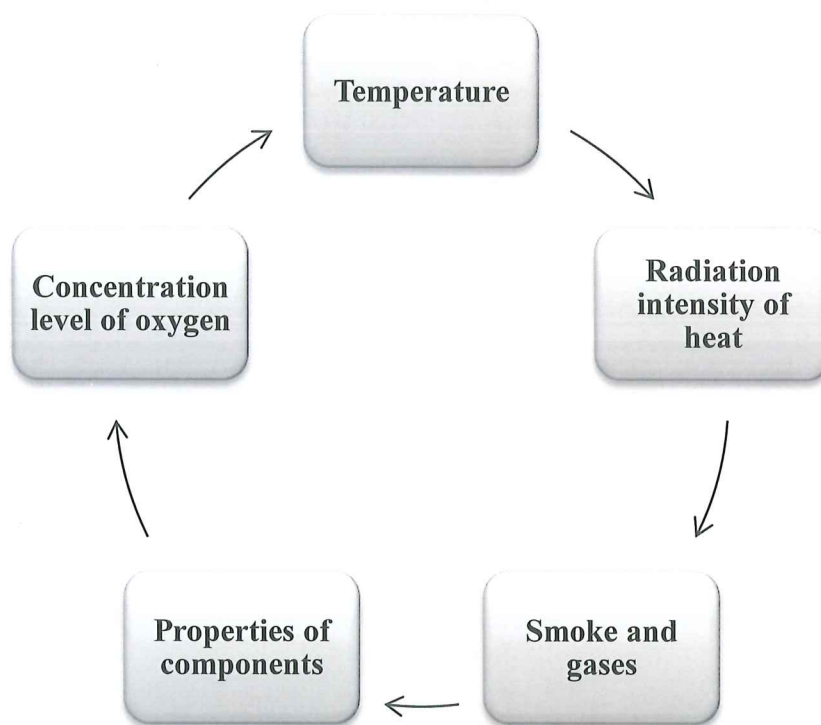


Figure 2.15: Key reasons for high flammability of thermoplastic polymers [42].

Combustion and flammable theory are dependent on several key factors including melt dripping, pyrolysis and vaporization. All of them are helping to create a type of liquid during combustion modes that will increase the area of flame. Figure 2.16 represents about factors and parameters which are controlling combustion and flame behavior of thermoplastic polymers. At the end, just before releasing toxic gases flashover gets ensured via combustion process of thermoplastic materials.

Flame geometry is one of the key factors that directly effects on behavior of flammability of thermoplastic polymers. However, temperature around the flame is also play a key role accordingly. Spreading of flame is an end result of physical and chemical action through three ways such as: Naturally, concurrently (un-stable) and oppositely (stable).

Unstable flame behavior is much speedier in comparison with stable flame flow. The key reason behind it is-elevated temperature at the place where burning have not taken any place. Heat absorption gets higher [42].

Behavior of flame can be controlled by adding different type of flame-retardant fillers. These flame retardant fillers help to break the common burning process even before a proper ignition takes place [40].

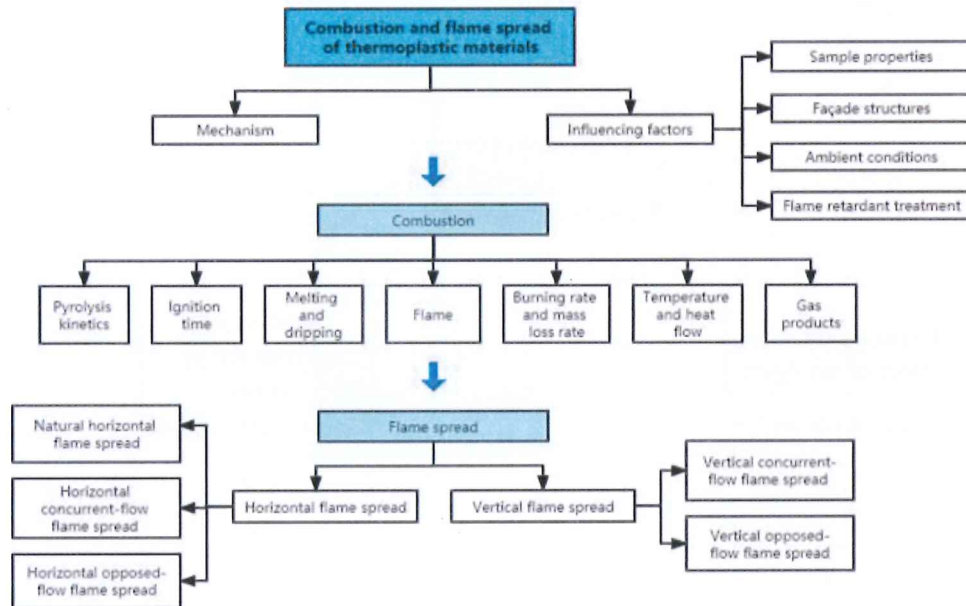
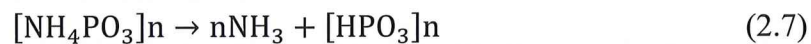


Figure 2.16: Factors and parameters of combustion and flammability of thermoplastic polymers [42].

2.7.7 Flame-retardant mechanism of APP/ZB

APP works as an intumescent filler. Synergism of APP occurs with condensation of internal phase. Decomposition process of APP tends to start when it is close to heat [43]. It can create a char formation that helps to close the immediate expansion of heat and mass transfer of jute-pp composites. But smoke generation is a drawback [44]. Temperature limit of decomposition of APP ($\approx 250^{\circ}\text{C}$) [45] and ZB ($\approx 290^{\circ}\text{C}$) [46] is not close to each other. Decomposition of APP does not even take place at 260°C . But at 250°C , initiation of ammonia and polyphosphoric acid can be noticed with an immediate release as Equation 2.7 [47].



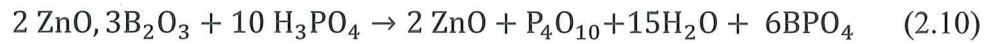
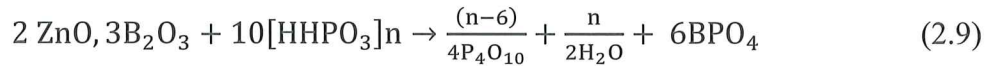
An endothermic reaction takes place to break down polyphosphoric acid and creates phosphoric acid and water. A complete unsteady phosphate ester formation occurs due to the reaction of polyphosphoric acid and hydroxyl group within substrate.

Through dehydration process, the phosphate ester responds to create char layer which is named as 'Intumescent Char' [45].

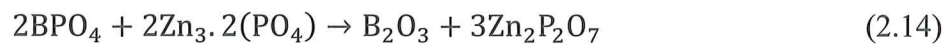
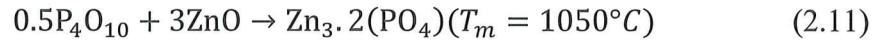
This intumescent behavior can be seen in the presence of the char and ammonia within temperature range of 360⁰C- 420⁰C. Water release occurs with the reaction of carbonaceous composition and polyphosphoric acid as Equation 2.8 mentioned below:



On the other side, decomposition of ZB tends to have at 290⁰C. Chemical reaction starts within the temperature of 300⁰C – 415⁰C where BP0₄ produces as Equation 2.9 and Equation 2.10 mentioned below:



When temperature rises to 580⁰C, formation of zinc orthophosphate occurs which melts around at 1050⁰C results P₂O₄ and ZnO. Both of these components come closer and produces zinc pyrophosphate. Moreover, to produce B₂O₃ composition a reaction is required in between borophosphate and zinc orthophosphate. A definite synergic mechanism occurs within APP and ZB when zinc phosphate and borophosphate (BP0₄) initiates following Equation 2.11 to Equation 2.14.



No more decomposition occurs within APP and ZB together [47].

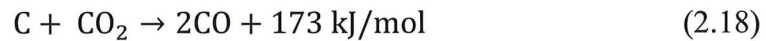
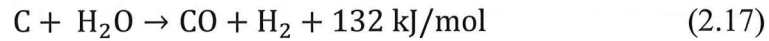
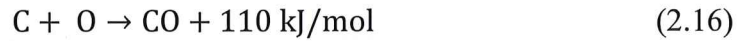
2.7.8 Flame-retardant mechanism of ATH/ZB

Both of these organic fillers, are having crystal type water component which is constituted into their chemical structures. Temperature range of Endothermic reaction for ATH is 220⁰ C-450⁰ C and for ZB is around 290⁰C – 450⁰C. Steam release happens for ATH is 34 wt.% and for ZB is 15 wt.%. These released steam causes dilution of combustible gases that releases temperatures and increases flame-retardant behavior of jute-pp composites.

During flame propagation of jute-pp composites with the application of ATH/ZB, oxygen feels hard to be penetrated due to protective barrier caused by ATH/ZB dehydration reaction and B_2O_3 and Al_2O_3 releases [48].

2.7.9 Flame-retardancy of natural fiber composites (NFCs)

Carbon is the key element which is responsible for flammable behavior of lignocellulosic natural fibers. Combustion of Calorimetric efficiency of carbon sources is being represented within Equation 2.15 to Equation 2.19 as follows:



When flame occurs heat release rate (HRR) can peak towards its highest level in the case of complete oxygen delivery. The amount of flammable gas release is less for flame retardant materials. Lignocellulosic fibers can be very helpful in terms of char formation specially when fibers are fully surface treated (Figure 2.17). There is a high chance of creation of flammable gases when fibers are untreated (Figure 2.18) [49].

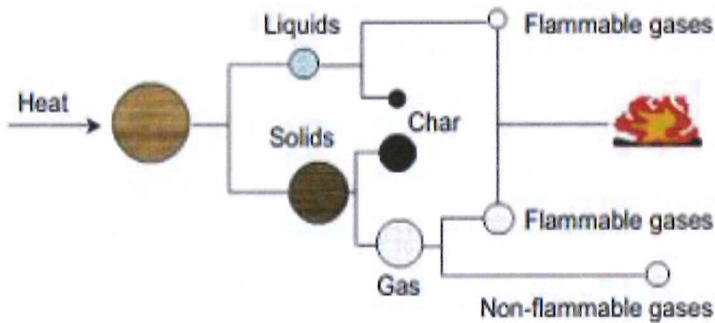


Figure 2.17: Treated lignocellulosic fibers [49].

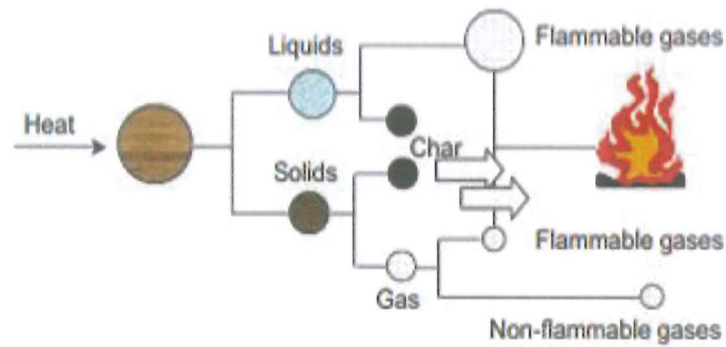


Figure 2.18: Untreated lignocellulosic fibers [49].

Because of their availability of hydroxyl group phosphorus-based flame-retardant materials can be a good option because while having thermal decomposition these type of flame retardants responsibly dehydrate cellulose to form char mechanism. To have more efficient result adding a nitrogen-based synergist can be a good option for better flame retardancy [50]. Burning mechanism for natural composites is being classified into five critical steps such as: (1) heating, (2) decomposition, (3) ignition, (4) combustion and (5) propagation. The amount of lignin is greatly responsible for char formation. On the other hand, high amount of cellulose is the key reason of flammability. Flame-retardancy of natural fiber polymer composites have been developed by several ways as follows:

- Creating fire barriers (coatings, additives etc.)
- Introducing flame retardants (Aluminum Hydroxide, Magnesium Hydroxide etc.)
- Introducing synergists (Zinc Borate, Antimony Trioxide etc.)
- Introducing Nanomaterials (Expandable Graphite) [51]

While adding flame retardants within natural fiber polymer composites thermal stability is critical. For producing flame-retardant natural fiber reinforced polymer composites, it is important to know about flammability and combustion nature of natural or plant-based fibers and polymeric matrix component.

While ignition occurs within natural fiber polymer composites combustion occurs where a lot of heat dense smoke and volatiles create and polymer matrix follows the path of thermal and oxidative decomposition phases very rapidly. Types of volatiles are depending on polymer types and their chemical structures. Generally, these volatiles are having carbon monoxide, hydrocarbons & carbon dioxide or hydrogen halides as noncombustible or non-flammable. Polymeric decomposition or burning happens due to the free radical of volatiles. Both physical (melting temperature, glass-transition, decomposition) and chemical properties of polymers are greatly responsible for quality products. At selected temperatures, polymeric phase transition occurs which is directly influencing viscosity, modulus, density along with thermal conductivity [8]. For better flame-retardant natural fiber reinforced polymer composites, char formation is necessary because it creates a barrier where heat created from the flame is being stopped to transfer towards the polymer [8,52]. In this case, highly crosslinked polymers are being more preferred to ensure low level of flammability along with high level of flame retardancy. Not only polymers, natural fibers are having great influence on flame retardancy. But their key problem is their internal cellulosic structures along with poor thermal degradation nature for low level of flame retardancy. In the case of thermal degradation, temperature is the key factor for natural fibers (400°C - 500°C). Such that it is important to know about the decomposition temperature ranges of cellulose (260°C - 350°C), hemicellulose (200°C - 260°C) & lignin (160°C - 400°C). In short, Flame retardancy of natural fibers fully depending on its fiber structures and orientation. If there are any kind of ashes and silica available then flame-retardant behavior of natural fiber improves [8].

During flame propagation of natural fibers, combustion contributes 8 to 12 kJ/g gases by heat generation. Compared to industrial polymers heat release rate of natural fibers is very low. This is another reason of adding natural fiber into polymer materials to make flame retardant composites. In this case, short fiber length (3-4mm) is critical parameter for thermoplastic materials and fabric structures (warp, weft etc.) for thermosets are needed to be considered significantly [52].

Six prime reasons can be listed down regarding the motive of applying flame retardant fillers with natural fiber reinforced polymer composites as follows:

- Reason 1: Delaying time to Ignition
- Reason 2: Reducing heat release rate
- Reason 3: Reducing the amount of spreading of flame within surface
- Reason 4: Reducing mass loss rate
- Reason 5: Reducing rapid propagation of fire
- Reason 6: Saving human lives [53]

Application of flame-retardant fillers for natural fiber composites is ancient way. During 1st century AD, salt water has been used for wood after the great incident of fire in Rome. In theaters, canvas have been protected from fire in 1638. Moreover, incombustible cloth has been introduced by Oxford at the time of 1684. An innovative coating concept brought in 1783 with the aim of reducing fire hazards. Application of inorganic salts have been carried out in between 18th to 19th centuries by the hand of William Henry Perkin and Gay-Lussac. Gay-Lussac have been proposed to use ammonium chloride and diammonium phosphate specially for wood and lignocellulosic components. During his time, many more inorganic salts like boric acid and borates, chlorides of calcium, magnesium, zinc and tin, sodium silicate, aluminum hydroxide and calcium sulphate have been practiced for the same purpose But, application trends have been changed drastically during World War II because during that time- making protective textiles and wooden flame retardant parts have been the emergency requirements for military applications. The use of ammonium chloride, ammonium phosphate, borax has been implemented. Problem of flame retardancy have been well issued by William Henry Perkin during 20th century. There has been a list of requirements made for textile materials from the concern of human safety. A first and foremost requirement have been ‘damage free cloth’. Second requirement have been ‘unaffected color or design’. Moreover, skin contamination should also to be avoided strictly. Lastly, ‘fiire proofing’ of textile materials have been followed accordingly.

The official concept of ‘Non-Flammable’ has been confirmed by Perkin and it went on for more than 150 years without any buzz. But with single component, flame retardancy problem is hard to be solved. Until the time of second world war, no more development of flame retardancy have been carried out. Several cellulosic fibers are available which can be used for flame retardancy along with polymers and flame retardant fillers. Such as cotton, viscose, flax, hemp, jute, sisal, ramie, abaca, cabuya and coir fibers. These fibers are flammable in general due to its nature of combustion. Flammability can be decreased in many ways. For example, blending cotton with polyester (10-20%) can reduce flammable nature but chemical treatment (tetrakis-(hydroxymethyl)-phosphonium chloride, N-methylol dimethylphosphonopropionamide, organophosphorus oligomer (HFPO) and dimethylol dihydroxyl ethylene urea (DMDHEU)) of flame retardants can show more better results. Figure 2.19 shows the efficiency of chemical treatment of flame retardants for cellulosic fabric. A significant change of heat release rate has been noticed (170 kW/m^2 - 20 kW/m^2) [49].

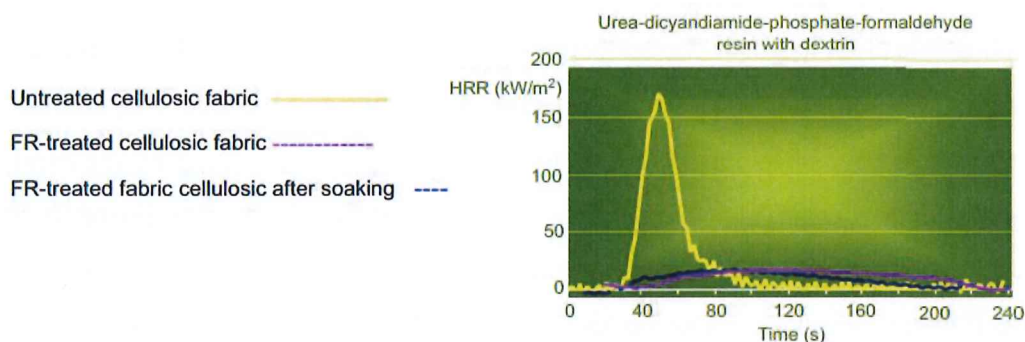


Figure 2.19: Flammable Behavior of coated fabric [49].

2.7.10 Flame-retardancy of thermoplastic-based natural fiber composites (NFCs)

Numerous polymeric materials have been used along with natural fibers for opening the door of flame-retardant applications. Such as- Polyethylene (PE), Polypropylene (PP), Polylactic acid (PLA), Polybutylene succinate (PBS), Polyamide 6 (PA6), Polybutylene terephthalate (PBT), Thermoplastic starch (TPS), High density polyethylene (HDPE) and so on [52,54,55]. The prime reason to incorporate natural fibers with polymers is to achieve lower amount of peak heat release rate (pHRR). It has been seen that for a 14% decrease in pHRR least amount of fiber amount is 10%.

The decrease rate improves more when flax fiber content increases up to 25% to incorporate with PLA/TPS. As a plasticizer glycerol have been used. Natural fiber like flax tends to create a barrier which helps to decrease the peak heat release rate. Time to ignition can be also reduced along with natural fibers. But the key issue is char formation. In this case, sometimes polymers are not promising in action sometimes. Char formation increase along with the increase of natural fiber content. Figure 2.20 represents some examples regarding peak heat release rate and natural fiber content [52]. Figure 2.21 shows the heat release rate curves for several natural fibers within the radiant level of 36 kW/m² [56].

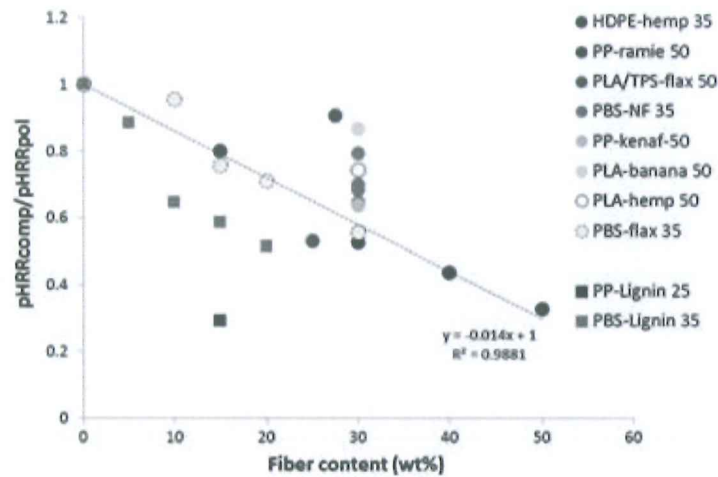


Figure 2.20: Evolution of relative pHRR versus fiber content for various non-flame retarded bio-composites [52].

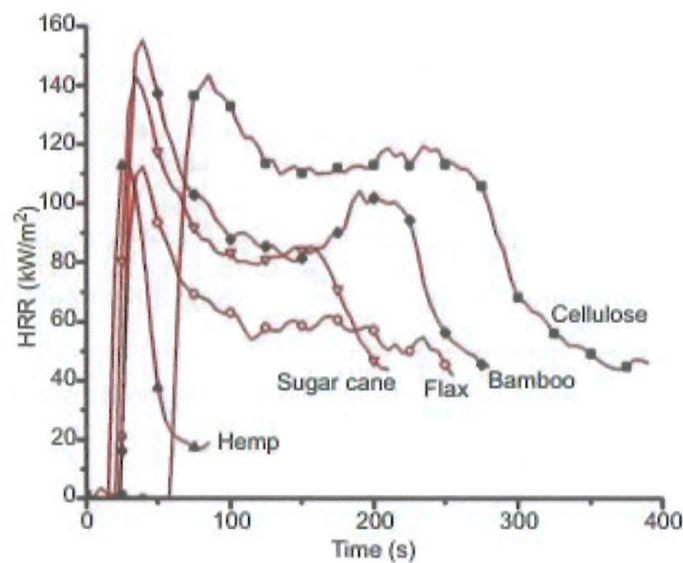


Figure 2.21: Heat release rate curves for natural fibers [56].

The value of limit oxygen index (LOI) has been greatly influenced by the use of natural fiber composites based on polymer materials. In terms of the value of limit oxygen index (LOI), prime concern is its increment along with the increase of natural fiber content. To increase the LOI value, several experimentations have been carried out. PP have been incorporated with 50% rice husk or sawdust. But the increase is not linear. LOI value can be decreased as well due the wicking effect. Dripping is pretty common in the usage of natural fibers. Kenaf fiber is also been used with PP where glow wire of flammability index has been greatly influenced. A Sharp drop of temperature have been noticed (725°C to 675°C) by the addition of 30% kenaf fibers with PP. However, flax fibers took it to the 650°C with the same amount like PP-Kenaf combination. From the UL-94 test (horizontal burning test), burning rates increases with the presence of natural fibers (Sisal, Rice husk) with PP. With the same weight limit (35%) of natural fibers like jute or coconut fibers along with PLA or PP, burning rate decreases significantly. Like these, many different sorts of natural fiber-based flame retardant composites have been made along with PBS and acrylic resins. The significant difference has been noticed within the value of time to ignition and peak heat release rate. These values differ from fiber to fiber. For example, the value of time to ignition is 52 seconds and the value of peak heat release rate is nearly 500 kW/m^2 . When jute is present time to ignition value have been reached to 110 seconds and peak heat release rate reached to 900 kW/m^2 . Peak heat release rate, time to ignition, mass loss and burning rates are being greatly influenced by homogenization of fibers and thermoplastic polymer materials. For that reason, short fibers can be a great option to choose while making composites with maleic anhydride-grafted polyethylene (MAPE). With the inclusion of MAPE, an external barrier effect can be seen within natural fiber based polymeric flame-retardant composites. Heat release rate reduces by the addition of MAPE [52]. Moreover, when ammonium polyphosphate (APP) has been used with PLA/ramie fibers combination LOI value increased instantly from 19.1 to 35.6 with UL-94 V-0 standard. Incorporation of nanomaterials like multiwalled carbon nanotubes (MWCNTs) can also be done with PLA/hemp/sepilolite nano clay combinations to reduce peak heat release rate. ATH (Aluminum trihydroxide)/Kenaf/PLA composite formation have also been investigated by Woo and Cho et al where inclusion of 50 wt.% ATH helped to achieve LOI value of 39.7.

Inclusion of Flame-retardant additives like aluminum diethylphosphinate (AlPi) and antimony oxide (Sb_2O_3) helps to increase the time to ignition for natural fiber composites as well. Some other flame-retardant treatment (DAP, $\text{P}(\text{MAPC}_1(\text{OH})_2)$, and $\text{P}(\text{MMA-co-MAPC}_1(\text{OH})_2)$) have been used for natural fiber like flax and PBS (Poly (1,4-butanediol succinate)). When it comes to use the flax fiber with phosphorus-based flame retardants to make composites, a covalent bond forms with flax fiber which is greatly influencing char formation with dehydration process. Another natural fiber named 'Bamboo' have been used with PBS but the value of time to ignition reached to 43 seconds with the peak heat release value of 339 kJ/m^2 . The reason of early ignition is the mismatch of degradation temperature of fiber (350°C - 380°C) and PBS (390°C). Degradation temperature of fibers highly influences the char layer formation, heat release rate and mass transportation. Specially for char formation lignin of natural fibers attributes most. For enhanced mechanical, chemical and flame-retardant properties natural fibers can be used with polymer materials [56] for sustainable industrial applications. Sustainable industrial applications of natural fiber composites ensure several key factors as follows:

- Lower level of density & higher amount of specific strength and stiffness
- Low energy consumption including CO_2 absorption & ensuring oxygen towards environment
- Cheaper than synthetic fibers and recycleable
- Easy manufacturing processes
- Level of acoustic insulation is higher in natural fibers
- Lower emission rate of toxic fumes
- Higher fuel efficiency
- Easy to transport [57].

2.7.11 Literatures of flame-retardant jute/PP composites

Jute fiber has been greatly used along with PP to make flame retardant composite materials. Several studies have been carried out.

From Table 2.12, it can be seen that numerous studies have been conducted with jute/PP flame retardant composites. Jute fibers have been used in between 40-75% while experimentation up to now for better flame retardancy. The range of LOI value have been achieved as 18.7-30.6. The key issue here is to reduce peak heat release rate [58–65]. In this case ammonium polyphosphate is beneficial to use from overall perception. It also effects on time to ignition (TTI) [58,59,62]. Figure 2.22 also shows that-in the presence of ammonium polyphosphate and natural fibers, it is possible to reach V-0 level of flame retardancy [66].

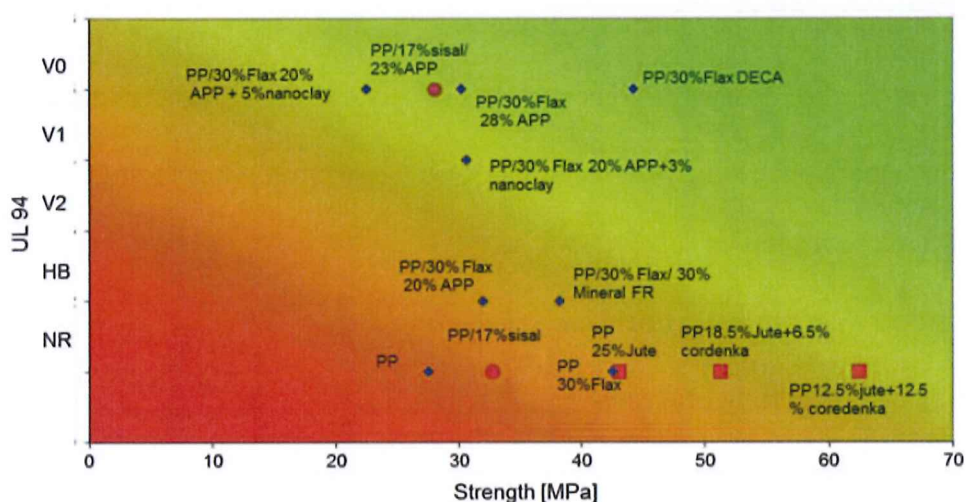


Figure 2.22: Flame retardant selection chart for natural fiber reinforced polymer composites based on flame retardant level and mechanical properties [66].

Mechanical properties have been also important to focus on while using jute/PP composition along with flame retardants. Mechanical properties have been greatly increased while using PVC/Jute/PP composition [64].

To improve flame retardancy of natural fiber reinforced polymer composites, several types of flame retardant materials available into the market such as bromine based, Chlorine-based, Phosphorus-based, Antimony oxide, Alumina trihydrate, magnesium hydroxide, boron, molybdenum and nitrogen-based are largely available into the global market. The usage of these flame retardants depending on its market area, product and its flame retardant philosophy regarding natural fibers [49].

From above all discussion, it is very clear to say that- there have been no such studies available where optimum fiber loading based on fiber type (long or short) carried out to make flame retardant jute-pp composites including maximum loading of flame retardants. Within the scope of this thesis, “jute/polypropylene (PP)/aluminum trihydrate (ATH)/zinc borate (ZB)” and “jute/polypropylene (PP)/ammonium polyphosphate (APP)/zinc borate (ZB)” combinations have been studied experimentally.

Table 2.12: List of flame retardant jute/PP composites.

Flame Retardant Composite	Composition (wt.%)	LOI (%)	Horizontal Burning (mm/min)	PHRR (Peak Heat Release Rate (kW/m ²))	Time to Ignition (s)	Total Smoke Production (m ²)	Effective Heat Combustion (MJ/kg)	Total Heat Release (MJ/m ²)	Burning Level (UL-94)	Ref.
Ammonium polyphosphate/Expandable graphite/Jute/PP	2/3/75/20	30.6	19.5 (s) (HB)	240	21	3.2	27.15	39.8	-	[58]
B-cyclodextrin/Ammonium polyphosphate/Melamine/Jute/PP	5/10/5/40/40	27.8	37 (s) (HB)	312	18	6.5	25.1	43.3	-	[59]
Diammonium phosphates/Jute/PP	5/60/35	-	19(HB)	-	-	-	-	-	-	[60]
B-cyclodextrin/Ammonium polyphosphate/Jute/PP	6.67/13.33/40/40	-	-	167 (Average)	-	6.6	-	-	-	[61]
Jute fiber/PP fiber/Ammonium polyphosphate/PP Powder	35/35/10/20	25.7	10.09 (HB)	-	-	-	-	-	-	[62]
Bisphenol A epoxy acrylate (BAEA)/2-hydroxyethyl methacrylic acid phosphate (PM-2)-polyurethane acrylate (PUA)/Jute/PP	BAEA 50 (mass%) PM-2:50(mass%)	26.1	8.54 (HB)	341.65	7	-	9.2	58.34	-	[63]
Jute/PP/PVC/Aluminum trihydrate (ATH) / Zinc Borate with Antimony trioxide (ZB-AT)	50/16.5/13.5/10/7.5/2.5	-	220 (HB)	-	-	-	-	-	-	[64]
Jute/PP	42/58	18.7	27(HB)	675	33	1121	31.7	95.5	121.2 minute (VB)	[65]

3. EXPERIMENTAL PROCEDURE

3.1 Materials

A commercial grade of Polypropylene (PP) homopolymer (Teldene-HO6ML) with melt flow rate of 6.0 g/10 min and density of 0.9 g/cm³ have been supplied by national petrochemical industrial company (NATPET), Saudi Arabia. Maleic anhydride grafted polypropylene (MA-g-PP, ATABOND[®] 3100) with melt flow index of 40-80 g/10 min and density of 0.91 g/cm³ supplied by TMB polymer, Turkey. Jute fiber as jute twin (2 ply rope) with density of 1.5 g/cm³ have been purchased from İPSAN[®], Turkey. Fine precipitated Aluminum hydroxide (ATH, EtiFine 706) have been supplied by ETİ ALÜMİNYUM, Turkey. Density of ATH is 2.4 g/cm³ and particle size distribution (μm) of ATH are D₁₀:0.3-0.6, D₅₀:0.8-1.5 and D₉₀:2.5-4.0. Ammonium polyphosphate (APP) with average particle diameter of >250 μm (5% w/w) and density of 1.61 g/cm³ have been supplied by ACAR CHEMICALS, Turkey. Zinc borate (ZB, Melos 4015, average particle diameter of 2.9 μm) with density of 3.64 g/cm³ have been supplied by MELOS KAUÇUK VE PLASTİK SAN. VE TİC. A.Ş, Turkey.

3.2 Formulations

3.2.1 Formulations of jute/PP composites

Table 3.1 represents designation and composition of jute/PP composites produced via compounding and hot press method with different loading levels of jute fibers.

Table 3.1: Designation and composition of jute/PP composites with different loading levels of jute fibers.

Sample Code	Jute (wt.%)	MA-g-PP (wt.%)	PP (wt.%)
PP 100	-	-	100
PP 95	-	5	95
LJF 20	20	5	75
SJF 20	20	5	75
SJF 30	30	5	65
SJF 40	40	5	55
SJF 50	50	5	45
SJF 60	60	5	35

3.2.2 Formulations of flame-retardants based jute/PP composites

Table 3.2 represents designation and composition of flame retardants-based jute/PP composites produced via compounding and hot press method with flame retardants loading at ratio of 80:20.

Table 3.2: Designation and composition of flame retardants-based jute/PP composites with flame retardants loading ratio of 80:20.

Sample Code	Jute (wt.%)	MA-g-PP (wt.%)	PP (wt.%)	ATH (wt.%)	ZB (wt.%)	APP (wt.%)
30(ATH_4/ZB_1)	30	5	35	24	6	-
30(ATH_4/ZB_1)	30	5	25	32	8	-
30(APP_4/ZB_1)	30	5	35	-	6	24
40(APP_4/ZB_1)	30	5	25	-	8	32

3.3 Sample Preparation

3.3.1 Grinding

PP and MA-g-PP have been grinded individually via an industrial grinder (Motor brand: GAMAK, Power: 37 kW, Ampere: Max.67 A, Frequency: 50 Hz, Voltage: 400 V, Revolution: 1465 rpm). Figure 3.1 shows grinding process of PP and MA-g-PP. First of all, PP or MA-g-PP have been fed through hopper. Selected materials have been passed through grinding section with high speed for grinding. Later on, through a take up channel grinded materials have been passed to a collector.

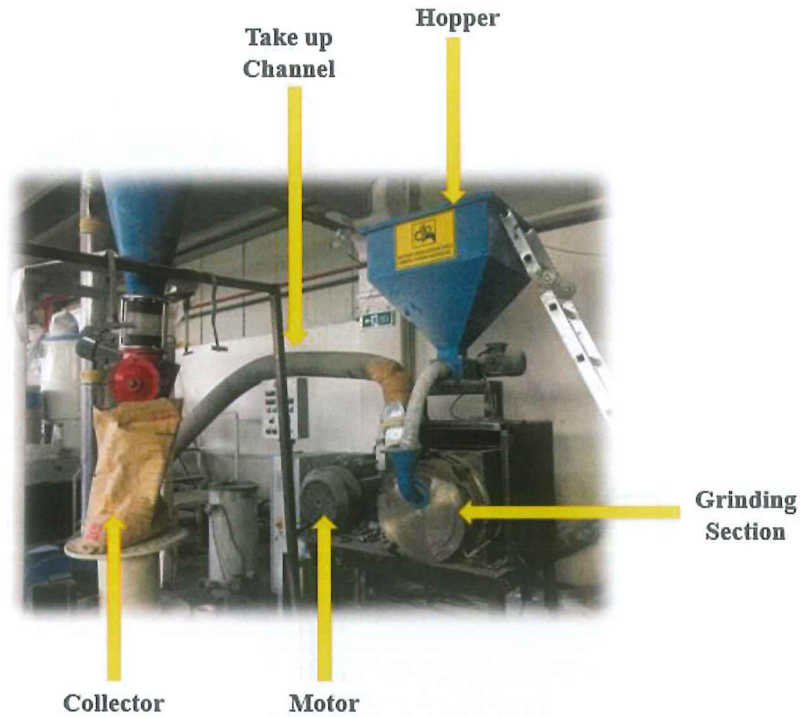


Figure 3.1: Grinding process of PP and MA-g-PP.

3.3.2 Fiber collection

Jute fiber has been obtained from jute twin (2 ply rope) by using a mechanical fiber cutting machine operated via 2 motors produced by GAMAK (Power: 1.5 kW, Ampere: Max. 3A, Voltage: 400V, Frequency: 50 Hz, Revolution: 2865 rpm) and 2 reducer produced by YILMAZ REDÜKTÖR (Reduction ratio: 7.25 and 12). Figure 3.2 shows process of jute fiber collection. Jute twin (2 ply rope form) have been fed to a cutting zone where rope formation of jute has been transformed to fiber form for collection.

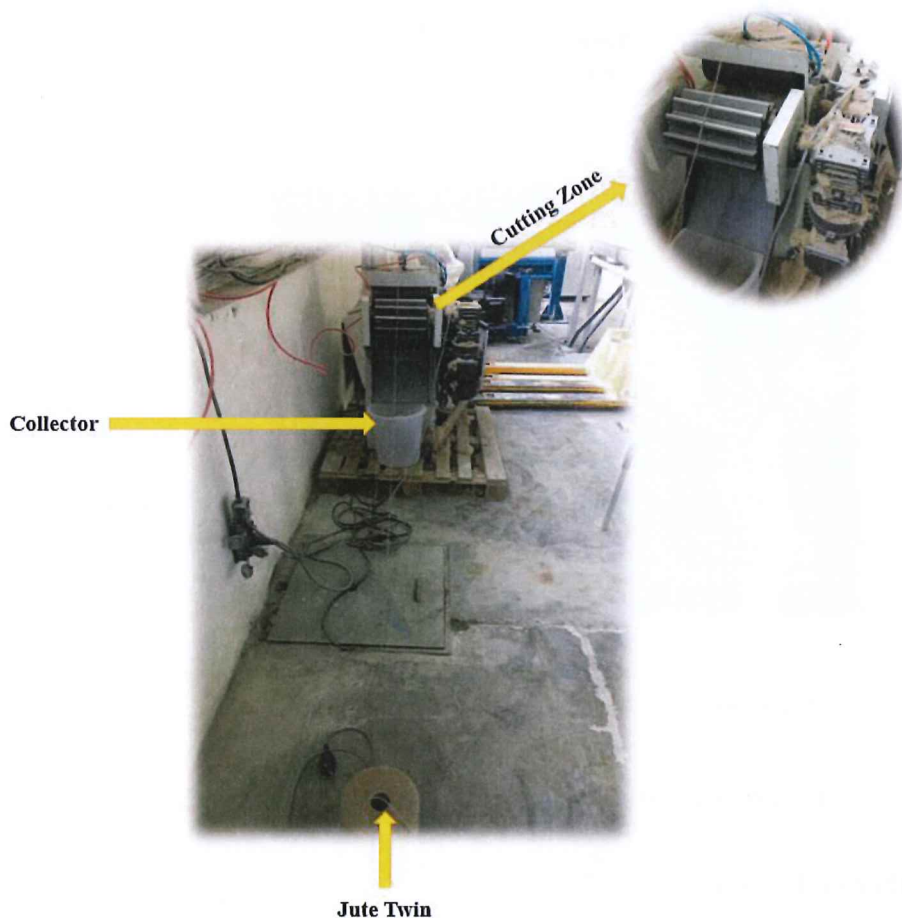


Figure 3.2: Fiber collection process.

3.3.3 Mechanical mixing

According to formulations (Table 3.1 and Table 3.2, materials are weighted and placed into a mechanical mixer produced by BOSFOR MAKINA (Model: UZK-T, Power: 3 kW, Ampere: 6A, Voltage: 380-460V, Frequency: 50-60 Hz, Revolution: 1200-1300 rpm and Capacity: Max. 12 kg). When temperature has been reached up to 70⁰-80 ⁰C within mixed materials, it has been considered as ‘ready’ for compounding via extrusion process. Total mixing time have been in between 40-60 minutes except LJF 20 (mixing time 15-20 minutes) according to formulations of Table 3.1. Total mixing time have been in between 60-80 minutes for all formulations of Table 3.2. Prime objective has been to ensure ‘homogenous mixture’ to ease melt compounding process via extrusion. Figure 3.3 represents process of mechanical mixing (stage 1 to stage 3) before compounding.

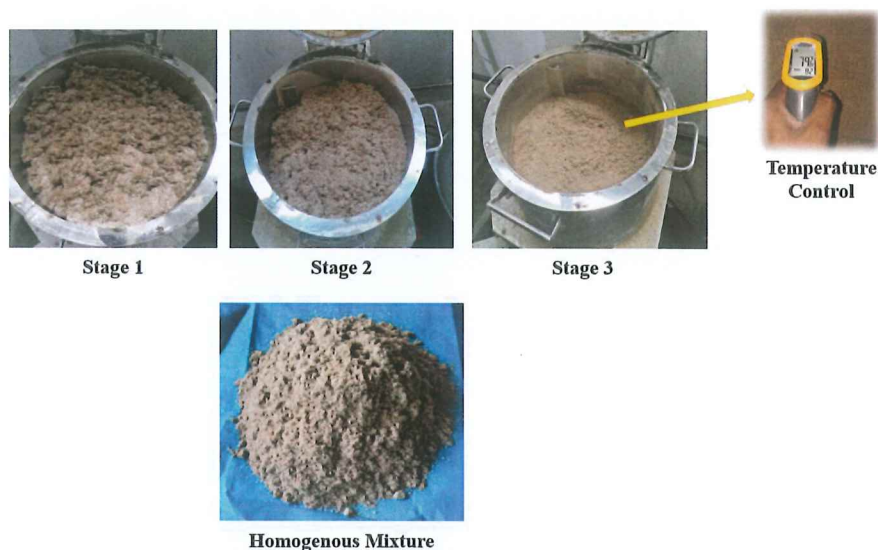


Figure 3.3: Process of mechanical mixing before compounding.

3.3.4 Melt compounding via extrusion process

Melt compounding of homogenous mixture have been carried out via a commercial twin extruder (E2X40) provided by Mimtek Makina San Tic Aş (Turkey) (Figure 3.4). Table 3.3 represents extrusion temperatures of six regions of twin extruder. Table 3.4 represents machine parameters for PP 100 and PP 95.

Table 3.3: List of temperatures for melt compounding via extrusion process.

Sample Code	Zone 1 (°C)	Zone 2 (°C)	Zone 3 (°C)	Zone 4 (°C)	Zone 5 (°C)	Zone 6 (°C)
PP 100	203.1	199.7	200.5	-	-	-
PP 95	203.1	199.7	200.5	-	-	-
LJF 20	174.8	180.9	186.9	174.3	189.6	185.5
SJF 20	174.8	180.9	186.9	174.3	189.6	185.5
SJF 30	171.2	180.4	187.7	171.3	196.7	184.5
SJF 40	171.2	180.4	187.7	171.3	196.7	184.5
SJF 50	172.1	181.1	184.9	175.1	204.2	192.9
SJF 60	172.1	181.1	184.9	175.1	204.2	192.9
30 (ATH_4/ZB_1)	170.3	180.8	194.0	195.0	200.2	210.7
40 (ATH_4/ZB_1)	170.3	180.8	194.0	195.0	200.2	210.7
30 (APP_4/ZB_1)	170.3	180.8	194.0	195.0	200.2	210.7
40 (APP_4/ZB_1)	170.3	180.8	194.0	195.0	200.2	210.7

Table 3.4: Machine parameters for PP 100 and PP 95.

Parameters	E2X40 EXT.
Rotational speed of Twin Extruder (Rev/Min)	70.4
Maximum Electric Flow Limit (A) - Working Electric Flow Limit (A)	24 - (14 to 15)
Mixing Speed (Rev/Min) (%)	4.3 (50)
Feeding Torque (Rev/Min) (%)	103-104 (100)
Velocity (%)	50
Spiral Torque (Rev/Min) (%)	16 (30)
Cold Cooling at Exit	Yes
40(APP_4/ZB_1)	30

It is to note that material degradation of jute fibers is a key factor to keep in mind while extrusion. For that reason, not only melt compounding temperatures differ but also machine parameters within twine extruder compared to PP 100 and PP 95. Table 3.5 represents machine parameters for LJF 20, SJF 20, SJF 30, SJF 40, SJF 50, SJF 60, 30 (ATH_4/ZB_1), 40 (ATH_4/ZB_1), 30 (APP_4/ZB_1), and 40 (APP_4/ZB_1).

Table 3.5: Machine parameters for LJF 20, SJF 20, SJF 30, SJF 40, SJF 50, SJF 60, 30 (ATH_4/ZB_1), 40 (ATH_4/ZB_1), 30 (APP_4/ZB_1), and 40 (APP_4/ZB_1).

Parameters	E2X40 EXT.
Rotational speed of Twin Extruder (Rev/Min)	52.8-69.1
Maximum Electric Flow Limit (A) - Working Electric Flow Limit (A)	24 - (17-18)
Mixing Speed (Rev/Min) (%)	5.2 (50)
Feeding Torque (Rev/Min) (%)	103-104 (100)
Velocity (%)	50
Spiral Torque (Rev/Min) (%)	16 (30)
Cold Cooling at Exit	Yes

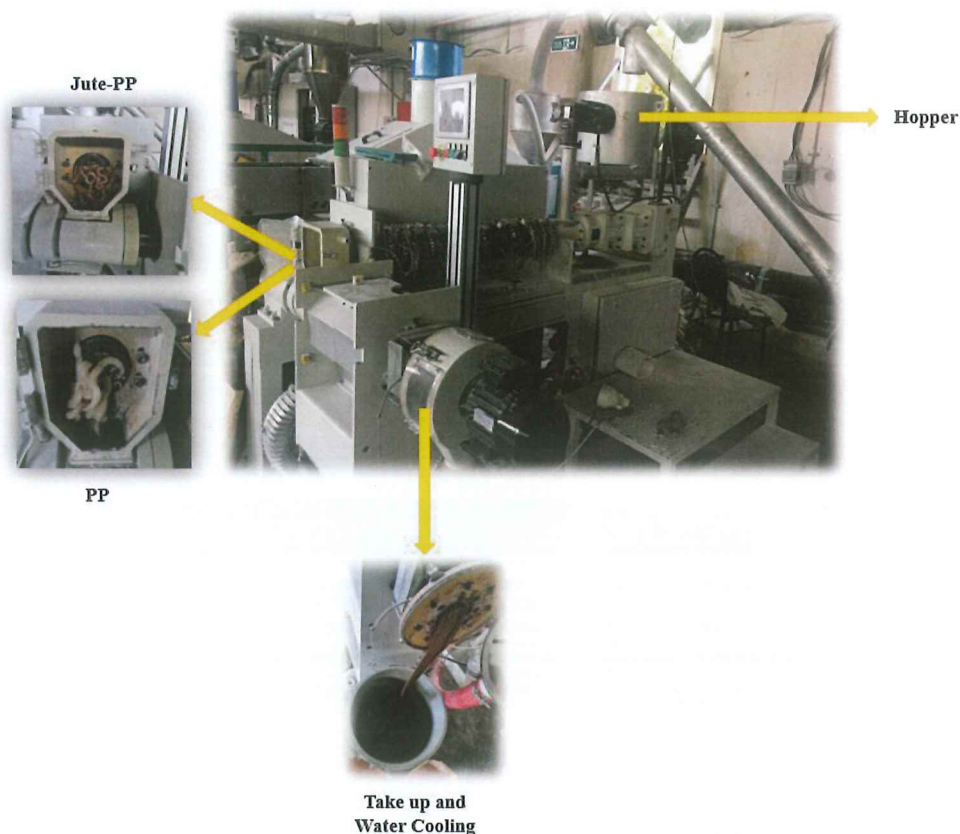


Figure 3.4: Melt compounding via extrusion process.

3.3.5 Granulation

Figure 3.5 represents process of granulation for all formulations regarding Table 3.1 and Table 3.2. After melt compounding via extrusion process, produced compound have been water cooled in room temperature for 30 minutes. Then, compound have been fed into a mechanical granulator (Power: 30kW, Ampere: 40-50A, Revolution:1200-1400 rpm) to obtain granules.

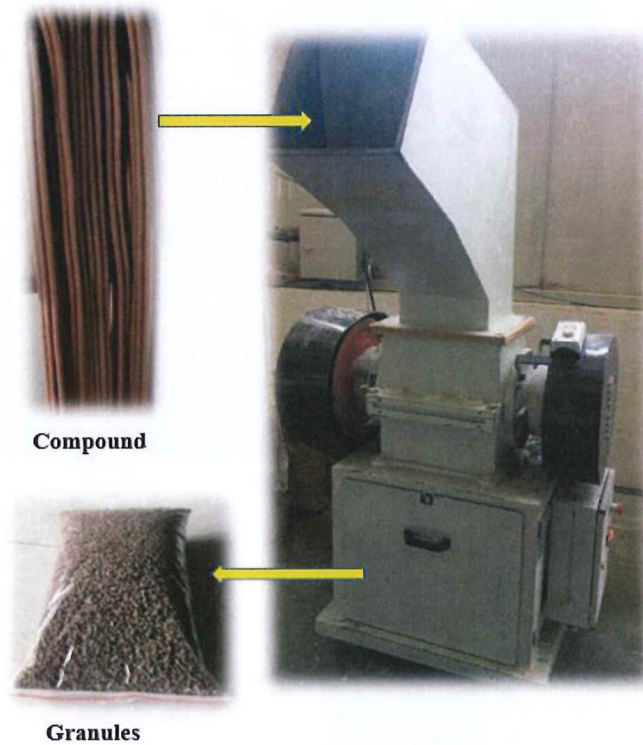


Figure 3.5: Process of granulation.

3.3.6 Composite preparation via hot press technique

Granules have been placed on testing mold to prepare test samples according to ASTM standards. Then, placed for hot pressing method (Figure 3.6). Testing conditions for hot press method for all samples have been as follows:

- ✓ Force: 8.5 Tonnes
- ✓ Temperature: 200 °C
- ✓ Time: 20 minutes

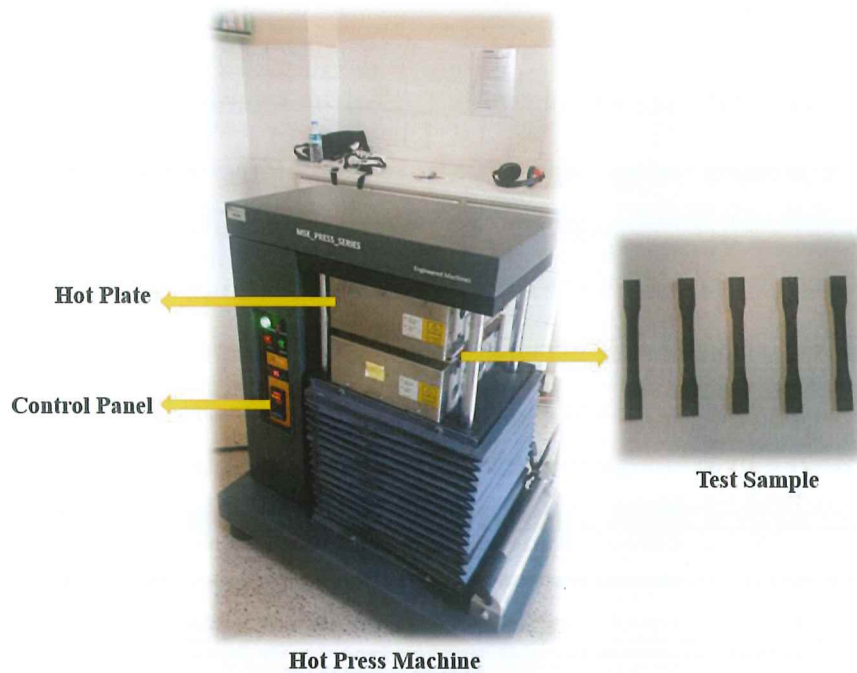


Figure 3.6: Composite preparation via hot press technique.

3.3.7 Overall manufacturing process

Figure 3.7 represents schematic of entire manufacturing process. Overall composite manufacturing process have been same for all formulations regarding Table 3.1 and Table 3.2 within the scope of this thesis.

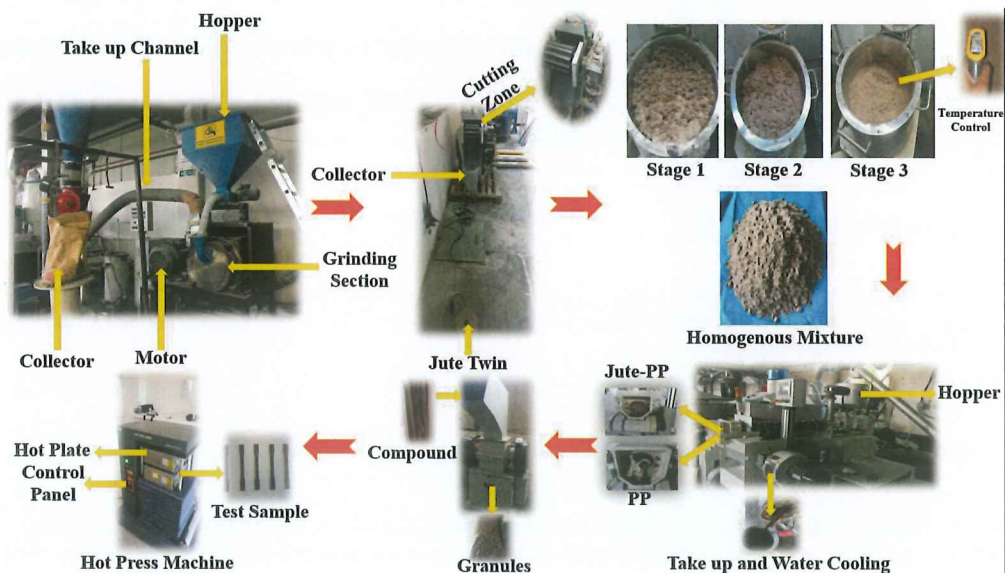


Figure 3.7: Schematic of complete manufacturing process.

3.4 Characterization

3.4.1 Morphological analysis via scanning electron microscopy (SEM)

The scanning electron microscopy (SEM) is one of the most frequently used instrumental method to characterize morphology, structure and composition regarding surface of selected sample materials at micro and nano scales [67–69].

Within the scope of this thesis, morphological analysis of all test samples has been performed via SEM (Tescan VEGA 3) in TEMAG laboratory, Istanbul Technical University. Before morphological characterizations, all samples have been collected from fractured surfaces of composite materials and then coated with gold-palladium alloy layer by sputter (Quorum SC7620) to control charge accumulation within the surface before placing into a microscopic chamber. During morphological observation, magnifications have been maintained as 100x, 500x and 1000x accordingly.

3.4.2 Mechanical tests

3.4.2.1 Tensile test

Tensile test is a well-known destructive technique which gives a detailed knowledge about strain to fracture (elastic to plastic region) of composite materials according to selected ASTM standards. It provides an in-depth information about the requirement of ‘force to break’ and ‘force for elongation at break’ of test sample within the range of the tensile test several information can be obtained through stress-strain curve as follows:

- ✓ Tensile strength
- ✓ Strain
- ✓ Young modulus
- ✓ Yield Strength
- ✓ Fracture stress [70].

Formulas regarding tensile test have been discussed below according to Equation 3.1 to Equation 3.3 [71]:

$$\text{Formula of yield strength } \sigma_y = \frac{F_y}{A_0} \quad (3.1)$$

σ_y : Yield strength (MPa);

F_y : Load at yield point (N);

A_0 : Cross-section area of test specimen (mm^2);

$$\text{Formula of ultimate tensile strength, } \sigma_u = \frac{F_{\max}}{A_0} \quad (3.2)$$

σ_u : Ultimate tensile strength (MPa);

$F_{\max}$: Maximum tensile load (N);

A_0 : Cross-section area of test specimen (mm^2);

$$\text{Formula of tensile strain at break, } \varepsilon_f = \frac{100 \times \Delta L_f}{L_0} \quad (3.3)$$

ε_f : Strain at break (%);

ΔL_f : Change of length at failure (mm);

L_0 : Original length (mm);

Within the scope of this thesis, tensile tests have been performed according to ASTM D638-10 via a universal testing machine (SHIMADZU AG-X plus series, Germany) with load cell of 100 kN at ITU Faculty of Textile Technologies and Design. Each specimen has been prepared according to dimension (length: 165 mm; length of narrow grip: 115 mm; width: 19 mm; gage length: 50 mm; gage width 13 mm; & thickness: 3.2 mm) of TYPE I (Figure 3.8) [72]. Cross head speed has been maintained at a constant rate of 5 mm/min. Tensile properties have been determined from the average values of 5 samples.

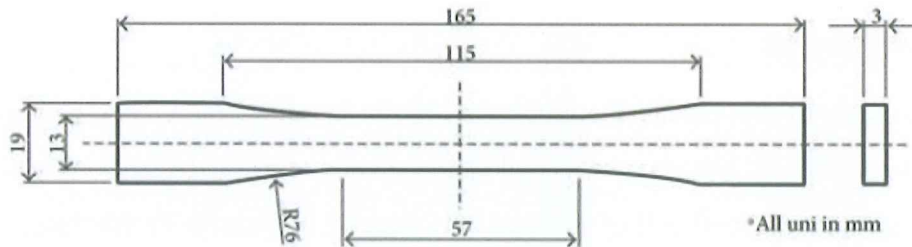


Figure 3.8: Tensile test specimen according to ASTM D638-10 (Type I) [72].

3.4.2.2 Flexural test (three point bending)

Flexural (3-point bending) test is a well-known method to optimize mechanical properties related to fiber reinforced polymer (FRP) composites. Several flexural properties like flexural strength, flexural strain, flexural modulus and yield stress can be optimized via flexural (3-point bending) test. To know flexural properties, firstly within the universal testing device a bending load is to be fixed with an apparatus. Then, sample material is to be fixed with 2 supporting pins by maintaining a certain span length. Within the scope of this test, span to thickness ratio differs (32:1 or 16:1). List of theoretical equations regarding flexural (3-point bending) test [73] have been provided according to Equation 3.4 and Equation 3.5 below:

$$\text{Formula of flexural stress, } \sigma = \frac{3FL}{2bd^2} \quad (3.4)$$

σ : Stress (MPa);

F: Load (N);

L: Span Length (mm);

B: Width (mm);

d: Thickness (mm);

$$\text{Formula of flexural strain, } \varepsilon = \frac{6hd}{L^2} \quad (3.5)$$

ε : Strain (%);

h: Displacement (mm);

d: Thickness (mm);

L: Span Length (mm);

Within the scope of this thesis, flexural tests (3point bending) have been performed according to ASTM D-790 via a universal testing machine (SHIMADZU AG-X plus series, Germany) with load cell of 1 kN at ITU Faculty of Textile Technologies and Design. Each specimen has been prepared with the length of 127 mm, width of 12.7 mm and thickness of 3.2 mm. Cross head speed has been maintained at a constant rate of 2 mm/min and span length have been maintained as 64 mm for all samples. Flexural properties have been determined from the average values of 5 samples.

3.4.2.3 Izod impact test (un-notched)

Izod impact test is conducted to calculate energy requirement by the help of a specified bar along with a pendulum. It can be carried out with samples in two forms: notched and un-notched. For un-notched test specimen requires no machining before proceeding the test and it requires during the need of breaking the test sample at a specified position. During test, test specimen is to be fixed with a clamp and pendulum is to be fixed at a specified degree which would be the release point for the entire test. When pendulum hits the test specimen, measurement of energy can be calculated within the test device via internal software. Fixing the test specimen to the clamp is critical because it might impact on the testing result. It is considered more here compared to accurate measurement of impact [74]. To calculate Izod impact strength of a test sample Equation 3.6 have been used:

$$\text{Impact Strength, } I = \frac{W_i}{b \cdot t} \quad (3.6)$$

I : Impact strength (kJ/mm²)

W_i : Impact energy (kJ);

b: Width (mm);

t: Thickness (mm);

Figure 3.9 shows a schematic of un-notched impact testing method.

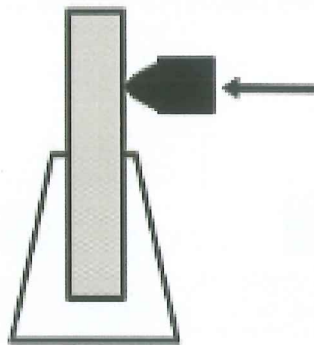


Figure 3.9: Un-notched Izod impact test [75].

Within the scope of this thesis, Izod impact testing have been carried out according to ASTM D-256 via COMTECH QC-639R (Impact pendulum tester) in room temperature at Nanocomposite Laboratory, Yeditepe University.

Un-notched test specimens have been produced according to the dimensions of length 63.5 mm, width 12.7 mm and thickness 3.2 mm. Energy have been used as 2.75 J. Angle have been maintained as 150°. Izod impact strength value has been calculated from the average of five samples of each formulation.

3.4.2.4 Density

Density measurement method is to use for ensuring concentration or purity of selected test samples. Definition of density for a test specimen is mass per unit volume. Measurement of density is generally depended on temperature. The reason is-contraction or expansion of substances of particle. A very common method to measure density is followed by Archimedes' principle where liquid immersion has been generally followed [76]. Archimedes technique is a combination of 2 measurements. One in air which is being considered as a reference fluid and another one is in preselected fluid. It can be distilled water, ethanol or acetone. This technique provides information about relative density of samples. Selection of secondary fluid is critical. It can have direct effect on density properties. Generally, ethanol is a good choice as a secondary fluid because distilled water causes air bubbles within the test sample. Formula of measuring density according to Archimedes is mentioned [77] ρ is density (g/cm³) and m is weight (g):

$$\rho_{\text{solid}} = \rho_{\text{fluid}} * \frac{m_{\text{air}}}{m_{\text{air}} - m_{\text{fluid}}} \quad (3.7)$$

Figure 3.10 represents Archimedes technique to calculate density of a solid sample.

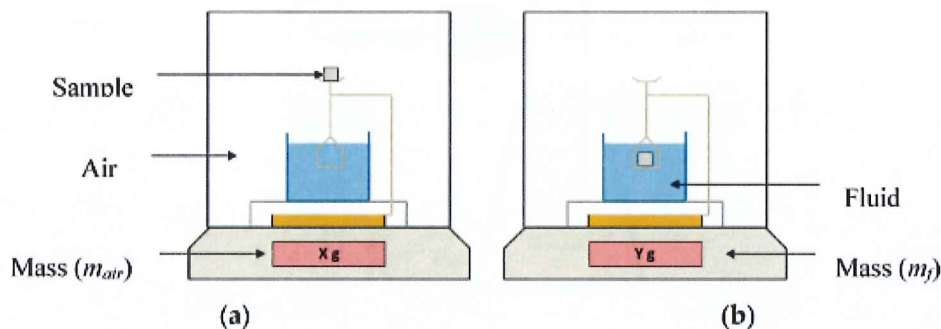


Figure 3.10: Archimedes technique: (a) test specimen measured in air (b) test specimen measured into the fluid [77].

Experimental density (g/cm^3) measurement have been carried out according to ASTM 4052 by ethanol alcohol immersion technique followed by Archimedes principle via a density measurement kit (RADWAG, Model: AS 220.R2, Poland) at Mimtek Makina San Tic Aş (Turkey). Density values have been measured from average of 5 measurements of each sample.

3.4.2.5 Wear test

Wear test is well known as ‘pin-on-disc’ test (Figure 3.12). For fiber reinforced polymer composites, wear and friction performance is necessary to analyze. During wear test, area of contact of remains consistent [78]. Within the scope of this test, a pin or ball by being stationary position presses against a disc that is within constant rotation. How much force is needed or how much force the pin or ball can restrain be keeping itself at a tangential position reflects on directly towards ‘friction measurement’. Wear displacement is measurable via a linear displacement transducer. Wear measurement is having direct relation to several aspects such as mass loss, gradual change within linear dimension, wear size analysis via optical view and measurement of wear volume via profilometric measurement [79].

Wear rate of test samples have been calculated according to Equation 3.8 [80]:

$$\text{Wear rate} = \frac{W_v}{L} \quad (3.8)$$

W_v : Volumetric wear loss

L : Sliding distance

Wear test have been carried out via a universal tribometer (Model: UMT-2, Bruker, Germany) at YUTAM (Erzurum Technical University) to investigate wear resistance and co-efficient of friction properties of jute-pp (SJF30) composites and flame retardant jute-pp composites ‘‘40 (ATH_4/ZB_1)’’ and ‘‘40 (APP_4/ZB_1)’’. Wear test parameters have been followed as- Applied load: 15N; Diameter: 4 mm (Al_2O_3 ball); Wear track: 18000 mm; Wear stroke length: 6 mm; Sliding speed: 10 mm/Sec; and Wear type: reciprocating (linear).

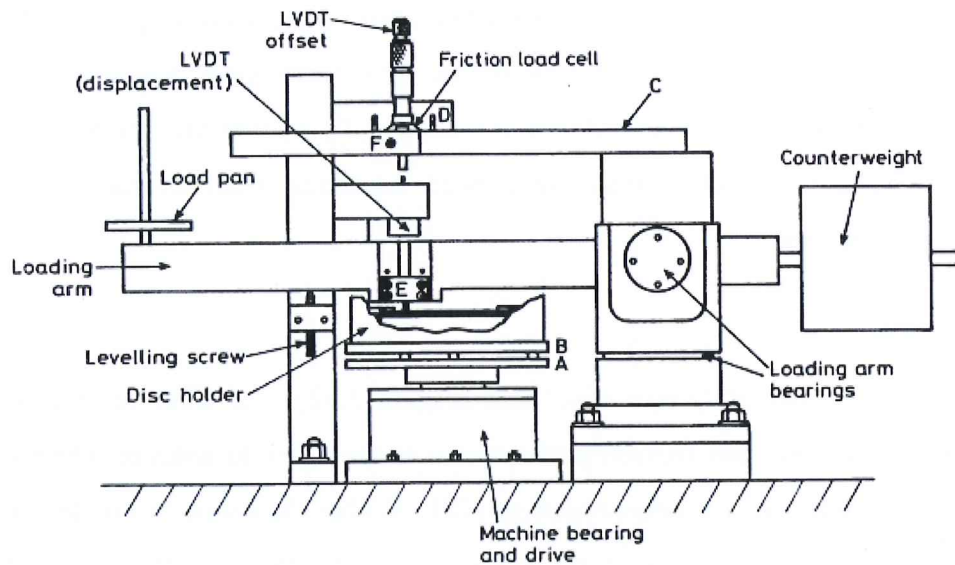


Figure 3.11: A schematic of pin-on disc test [79].

3.4.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a process of measuring mass of polymers. This measuring process is dependent of temperature or time within a controlled atmosphere. Commercially, TGA devices providing a stable temperature range of 1000°C or even more depending on application of selected polymers. Mass loss and mass gain of polymers both can be observed. Mass gain is related to heating rates at slow pace which causes degradation at an atmosphere that is in oxidization phase. Key reason for mass loss is 'evaporation of several volatile compound'. TGA method focuses on mass loss processes via its characterization techniques by extracting information related to thermal stability and composition. While performing TGA several parameters regarding temperature can have strong influence on desired results of test samples. These are as follows:

- ✓ Heating rate
- ✓ Thermal conductivity
- ✓ Enthalpy
- ✓ Test arrangement including sample, surface and sensor [81]

Within the scope of this thesis, to observe the thermal behavior of jute-pp and flame retardant jute-pp composites thermogravimetric analysis (TGA) have been performed at Thermal Analysis Laboratory and Nanotechnology Laboratory of Istanbul University-Cerrahpasa. In total, 7 samples have been considered for thermal characterization. All samples have been heated within the range of 20⁰C to 600⁰C in a nitrogen (N₂) atmosphere. A 5 g of each of 7 samples have been characterized via TGA heating rate of 10 ⁰C/min.

3.4.4 Fourier-transform Infrared Spectroscopy (FTIR) Analysis

Fourier-transform infrared (FTIR) analysis is known a convenient non-destructive method where calibration is not required externally. Within this method, scanning is very flexible due to variable speed. Sensitivity can be increased because its scanning capacity limit is to catch up every second and add all together for creating a ratio out random noise. Optical efficiency of this analysis method is excellent even with single part of motion. It is a well-defined method which excludes problems of dispersive devices. Major drawback has been slow scanning during analysis. There had been an extreme requirement to have test sample along with all infrared frequencies continuously. For those reasons, ‘interferometer’ had come which helped to input different type of signal with frequencies within all ranges. Each signal has been processed at each second such that reduction of time element for each sample occurred. An infrared energy emission occurs via a source which glows. Beam ensures its way within an aperture that is having the power to control energy level within the sample. Spectral encoding takes place at interferometer and a signal pass by. Later on, entry of beam occurs into the place of sample. There, transmission or reflection can be noticed. Absorption of energy with specified frequencies occurs within the sample surface, then beam reaches for final step towards detector for calculation accordingly. For clear measurement, an interferogram type of signal have been used. For focusing and making accurately parallel IR radiation, a mirror have is being placed. This mirror is basically holding value of low coefficient of thermal expansion. Each window is completely specified for each sample. Lastly, a digital signal measurement process has been used via a computer within the place of Fourier transformation. For result analysis, a clear presentation of ‘intensity vs frequency’ is to followed that has been created via an IR spectrum [82].

Within the scope of this thesis, Fourier transform infrared spectroscopy (FTIR) have been performed at 7 test samples including jute-pp and flame retardant jute-pp composites via Bruker ALPHA FTIR spectrometer at Iron and Steel Institute, Karabuk University. Number of recorded scans have been 24 maintained the range between 400-4000 cm^{-1} .

3.4.5 Flammability tests

3.4.5.1 UL-94 test (vertical burning)

UL-94 (vertical burning) test have been generally used for polymer-based material depending on applications. The prime objective of this test has been to characterize materials on the basis of heat and flame response within a balanced condition. Within this test method, characterization of burning behavior have been highly dependent on several factors such as after flame time, after glow time, flame application time, total burning time and most importantly dripping condition. Evaluation of test results depending on 'sample burning' from a 20 mm source of flame. Flame impingement time is 10 seconds. For each test specimen, this flame impingement followed times. Supplied gas should be 98% pure methane or natural gas with energy density value of 37 MJ/m^3 . Cotton ignition is to be followed strictly throughout the test method from the distance of test specimen (300 mm) which gives information about dripping phenomena (Figure 3.12). Materials classification (Table 3.6) according to the test standard is to be strictly followed. Total after flame time (t_f) for each set of 5 samples can be calculated by the Equation 3.9 below, where $t_{1,i}$ is the after-flame time regarding first flame application of i^{th} test sample (s) and $t_{2,i}$ is the after-flame time regarding second flame application of i^{th} test sample (s) [83].

$$t_f = \sum_{i=1}^5 (t_{1,i} + t_{2,i}) \quad (3.9)$$

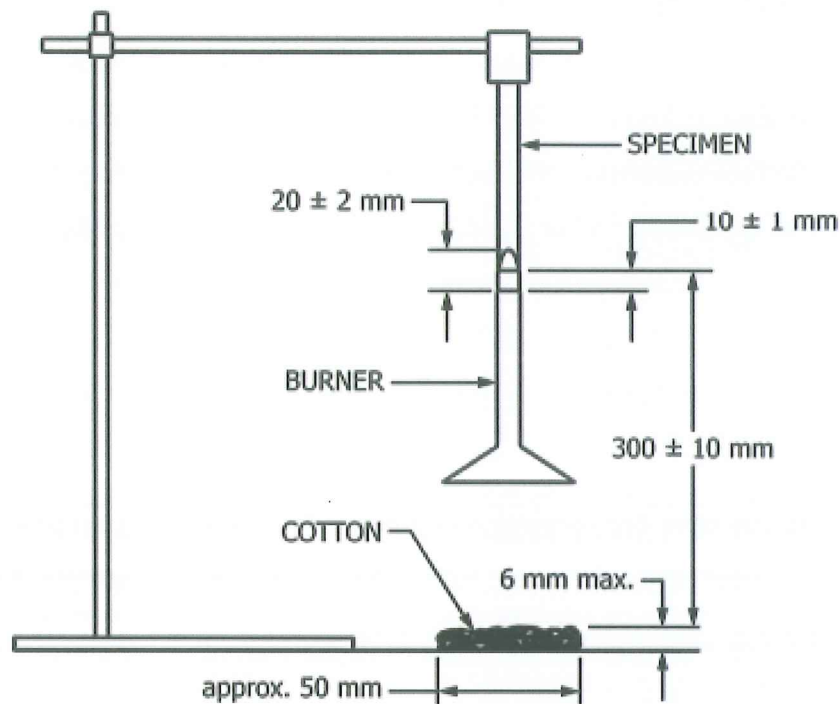


Figure 3.12: Vertical burning test method to determine burning classification (V-0, V-1 and V-2) [83].

Table 3.6: Standard classifications of UL-94 (Vertical Burning) test for materials [83].

Criteria Conditions	V-0	V-1	V-2
Afterflame time for each individual specimen, t_1 or t_2	$\leq 10s$	$\leq 30s$	$\leq 30s$
Total afterflame time for any condition set (t_1 plus t_2 for the five specimens)	$\leq 50s$	$\leq 250s$	$\leq 250s$
Afterflame plus afterglow for each individual specimen after the second flame application ($t_2 + t_3$)	$\leq 30s$	$\leq 60s$	$\leq 60s$
Afterflame or afterglow of any specimen up to the holding clamp	No	No	No
Cotton indicator ignited by flaming particles or drops	No	No	Yes

Within the scope of this thesis, vertical burning test (UL 94-VB) have been carried out via MTM-1 instrument at Mechanical Engineering Faculty of Istanbul Technical University according to ASTM D3801-20a. Geometry of each test specimen is $125 \times 13 \times 4 \text{ mm}^3$. Before conducting test, 2 sets of test specimen have been produced. Each set consist of 5 specimens. After producing test specimen according to standard dimension, specimens of first set have been placed into a room temperature prior to 48 hours of actual test. Relative humidity of the laboratory room has been 50%. Specimens of second set, have been kept into oven for 168 hours by maintaining 70°C prior to test.

Then, all specimens have been placed into a desiccator for cooling up to 4 hours at room conditioning just before the actual burning test. Average of 10 samples have been considered for the actual result of vertical burning test.

3.4.5.2 UL-94 (horizontal burning)

UL-94 (horizontal burning) test method have been generally used for investigating ‘linear burning rate’ of specified test specimen within a controlled condition by implementing heat and flame. This method has been widely used for polymer materials according to different applications. To investigate about linear burning rate of a sample, it is to be clamped tightly horizontally just above a wire gauze. Burner is to be fixed at 45° angle precisely according to Figure 3.13. It is important to know about the ‘behavior of selected material’. If shrinking occurs within a selected material after flame application for 30 seconds, material is being considered as an ‘unsuitable’ candidate for this test. Too much distortion of test samples should not be an acceptable condition while proceeding this test. When flame reaches to 25 mm mark of test specimen, a complete withdrawal of burner should be carried out. Then, burning time (t) is to be considered. This burning time have been referred as ‘elapsed time’. If the test specimen continues to burn and reaches to 100 mm mark, elapsed time is being calculated. If the test specimen does not reach to 100 mm mark, then it is important to consider the elapsed time according to the point of stoppage of burning. Equation 3.10 is to be used for calculating linear burning rate where L is the length (mm) of burning section after the test, t is the required time to burn (s) and V is the linear burning rate (mm/min) [84]:

$$V = \frac{60 L}{t} \quad (3.10)$$

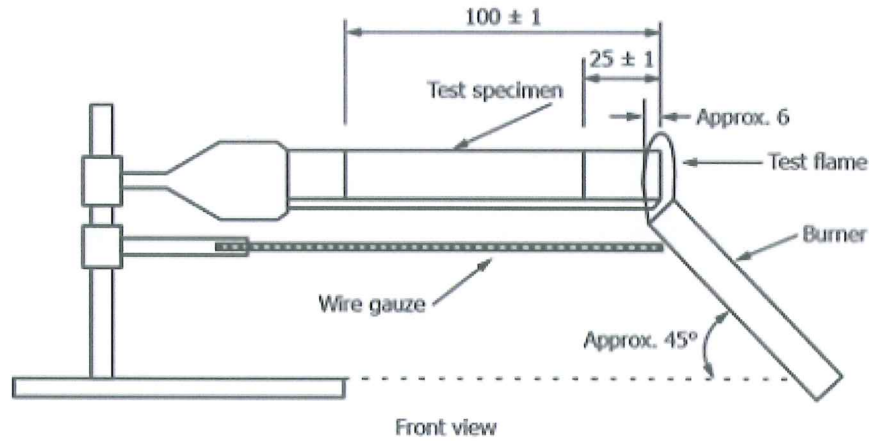


Figure 3.13: Horizontal burning test method to determine burning rate [84].

Within the scope of this thesis, horizontal burning test (UL 94-HB) have been carried out via MTM-1 instrument at Mechanical Engineering Faculty of Istanbul Technical University according to ASTM D635-18. Geometry of each test specimen is $125 \times 13 \times 4 \text{ mm}^3$. Before conducting test, 10 test specimens have been selected for conditioning for 48 hours at conditioning atmosphere. All of these test specimens have been taken out from conditioning atmosphere before 1 hour prior to conducting the test. Temperature of lab atmosphere have been around 15° to 35° C with relative humidity of less than 75%. Before conducting this flame test, test specimen has been placed tightly with a clamp horizontally just above to a wire gauze where burned portion can be dripped. Application of flame have been maintained for 30 seconds at a flame height of 20 mm into a sample depth of 6 mm without any break. Flaming position remained at 45° angle. Linear burning rate have been calculated from the average value of 10 samples from each formulation.

3.4.5.3 Limiting oxygen index (LOI)

Limit Oxygen Index (LOI) test (Figure 3.14) is to measure the oxygen concentration level at minimum stage via a gas mixture of oxygen and nitrogen which will help to proceed flame combustion of selected polymer materials [85]. Information about minimum oxygen concentration helps for characterization of flammable behavior of materials at environment. Ignition behavior can be criticized well at atmospheric condition because there is 20.8% of oxygen and 78% of nitrogen exist at our atmosphere.

Since, oxygen and nitrogen concentration are fixed within our environment, mixture of oxygen and nitrogen at various concentrations directly can affect into the burning manner of selected test specimen.

While proceeding test, provided flame ignition remains from the top of the mounted tube where test specimen is to be fixed for ignition. To observe into the ‘length of burn’ is critical because if it crosses above 50 mm it is to be considered as ‘failed test’. With the result of minimum oxygen concentration, burning behavior can be discussed briefly [86]. Classification of LOI test can be segmented into 3 zone such as: flammable ($LOI < 20.95$), slow burning ($21 < LOI < 28$) and self-extinguishing ($28 < LOI < 100$) [87].

Limit oxygen index (LOI) tests have been carried out according to ASTM D2863 via a laboratory scale LOI device (Brand: PROWHITE, Model: K016-OI-1X, Turkey) at BUTEKOM, Turkey. Ignition of flame have been maintained for 180 seconds. As a conditioning parameter room temperature have been maintained as $20 \pm 2^\circ \text{C}$ and relative humidity have been maintained as $65 \pm 4\%$. Fifteen test specimens from each sample have been considered during the test with dimension of $100 \times 10 \times 4 \text{ mm}^3$. Average values of 15 specimens from each sample have been taken out for the standard LOI test result.

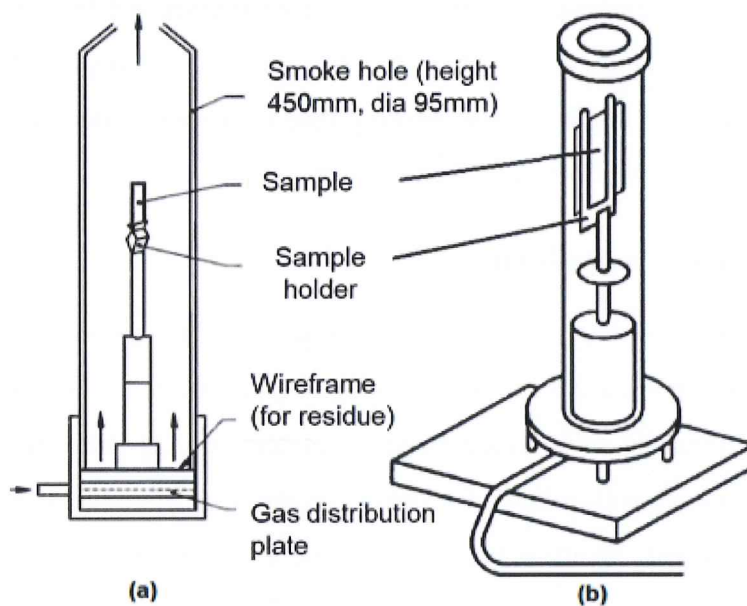


Figure 3.14: Standard limit oxygen index (LOI) test method [85].

3.4.5.4 Cone Calorimetry

Cone calorimetry test analysis is to characterize burning behavior of polymer materials via heat release mechanism from a burned test specimen. Burning of the test specimen is directly connected to consumption of oxygen concentration while maintaining the combustion process. Heat flow range is generally in between 10 kW/m^2 to 100 kW/m^2 . While proceeding the test, sample have been placed into a closed tray with an enclosure of aluminum foil (Figure 3.15). Entire testing process is very sensitive to heat exposure. Such that- mass loss rate of test specimen can be determined while proceeding the combustion process. Several functions are available to characterize the burning behavior of test specimen along with this test. Such as:

- ✓ Peak heat release rate (pHRR)
- ✓ Heat release rate (HRR)
- ✓ Total heat release rate (THR)
- ✓ Mass loss rate (MLR)
- ✓ Effective heat combustion (EHC)
- ✓ Time to ignition (TTI)
- ✓ Smoke production rate (SPR) and so on [88].

Cone calorimetry test have been carried out according to ASTM E-1354 via Fire Testing Technology (FTT) (Model: iCone® Classic, United Kingdom) at BUTEKOM, Turkey. Heat flux at 50 kW/m^2 . Test conditions have been followed by maintaining room temperature at 23°C and relative humidity have been maintained as 50%. A total of 5 test specimens from 1 sample have been considered during the test with dimension of $100 \times 10 \times 4 \text{ mm}^3$. Average values of 5 specimens for 1 test specimen have been taken out for the standard cone calorimetry test result.

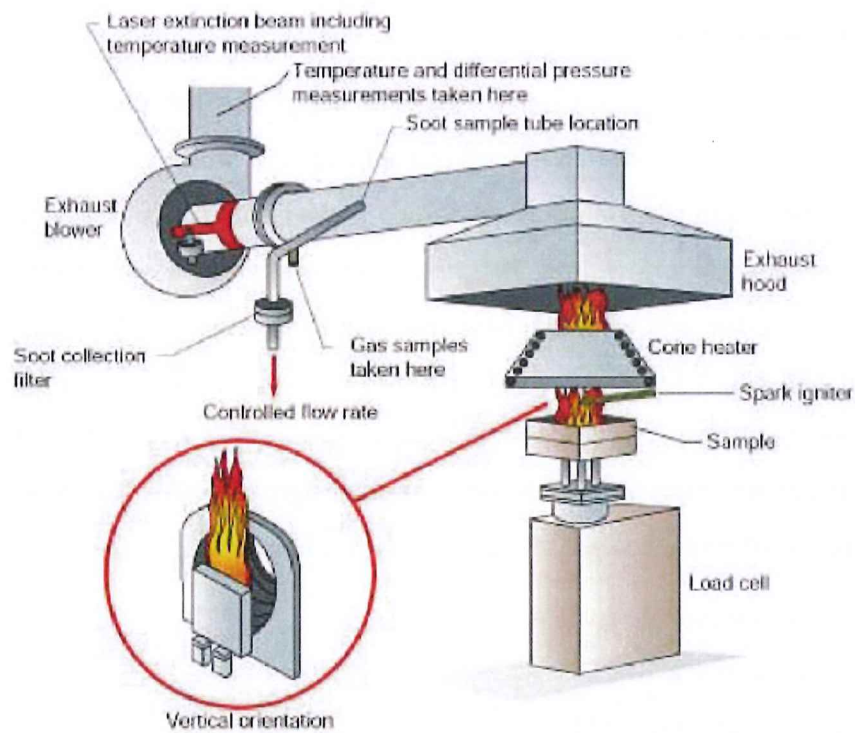


Figure 3.15: Cone calorimetry test method [89].

4. RESULTS AND DISCUSSION

4.1. Scanning Electron Microscopy (SEM) Analysis

4.1.1 Microstructure and morphology analysis of short jute fiber (SJF) and long jute fiber (LJF) with polypropylene (PP)

Fractured surfaces of tensile samples regarding LJF 20 and SJF 20 have been analyzed via SEM to understand the microstructure and morphology of short jute fiber (SJF) and long jute fiber (LJF) along with Polypropylene (PP) matrix. Figure 4.1 represents SEM micrographs of LJF 20 and SJF 20. A very little difference has been observed in both samples. Fractures are clearly visible from micrographs of LJF 20 and SJF 20. A good interfacial bonding in between jute fibers and polypropylene matrix in the presence of MA-g-PP has been noticed from SEM micrographs of LJF 20 and SJF 20. By introducing high amount of jute fibers into polypropylene matrix, produced composite materials can be easily triggered by fiber debonding and fiber pull out. However, SJF 20 is not having an increase amount of fiber pull out compared to LJF 20 which is greatly responsible for decreasing or increasing mechanical performance.

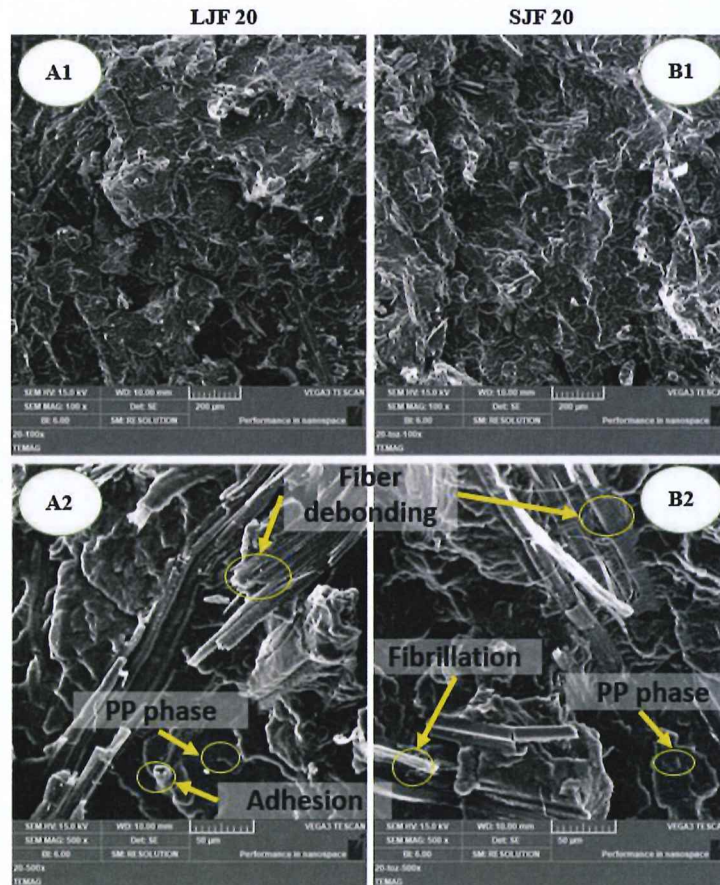


Figure 4.1: SEM micrographs of jute/PP composites (A1-A2) LJF 20 and (B1-B2) SJF 20.

4.1.2 Microstructure and morphology analysis of short jute fiber (SJF) with polypropylene (PP) at different loading levels

Fractured surfaces of tensile samples of SJF 30, SJF 40, SJF 50 and SJF 60 have been analyzed via SEM to understand the microstructure and morphology of short jute fiber (SJF) along with Polypropylene (PP) matrix at different loading levels (30 wt.%, 40 wt.%, 50 wt.% and 60 wt.%) in the presence of MA-g-PP. Figure 4.2(a) and Figure 4.2(b) represent SEM micrographs of SJF 30 and SJF 40 respectively. Figure 4.3(a) and Figure 4.3(b) represent SEM micrographs of SJF 50 and SJF 60 respectively. It is clear from SEM micrographs of all samples (Figure 4.2 and Figure 4.3) that adding more than 30 wt.% of jute fibers with PP in terms of weight percentage is not so suitable to increase mechanical properties. Key reasons lying behind that are ‘un-even homogenization’, ‘poor adhesion’ and ‘fiber pull out’. When fiber pull out occurs, there is a tendency to carry less load which is greatly responsible for reducing mechanical performances, mainly tensile strength.

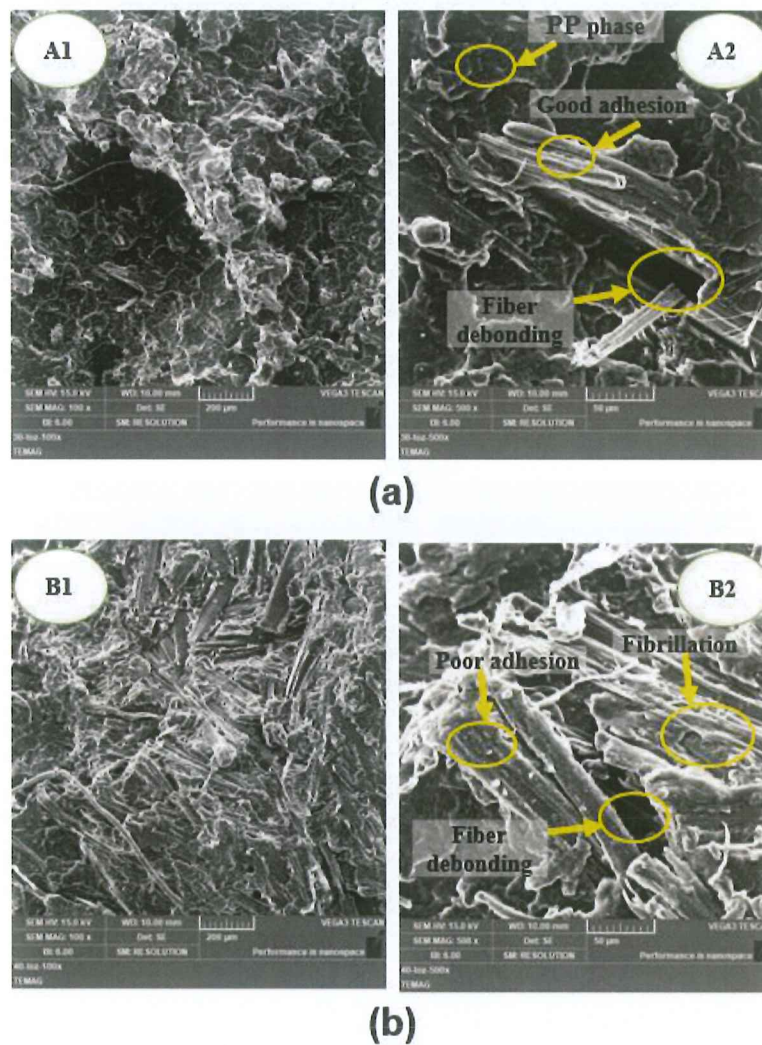


Figure 4.2: SEM micrographs of jute/PP composites: (a) SJF 30 and (b) SJF 40.

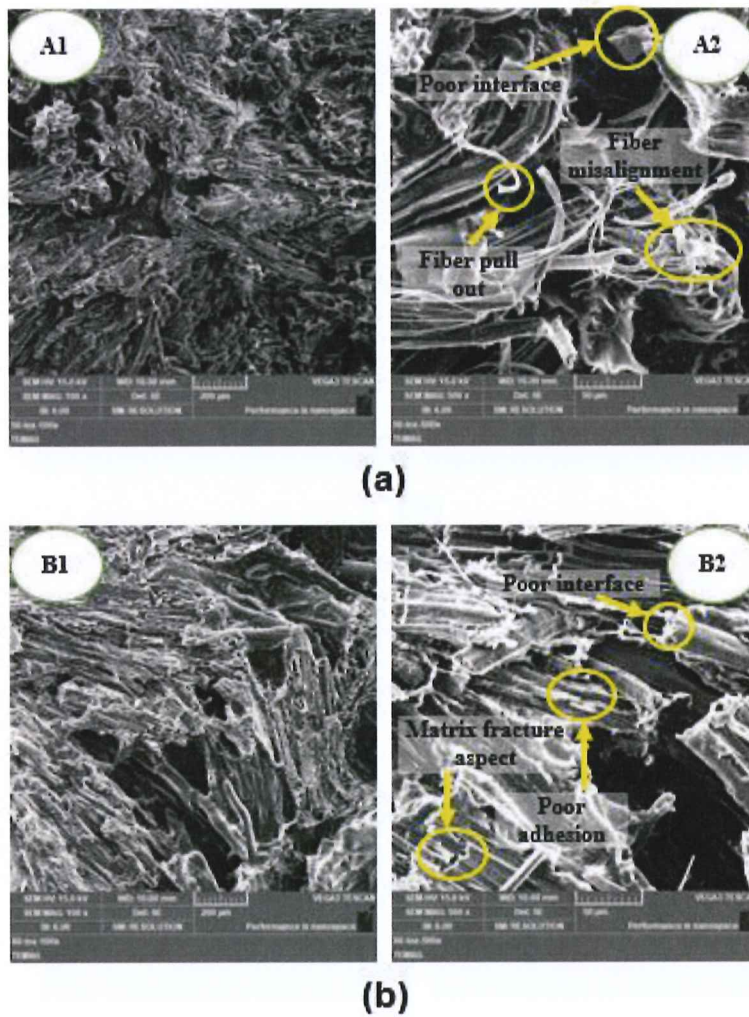


Figure 4.3: SEM micrographs of jute/PP composites: (a) SJF 50 and (b) SJF 60.

4.1.3 Microstructure and morphology analysis of short jute fiber (SJF), polypropylene (PP) and flame retardants (FRs)

Figure 4.4(a) & Figure 4.4(b) represent SEM micrographs of fractured surfaces of tensile samples of flame-retardant jute-PP composites regarding 30 (ATH_4/ZB_1) and 40 (ATH_4/ZB_1). Figure 4.5(a) and Figure 4.5(b) represent SEM micrographs of fractured surfaces of tensile samples of flame-retardant jute-PP composites regarding 30 (APP_4/ZB_1) and 40 (APP_4/ZB_1). There are several key factors which are directly related to mechanical properties of flame-retardant jute-pp composites as follows:

- ✓ Size and distribution of flame-retardant fillers [90] along with jute, PP and MA-g-PP.
- ✓ Weight percent of fillers [90] within formulations
- ✓ Co-relation of physical properties [90] in between flame-retardant fillers and PP
- ✓ Interfacial bonding of flame retardant fillers [90] and PP

Interfacial bonding is having a significant impact because it is directly related to the immediate effect within mechanical properties. It is directly responsible for changing the deformation process locally and tending to break the process. It is also having a significance regarding the change of crystalline behavior [90]. From Figure 4.4, it is clearly seen that compatibility of ATH/ZB is noticeable along with jute fiber, PP and MA-g-PP. Even with poor adhesion, amount of fiber pull out is less visible (Figure 4.4(a)) for '30 (ATH_4/ZB_1)'. However, high amount fiber pulls out and voids (air trapped) are being noticed (Figure 4.4(b)) for '40 (ATH_4/ZB_1)' which can cause a negative effect on mechanical performances and poor flame retardancy. 'Matrix cracking' and 'fiber pull out' are other possible reasons attributed for overall poor mechanical performances of '30 (ATH_4/ZB_1)' and '40 (ATH_4/ZB_1)' compared to '40 (APP_4/ZB_1)'.

On the other hand, from Figure 4.5(a) it is clearly seen that-MA-g-PP is fully compatible with jute, PP, APP and ZB. However, formation of poor adhesion is visible which can be attributed to lack of proper homogenization while mixing for '30 (APP_4/ZB_1)'. Due to this reason, there might be a high chance to have poor mechanical performances and flame retardancy regarding to '30 (APP_4/ZB_1)'. Better interfacial adhesion and less fiber pull out have been observed (Figure 4.5(b)) for '40 (APP_4/ZB_1)' which can be attributed towards better mechanical performances and flame retardancy. Agglomeration becomes less due to increasing of flame retardants. Hence, mechanical properties and flame-retardant properties of flame-retardant based jute-PP composites have been significantly improved.

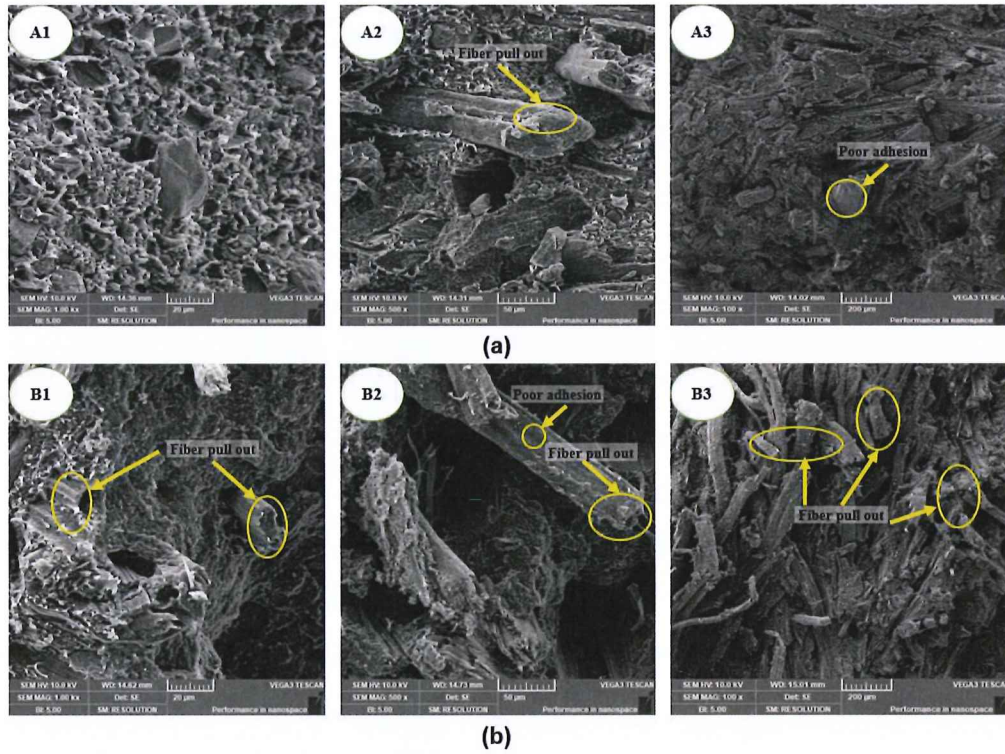


Figure 4.4: SEM micrographs of flame retardant jute-pp composites:
(a) 30 (ATH_4/ZB_1) and (b) 40 (ATH_4/ZB_1) at different magnifications.

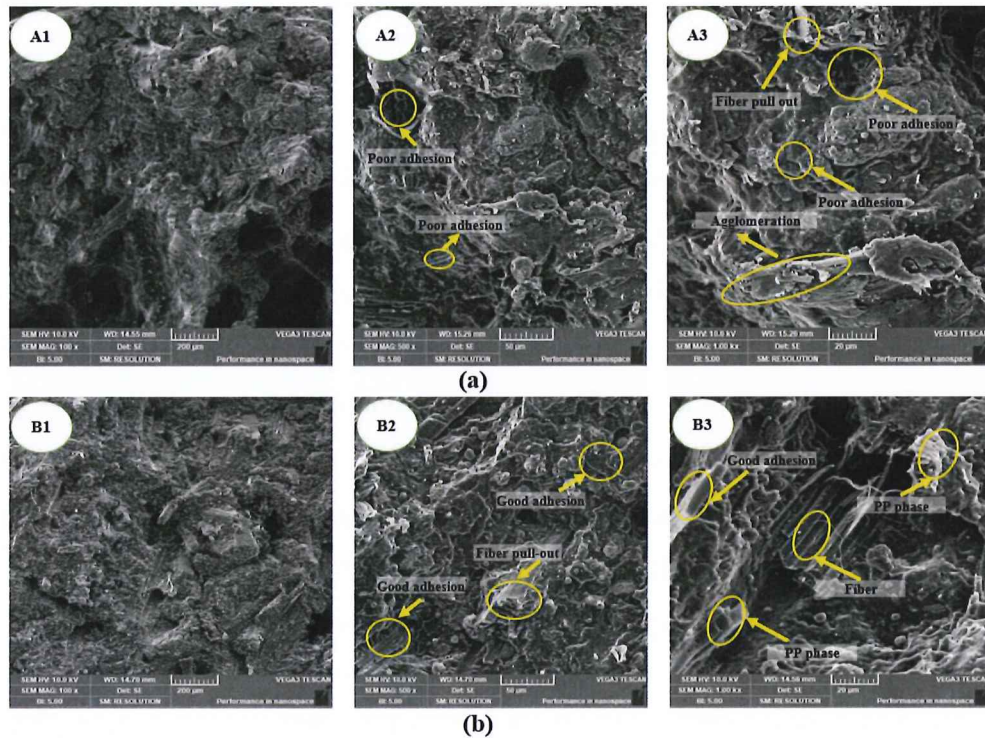


Figure 4.5: SEM micrographs of flame retardant jute-pp composites:
(a) 30 (APP_4/ZB_1) and (b) 40 (APP_4/ZB_1) at different magnifications.

4.2. Tensile Test Results Analysis

4.2.1 Effect of coupling agent & reinforcer length

PP is basically a non-polar polymer [91] and jute fiber is fully polar because it is having polar groups within its backbone [91,92]. Such that, to incorporate with each other with better interfacial adhesion MA-g-PP is being used with 5 wt.%. Here, prime aim of using MA-g-PP is to create an immediate chemical reaction in between anhydride group and hydroxyl group of jute fibers [93]. From Figure 4.6 and Table 4.1, it is clearly observed that- when MA-g-PP have been incorporated with PP the performance of tensile strength and strain values have been increased very low compared to Neat PP (PP 100). However, when jute yarn has been incorporated in the form of 'long jute fiber (LJF 20)' and 'short jute fiber (SJF 20)' with PP (75 wt.%) in the presence of 5 wt.% of MA-g-PP tensile strength and young modulus of jute-pp composites have been increased noticeably. On the contrary, strain values have been decreased sharply. When 20 wt.% of jute fibers have been incorporated with PP and MA-g-PP, ultimate tensile strength values have been achieved up to 37.32 ± 0.75 MPa for SJF 20 which shows short jute fiber have been homogenously compounded compared to 20 wt.% of long jute fibers (LJF 20). LJF 20 have been reached up to 32.98 ± 0.74 MPa. Strain values of jute-PP composites (LJF 20 and SJF 20), have been decreased up to 76.60-81.36% (average) compared to MA-g-PP incorporated PP (PP 95). Besides that, 22.50-44.07 % (average) increment have been noticed within young modulus or tensile modulus. Stress transfer occurs from the matrix to the fiber with more enough interfacial bonding [91].

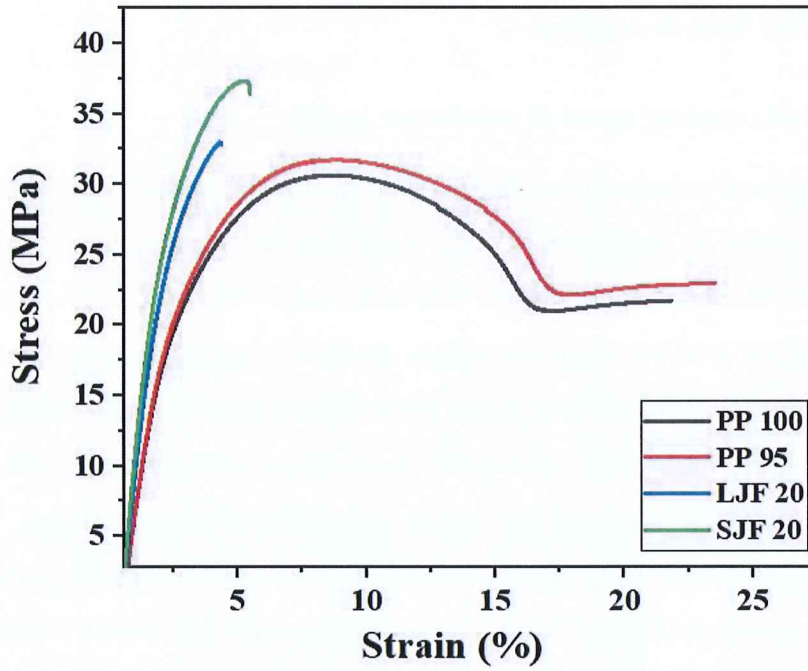


Figure 4.6: Effect of coupling agent and reinforcer length in tensile strength.

Table 4.1: Tensile test results of PP 100, PP 95, LJJF 20 and SJF 20.

Sample Code	Ultimate Tensile Strength (MPa)	Strain (%)	Young Modulus (MPa)
PP 100	30.62±0.69	21.81±2.93	1251±60
PP 95	31.73±0.74	23.51±7.66	1257±56
LJJF 20	32.98±0.74	4.38±0.14	1540±105
SJF 20	37.32±0.75	5.50±0.14	1811±81

4.2.2 Effect of short jute fiber loading

Figure 4.7 and Table 4.2 shows the influence of SJF on tensile strength. Loading level of SJF have been maintained up to 60 wt.% into PP in the presence of constant 5 wt.% MA-g-PP. Tensile strength has been increased incredibly up to 37.32 MPa when 20 wt.% of SJF have been incorporated to PP including MA-g-PP. There has been a strong recommendation from literatures to use MA-g-PP for achieving better interfacial adhesion within PP and short jute fibers [94,95].

Wetting behavior of jute fibers [95] have been also established by the help of PP (matrix). Internally, a chemical (ester bond) and physical (hydrogen bond) co-relation have been established in between MA-g-PP and cellulose of jute fibers.

A better adhesion behavior has been noticed via SEM (Figure 4.2(a)) in between jute fibers and PP (matrix) which helps to achieve better mechanical properties in terms of tensile strength. A shear amount of stress has been transferred [93]. However, when short jute fiber content has been increased gradually by 10 wt.% tensile strength values have been tending to decrease sharply. SJF 30 shows a clear decrease of 21% compared to SJF 20 and 3.72% compared to PP 100 in tensile strength values. When short jute fiber (SJF) content has been increased up to 40 wt.%, tensile strength values have been dropped significantly up to 37.91% compared to PP 100. However, a slight increase in tensile strength has been noticed for SJF 60 (19.5 ± 2.04 MPa) compared to SJF 50 (18.47 ± 3.34 MPa). Possible reason is attributed to homogeneity of fibers or deviation of temperatures during extrusion. It is clearly seen that- when wt.% of short jute fiber have been increased tensile strength values have been decreased. Possible reasons are attributed towards:

- ✓ ‘Ductile to brittle fracture’ transition
- ✓ Un-even stress transfer
- ✓ Agglomeration of fibers [95].

With the increase of fiber content, strain values decrease. A sharp decrease of 50.18% have been observed in comparison with SJF 20 regarding SJF 60. Moreover, a clear increase (44.7%) has been observed for SJF 20 compared to PP 100 regarding young modulus. Reason is attributed towards ‘good interfacial adhesion’ or ‘strong chemical bonding’ of short jute fiber and PP (matrix) [95]. But young modulus remains consistent when short jute fiber content is in the range of 30 wt.%-60 wt.% which ensures transitioning elastic deformation without showing any types of internal fractures [95]. From above discussion, it can be concluded that SJF 30 is the best choice to choose without lessen the tensile properties of jute-pp composites.

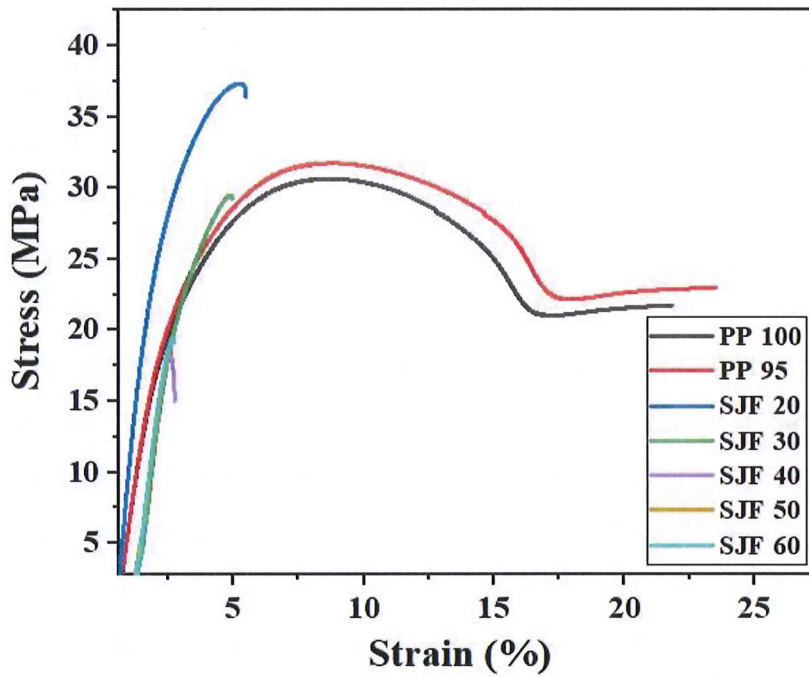


Figure 4.7: Effect of short jute fiber loading in tensile strength.

Table 4.2: Tensile test results of PP 100, PP 95, SJF 20, SJF 30, SJF 40, SJF 50 and SJF 60.

Sample Code	Ultimate Tensile Strength (MPa)	Strain (%)	Young Modulus (MPa)
PP 100	30.62±0.69	21.81±2.93	1251±60
PP 95	31.73±0.74	23.51±7.66	1257±56
SJF 20	37.32±0.75	5.50±0.14	1811±81
SJF 30	29.50±2.74	4.97±0.84	1517±198
SJF 40	19.01±1.74	2.76±0.17	1439±71
SJF 50	18.47±3.34	2.56±0.14	1421±198
SJF 60	19.50±2.04	2.74±0.24	1468±154

4.2.3 Effect of loading of flame retardants (FRs) with short jute fiber (SJF)

Including 30 wt.% of flame retardants (ATH/ZB) with SJF 30 have been significantly reduced tensile strength. Possible reason is attributed towards ‘Brittleness’ [96]. On the other hand, including 30 wt.% of flame retardants (APP/ZB) with SJF 30 have been significantly increased tensile strength. Possible reason is attributed towards proper homogenization and compatibility of APP/ZB with jute/PP composites.

However, '30 (ATH_4/ZB_1)' have been showed poor performance (25.23 MPa) compared to '30 (APP_4/ZB_1)' due to poor adhesion within fiber and matrix in fracture mode [97].

Moreover, when 40 wt.% of flame retardants have been incorporated with SJF 30, '40 (ATH_4/ZB_1)' have been shown an immediate negative impact of including FRs due to poor adhesion [98], fiber pull out and voids (Figure 4.4(b)). Maximum tensile strength has been achieved up to 34.18 ± 2.02 MPa. A clear increase of 15.18% compared to SJF 30 and of 11.62% compared to neat PP. Compounding with high loading of inorganic materials (APP and ZB) as fillers with jute and PP causes to show better interfacial adhesion which leads to increase tensile properties. Less strain values formed due to fiber debonding and unwanted voids [99]. From Table 4.3 and Figure 4.8, it is clear that- including 'ATH-ZB' or 'APP-ZB' flame retardants in polymer based natural fiber composites causes an immediate decrease within its strain values. Young modulus has been increased noticeably for all flame retardants based jute/PP composites compared to PP 100 and PP 95 except '40 (ATH_4/ZB_1)'. For ATH-ZB flame retardant-based jute-pp composites range of increment have been in between 26.5% to 27% compared to PP 95 and PP 100. '40 (APP_4/ZB_1)' flame retardant-based jute-PP composites have been shown maximum level of young modulus up to 232.5% in comparison with PP 95 which attributes towards 'hardening effect within APP and PP matrix'. While compounding, particles of APP tend to restrict the position of long chain of PP. Due to this reason, modulus of elasticity have been improved [100]. Area of plastic deformation have been reduced due to inclusion of flame retardants with jute/PP composites (Figure 4.8). However, '30 (APP_4/ZB_1)' have been performed poorly in terms of young modulus increment compared to '40 (APP_4/ZB_1)', only 171.7% increase have been achieved comparing PP 95. Compared to SJF 30, ATH-ZB flame retardants-based jute-pp composites are better performer in terms of young modulus. But '40 (APP_4/ZB_1)' have been consistently better (175.52% compared to SJF 30). Possible reason of reduced young modulus value for '40 (ATH_4/ZB_1)' is attributed to unexpected temperature rise while compounding via extrusion process.

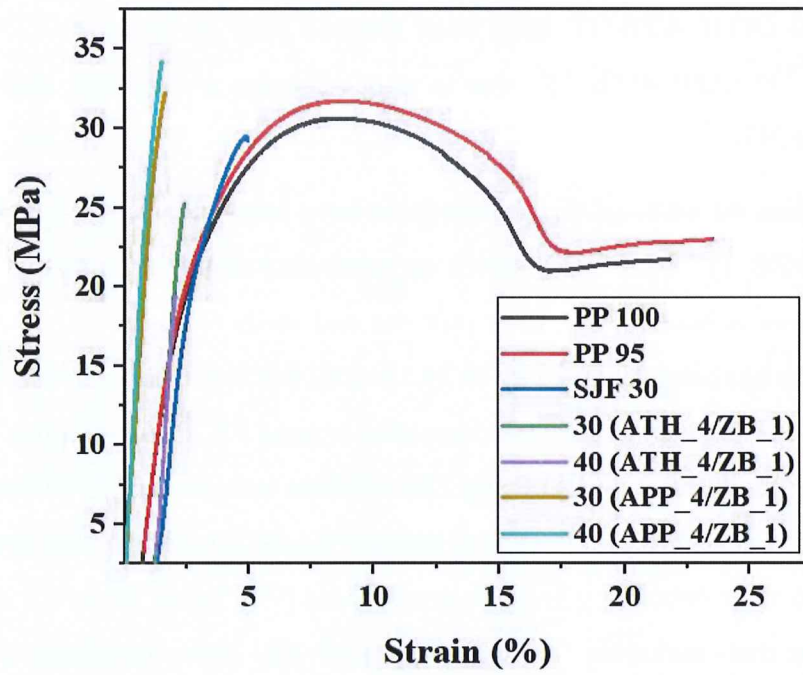


Figure 4.8: Effect of loading of flame retardants (FRs) with short jute fiber (SJF) in tensile strength.

Table 4.3: Tensile test results of PP 100, PP 95, SJF 30, 30 (ATH_4/ZB_1), 40 (ATH_4/ZB_1), 30 (APP_4/ZB_1) and 40 (APP_4/ZB_1).

Sample Code	Ultimate Tensile Strength (MPa)	Strain (%)	Young Modulus (MPa)
PP 100	30.62±0.69	21.81±2.93	1251±60
PP 95	31.73±0.74	23.51±7.66	1257±56
SJF 30	29.50±2.74	4.97±0.84	1517±198
30 (ATH_4/ZB_1)	25.23±2.01	2.43±0.22	1588±233
40 (ATH_4/ZB_1)	19.42±1.02	3.66±0.67	1505±301
30 (APP_4/ZB_1)	32.24±2.37	1.67±0.1	3416±131
40 (APP_4/ZB_1)	34.18±2.02	1.55±0.36	4180±174

4.3 Flexural Test Results Analysis

4.3.1 Effect of coupling agent & reinforcer length

Figure 4.9 and Table 4.4 shows the resultant effect of MA-g-PP and jute fiber length (short jute fiber and long jute fiber) with PP. In the case of improving flexural strength, there have been a very little change in flexural strength when 5 wt.% of MA-g-PP is being added to PP. Inclusion of jute fiber with PP in the presence of 5 wt.% of MA-g-PP, flexural strength has been improved noticeably for both-long and short fiber form. Here, jute fiber has been proved as an excellent reinforcer with PP and MA-g-PP. Flexural strain reached up to the lowest 10.49% of mark for short jute fiber (SJF) due to the fracture [101] of PP.

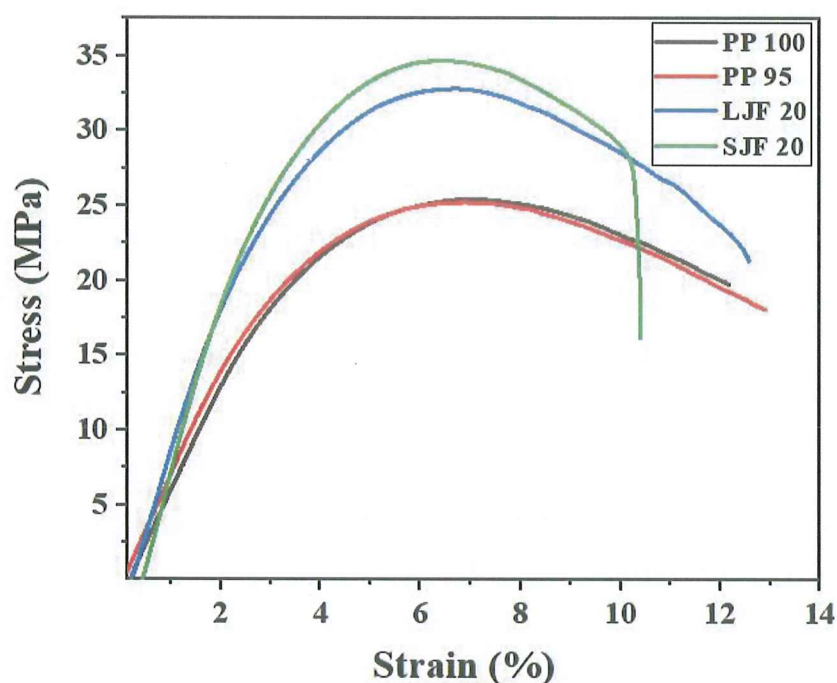


Figure 4.9: Effect of Coupling Agent & Reinforcer Form in Flexural Strength.

Flexural modulus has been reached from 706 ± 7 MPa to 1181 ± 95 MPa (clear average increase of 67.2%). Short jute fiber (SJF) has been improved property of stiffness of jute-pp composites which is directly linked to the concept of 'aspect ratio' of natural fibers [102]. More homogenous behavior has been noticed via SEM with short jute fiber (SJF) compared to long jute fiber (LJF) (Figure 4.1).

Table 4.4: Flexural test results of PP 100, PP 95, LJF 20 and SJF 20.

Sample Code	Flexural Strength (MPa)	Flexural Strain (%)	Flexural Modulus (MPa)
PP 100	25.46±0.37	12.17±0.15	706±7
PP 95	25.23±0.78	12.90±0.41	728±20
LJF 20	32.82±0.30	12.57±0.73	1066±39
SJF 20	34.68±1.70	10.49±1.26	1181±95

4.3.2 Effect of short jute fiber loading

Loading effect of short jute fiber on flexural strength is being represented in Figure 4.10 and Table 4.5. Inclusion of short jute fiber (SJF) tends to increase flexural strength gradually which proves the concept of ‘Effect of reinforcing’ [102]. However, high amount of reinforcing effect has been noticed up to 40 wt.% of loading of SJF (38.98±3.45 MPa). When fiber content has been increased up to 50 wt.% (SJF 50) flexural strength have been reached to 34.79±5.27 MPa and for 60 wt.% (SJF 60) flexural strength have been decreased to 37.77±1.23 MPa. From Table 4.5, it is evident that flexural strength value has been tended to decrease after 40 wt.% of loading of short jute fiber (SJF). After 40 wt.% loading of short jute fiber (SJF), fiber pull out, fiber misalignment, void and poor adhesion in between jute-pp composites have been observed noticeably via SEM (Figure 4.3). Necking behavior of jute-pp composites have been consistently increased with the increase of short jute fiber loading (Figure 4.10). Compared to PP 100, percentage decrease of strain value (average) is 64% when fiber loading limit have been reached to 60%. Moreover, flexural modulus has been reached up to 19.37±121 MPa (174.3% increase). This can be attributed towards good interfacial bonding.

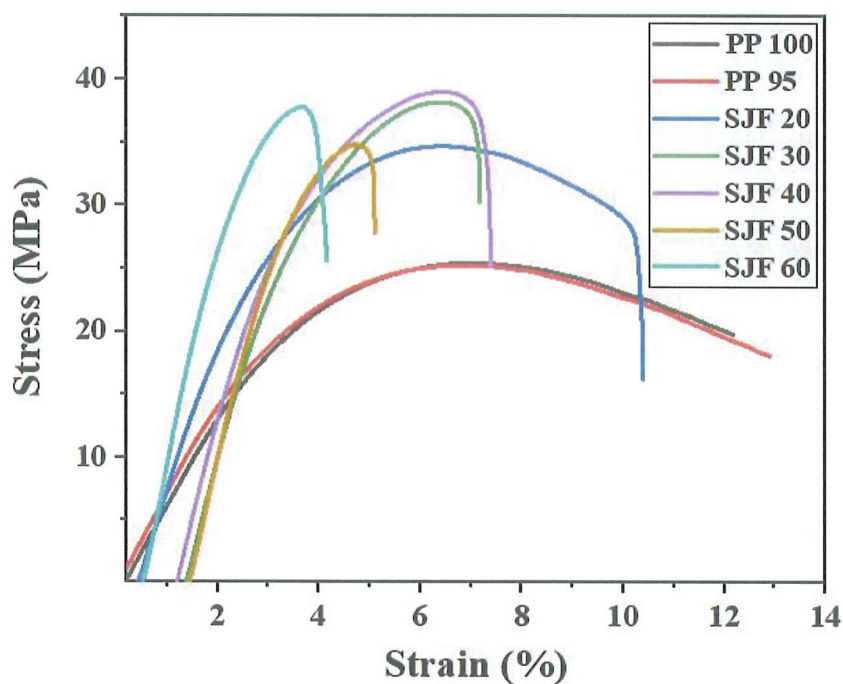


Figure 4.10: Effect of short jute fiber loading in flexural strength.

Table 4.5: Flexural test results of PP 100, PP 95, SJF 20, SJF 30, SJF 40, SJF 50 and SJF 60.

Sample Code	Flexural Strength (MPa)	Flexural Strain (%)	Flexural Modulus (MPa)
PP 100	25.46±0.37	12.17±0.15	706±7
PP 95	25.23±0.78	12.90±0.41	728±20
SJF 20	34.68±1.70	10.49±1.26	1181±95
SJF 30	38.17±1.9	7.18±1.17	1488±88
SJF 40	38.98±3.45	7.39±1.07	1489±66
SJF 50	34.79±5.27	5.10±0.91	1745±9
SJF 60	37.77±1.23	4.36±0.57	1937±121

4.3.3 Effect of Loading of Flame Retardants with Short Jute Fiber Loading

Figure 4.11 and Table 4.6 represents flexural strength results regarding the effect of loading of flame retardants with short jute fiber and PP. It is evident that- ATH/ZB or APP/ZB is exponentially good with short jute fiber and PP in the case of increasing flexural strength. Compared to PP 95, flexural strength increment range is in between 62.02%-174.7% for all formulations with flame-retardants.

On the other hand, compared to SJF 30 range is in between 7.09%-81.5% (average). The increase in flexural modulus have been attributed towards proper homogenization during mixing and compatible behavior of FRs with short jute fiber and PP. Effect of 40 wt.% loading of APP/ZB (''40 (APP_4/ZB_1)'') have been proven more evident (69.31 ± 6.30 MPa). However, a slight increase in flexural strength have been noticed for ''30 (APP_4/ZB_1)'' (49.70 ± 2.97 MPa) compared to SJF 30 (38.17 ± 1.9 MPa). This can be attributed to better homogenization, processing while manufacturing. In order to be sure, these measurements has been repeated and obtained this results. Unexpectedly, high values are attributed to the efficacy of sample preparation.

In terms of overall flexural properties, ''40 (APP_4/ZB_1)'' have been significant compared to ''40 (ATH_4/ZB_1)''. From Figure 4.4(b), uneven distribution of rigid flame retardants with short jute fiber and PP or cavities (voids) has been noticed within sample of ''40 (ATH_4/ZB_1)'' from thermal degradation [103]. Rigid particle sizes of flame retardants have been played a key role to increase flexural strength [41] of jute-PP composites.

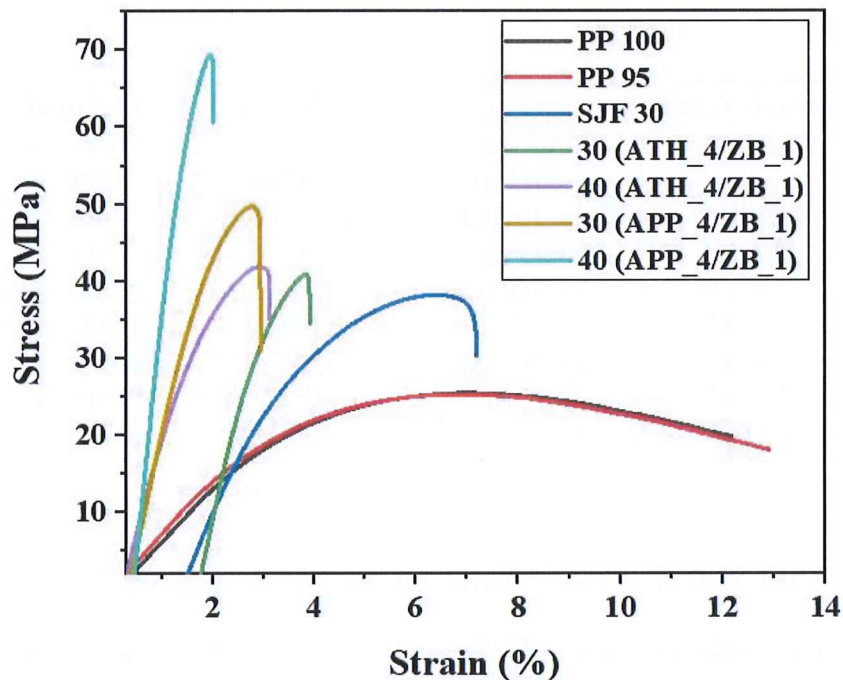


Figure 4.11: Effect of loading of flame retardants (FRs) with short jute fiber (SJF) in flexural strength.

Table 4.6: Flexural test results of PP 100, PP 95, SJF 30, 30 (ATH_4/ZB_1), 40 (ATH_4/ZB_1), 30 (APP_4/ZB_1) and 40 (APP_4/ZB_1).

Sample Code	Flexural Strength (MPa)	Flexural Strain (%)	Flexural Modulus (MPa)
PP 100	25.46±0.37	12.17±0.15	706±7
PP 95	25.23±0.78	12.90±0.41	728±20
SJF 30	38.17±1.9	7.18±1.17	1488±88
30 (ATH_4/ZB_1)	40.88±1.64	3.92±0.62	3108±227
40 (ATH_4/ZB_1)	41.82±5.72	3.12±0.48	2637±248
30 (APP_4/ZB_1)	49.70±2.97	2.94±0.37	3079±160
40 (APP_4/ZB_1)	69.31±6.30	2.01±0.19	6499±706

Flexural strain has been decreased noticeably for all. From Table 4.6, least amount of strain (%) up to 2.01±0.19 has been clearly observed for ‘‘40 (APP_4/ZB_1)’’. Compared to SJF 30, strain value of ‘‘40 (APP_4/ZB_1)’’ have been decreased to 72% (average) and compared to PP 95 and PP 100 the percentage amount of average strain value has been decreased up to around 83.48%-84.41%.

Increase of flexural stress and flexural modulus is directly related to fiber loading ratio. Addition to that, inclusion of high flame-retardant fillers with high amount jute fiber into PP increases flexural properties like ‘reinforcement of particles’ in elastic region. A clear displacement of PP is noticeable while adding more flame retardants [104]. Flexural modulus has been increased sharply for all formulations of flame retardants compared to PP 100, PP 95 and SJF 30 for these reasons. However, increasing the amount of (ATH_4/ZB_1) by 10 wt.% decreases flexural modulus immediately which shows behavior of particles of ATH is more vulnerable compared to APP along with ZB. Voids are visible (Figure 4.4(a)) with samples of ‘‘30 (ATH_4/ZB_1)’’.

4.4 Izod Impact Test Results Analysis

Izod impact tests have been carried out to investigate about the impact resistance of produced composites. Figure 4.12 and Figure 4.13 shows overall impact properties of jute-pp and flame-retardant based jute-pp composites.

PP 95 and PP 100 exhibit highest energy absorbance compared to all samples. Length of fibers (LJF 20 and SJF 20) have not affected on impact properties (Figure 4.12). Capacity of absorbing energy gradually decreases with high fiber loading into polymer matrix [105] and it is identically noticed from Figure 4.12. When amount of SJF is in between 30 wt.%- 60 wt.%, impact properties are nearly close to each other. However, a sudden decrease has been noticed for SJF 50 due to poor homogenization while manufacturing. Inclusion of high loading of flame retardants with polymer based natural fiber composites decreases impact properties [106] due to a creation of discontinuities within polymer matrix. Stress concentration of flame retardant composites gets disrupted because each flame-retardant fillers become an initiator of unwanted cracks with micro phases [105,107]. From Figure 4.13, it can be observed that- jute-pp composites based on ATH/ZB flame retardants are more capable of absorbing energy compared to APP/ZB flame retardants. It is evident to see that- values of impact strength have been sharply decreased above 95% for all formulations regarding flame retardants (Figure 4.13) which supports above explained theory of ‘discontinuity of polymer matrix’ and ‘crack initiation’.

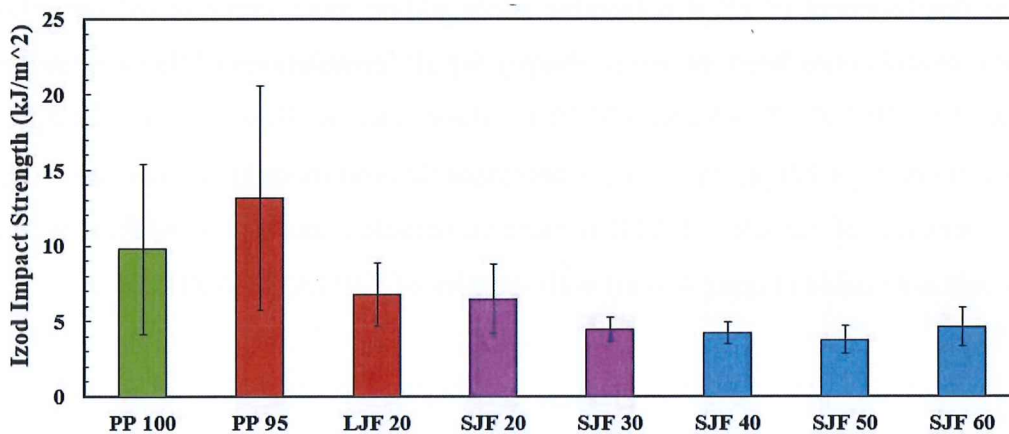


Figure 4.12: Izod impact strength of jute/PP composites without any flame retardants at different loadings level of jute fibers.

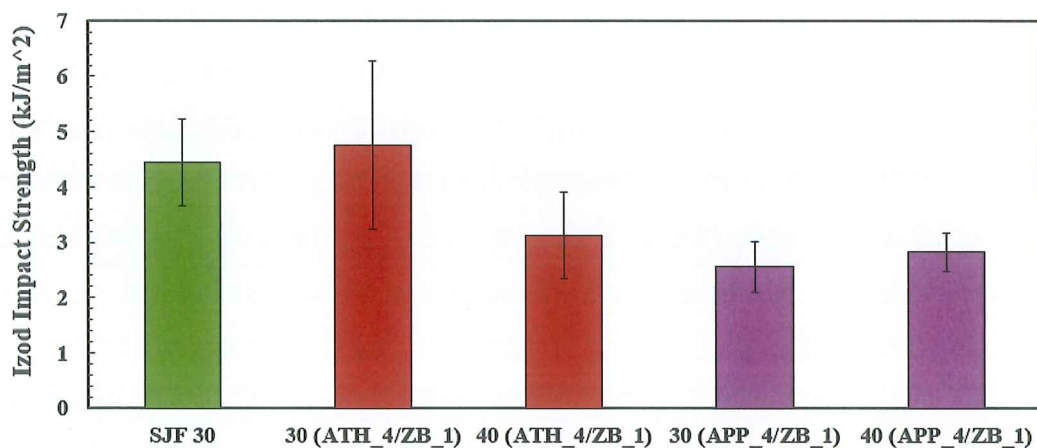


Figure 4.13: Izod impact strength of jute/PP composites with flame retardants (ATH/ZB and APP/ZB) at different loadings level of flame retardants with 30 wt.% of short jute fiber (SJF).

4.5 Density Test Results Analysis

Figure 4.14 represents, density of jute/PP composites with and without flame retardants. Effect of fiber length is critical in terms of changing density properties. From Figure 4.14, it is evident that LJF 20 is more beneficial compared to SJF 20 in terms of making lightweight jute-pp composites. However, other mechanical properties like tensile strength and flexural strength are key factors. As we have noticed from these results, SJF 20 is more convenient choice. Increasing natural fiber content increases density of produced composites [108]. Up to 30 wt.% of jute fibers increment have been noticed up to 0.943 g/cm³. A clear increase of 4.27% compared to PP 100. However, densities have been decreased linearly for SJF 40 and SJF 60. The possible reason is attributed towards voids and pores due to creation of ‘bubbles of air’ [109,110] within produced jute/PP composites. In addition to that, large amount of decrease (3.72%) has been noticed for SJF 50 due to creation of excessive voids and lack of proper homogenization during mixing phase.

On the other side, inclusion of flame-retardant fillers at different ratios (30 wt.% and 40 wt.%) with constant weight fractions (30 wt.%) of short jute fibers have been increased densities of produced flame retardant jute/PP composites. Density of reinforcement fillers is largely responsible to increase the density of polymer based natural fiber composites.

Within the scope of this thesis, density of applied ATH (2.42 g/cm^3) is greater than applied jute fiber (1.5 g/cm^3), PP (0.9 g/cm^3) and MA-g-PP (0.91 g/cm^3). Hence, density of flame-retardant jute/PP composites has been increased noticeably compared to PP 100 and PP 95 when wt.% of flame retardants have been increased from the level of 30 wt.% to 40 wt.%. From Figure 4.14, it has been clearly seen that-“40(ATH_4/ZB_1)” has been increased maximum up to 1.185 g/cm^3 . A clear increase of 7.9% compared to “30 (ATH_4/ZB_1)”. An overall increase of 30.93% have been achieved compared to PP 100 for “40 (ATH_4/ZB_1)”. Compared to ‘(ATH_4/ZB_1)’, density is much lower for ‘(APP_4/ZB_1)’. Possible reason is attributed towards filler density itself. Densities of APP and ZB are such as: 1.61 g/cm^3 , 3.64 g/cm^3 . When formulation of “30 (APP_4/ZB_1)” have been applied instead of “30 (ATH_4/ZB_1)” to produce flame retardant jute-pp composites, value of density has been decreased noticeably up to 4.48%. Moreover, when formulation of “40 (APP_4/ZB_1)” have been applied instead of “40 (ATH_4/ZB_1)” value of density has been also decreased gracefully up to 4.41%. An overall increase of 25.4% have been achieved for “40 (APP_4/ZB_1)” which ensures that “40 (APP_4/ZB_1)” is more lightweight and flexible compared to “40 (ATH_4/ZB_1)”.

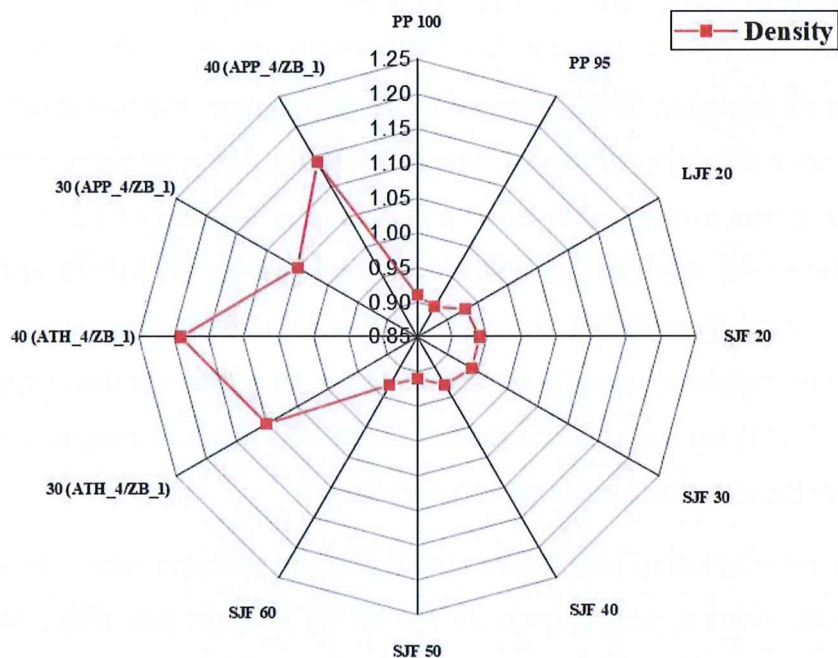


Figure 4.14: Density (g/cm^3) properties of jute/PP composites with and without flame retardants at different level of loadings.

4.6 Wear Test Results Analysis

SJF 30 contains 30 wt.% of short jute fiber along with 65 wt.% PP and 5 wt.% MA-g-PP. Wear rate is directly related to high amount of cellulose based natural fibers [111]. Since, SJF 30 is having an optimum level of jute fibers along with PP matrix, wear rate decreases (Figure 4.15). It is a clear indication of increasing wear resistance because of showing an optimum amount of stress transfer and distribution [111] along with PP matrix and short jute fibers. When high amount of flame retardant fillers (30 wt.% and 40 wt.%) get attached to PP matrix with a constant amount (30 wt.%) of short jute fibers, there is a high chance to create 'agglomeration' and 'fiber debonding' due to poor interaction of fibers [111], fillers and PP altogether. Interfacial adhesion decreases [111] such that within the scope of this thesis- '30 (ATH_4/ZB_1)', '40 (ATH_4/ZB_1)', '30 (APP_4/ZB_1)', and '40 (APP_4/ZB_1)' have been shown poor resistance to wear causing higher wear rate [112]. From Figure 4.15, it has been clearly noticed that- compared to SJF 30, wear rate of '40 (ATH_4/ZB_1)' have been increased to 223.44%. On the other hand, rate of increase of wear have been reached up to 198.74% for '40 (APP_4/ZB_1)' compared to SJF 30. Lastly, wear rate of '40 (ATH_4/ZB_1)' is 7.6% higher than '40 (APP_4/ZB_1)'. This may be attributed towards a key factor like 'high pin temperature' [112] while sliding with stainless steel ball.

Figure 4.16 shows plot of 'COF vs Time(s)' for sample of SJF 30, '40 (ATH_4/ZB_1)' and '40 (APP_4/ZB_1)'. It is evident that- value of COF have been lower for SJF 30 compared to '40 (ATH_4/ZB_1)' and '40 (APP_4/ZB_1)'. The key reasons may be attributed to rolling action regarding sample debris [111]. Moreover, value of COF has been found highest for '40 (ATH_4/ZB_1)'. This happened due to creation of a smaller number of debris [111] which is directly related to high value of COF. A moderate value of COF has been achieved for '40 (APP_4/ZB_1)'.

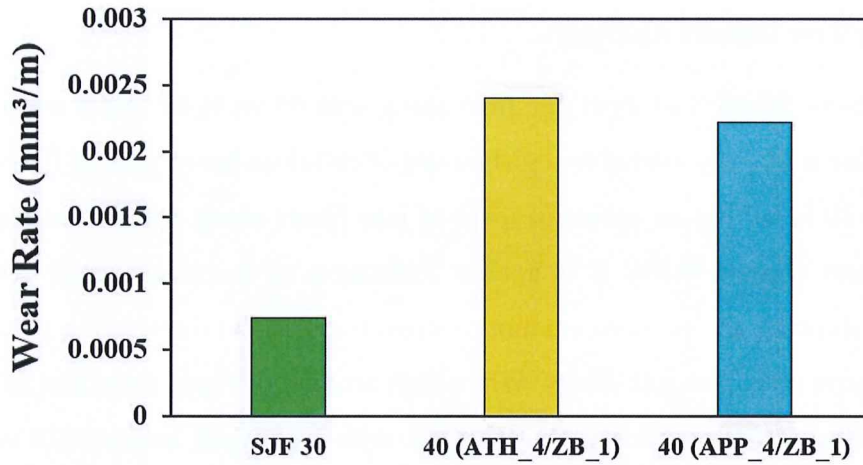


Figure 4.15: Wear rate (mm³/mm) of flame retardant jute/PP composites.

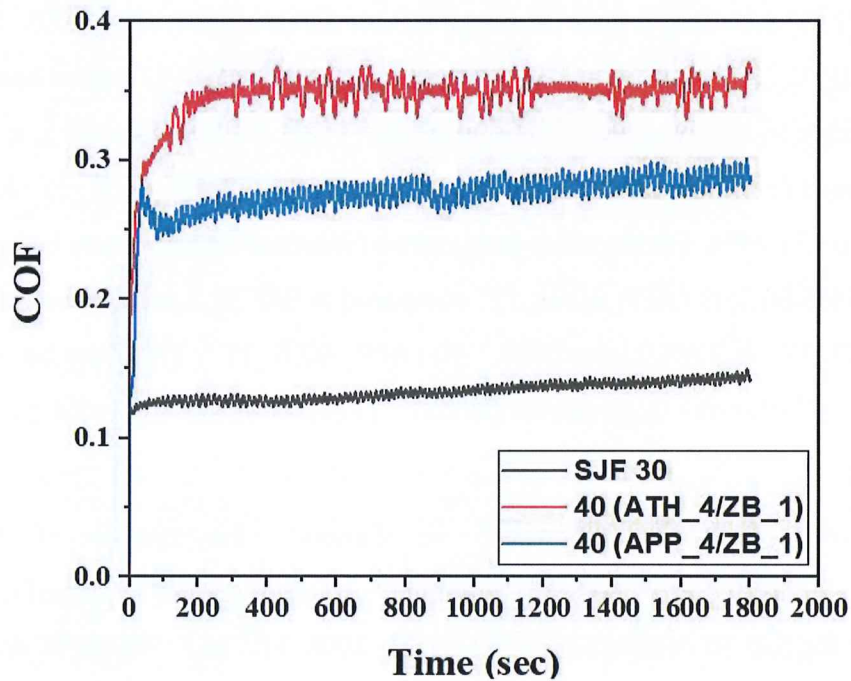


Figure 4.16: COF vs Time(s) graph of flame-retardant jute/PP composites.

4.7 Thermogravimetric (TGA) Results Analysis

The influence of temperature on the mass changes of Jute, Jute/ATH-Zinc borate, and Jute/APP-Zinc borate reinforced PP composites are being presented in Figure 4.17. As shown in Figure 4.17(a, c, and e), the PP 100 and PP 95 exhibited one step degradation characteristic, while they had a wavy form with jute additive. With the addition of maleic, the PP structure became more stable and the decomposition temperature was shifted by about 10 °C (Figure 4.17(b, d, and f)).

In addition, the maximum degradation rate temperature of composite structures containing 40 (ATH_4/ZB_1) and 40 (APP_4/ZB_1) was found to be 481.58 % and 481.62%, respectively, with an increase of approximately 16 °C compared to SJF 30 composite. The temperature causing 10% weight loss decreased with the addition of APP/ZB (Table 4.7). Furthermore, PP completely burned at 600 °C, while 31.67 % and 36.25 % ash remained in composite structures containing 40 (ATH_4/ZB_1) and 40 (APP_4/ZB_1), respectively.

Table 4.7: TGA data of PP/Jute/ATH/APP/ZB.

Samples	T _{10%} (°C) ^a	T _{max} (°C) ^b	Ash (%) at 600 (°C) ^a
100 PP	388.9	452.85	0
95 PP	429.2	462.15	0
30 SJF	331.6	465.65	4.83
30 (ATH_4/ZB_1)	300.5	481.51	23.91
40 (ATH_4/ZB_1)	295.5	481.58	31.67
30 (APP_4/ZB_1)	257.9	478.67	29.8
40 (APP_4/ZB_1)	314.5	481.62	36.25

^aTemperature at 10% weight loss.

^bThe maximum degradation rate temperature.

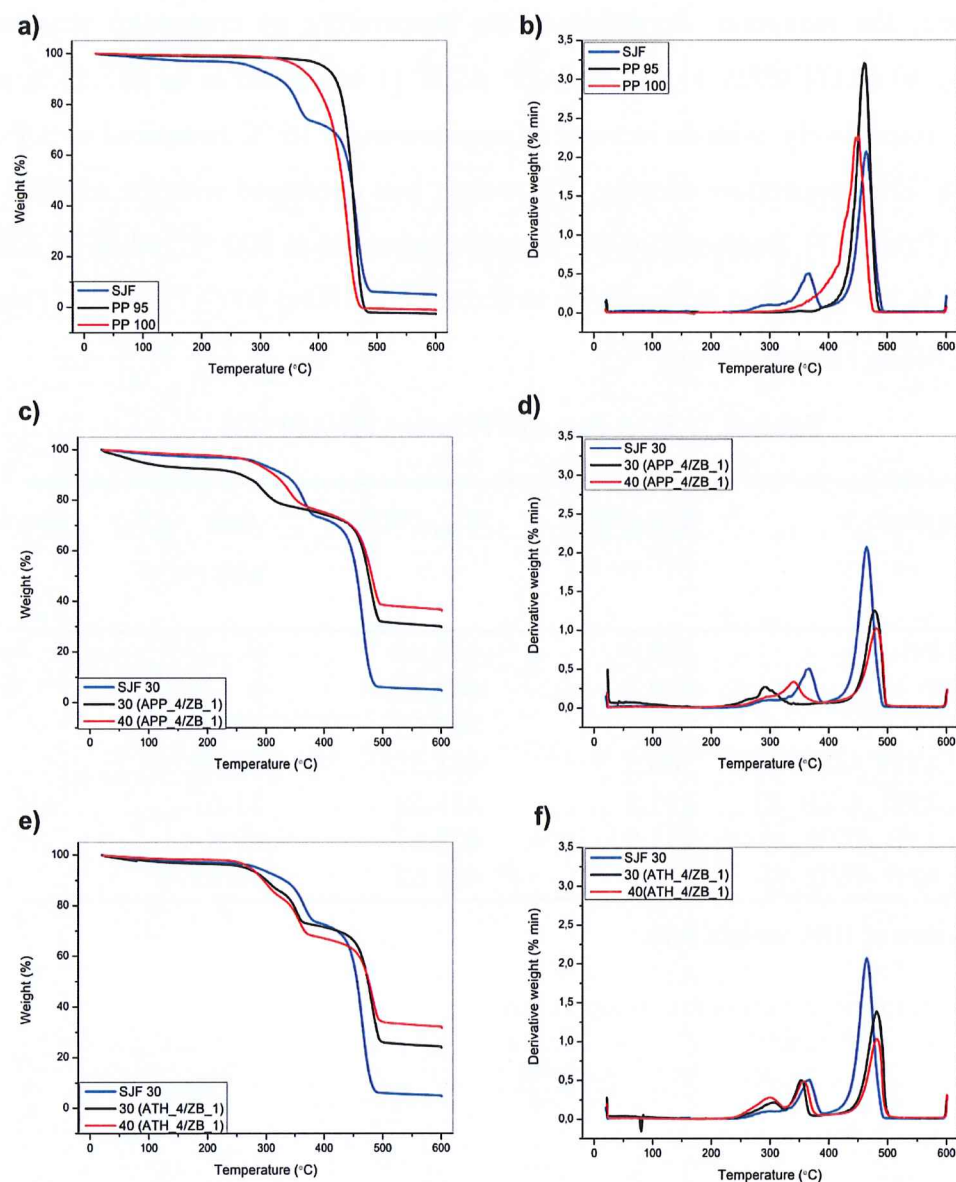


Figure 4.17: TGA and DTG curves of SJF 30 (a,b), PP 95 (a,b), PP 100 (a,b), 30 (APP_4/ZB_1) (c,d), 40 (APP_4/ZB_1) (c,d), 30 (ATH_4/ZB_1) (e,f) and 40 (ATH_4/ZB_1) (e,f).

4.8 Fourier-transform Infrared Spectroscopy (FTIR) Test Results Analysis

The reaction mechanism can be explained by the FTIR spectra of PP 100, PP 95, ATH_4/ZB_1, and APP_4/ZB_1, as shown in Figure 4.18. The multipieaks at 2747-3039 cm^{-1} can be attributed to C-H stretching vibration, and the peaks at 1454 cm^{-1} and 1374 cm^{-1} are generated by C-H bending vibration of PP backbone. In addiaton, PP 95 was showed new absorption peak at 1648 cm^{-1} that assign to symmetrical and asymmetrical streching of anhydride groups. The most characteristic properties in the FTIR spectrum of Jute fiber are the new peaks at 3765 cm^{-1} and 1731-2002 cm^{-1} attributing to vibration of O-H in cellulose, C=O stretching vibration of hemicellulose, respectively. From Figure 4.18, it has been clearly observed that all peaks regarding to formulatios including with '(ATH_4/ZB_1)' and '(APP_4/ZB_1)' have shown almost similarr peaks compared to SJF 30. Possible reason is attributed to extrusion process where ATH, APP and ZB have been behaved as only fillers without making any changes within chemical structures of newly produced flame retardant jute-pp composites.

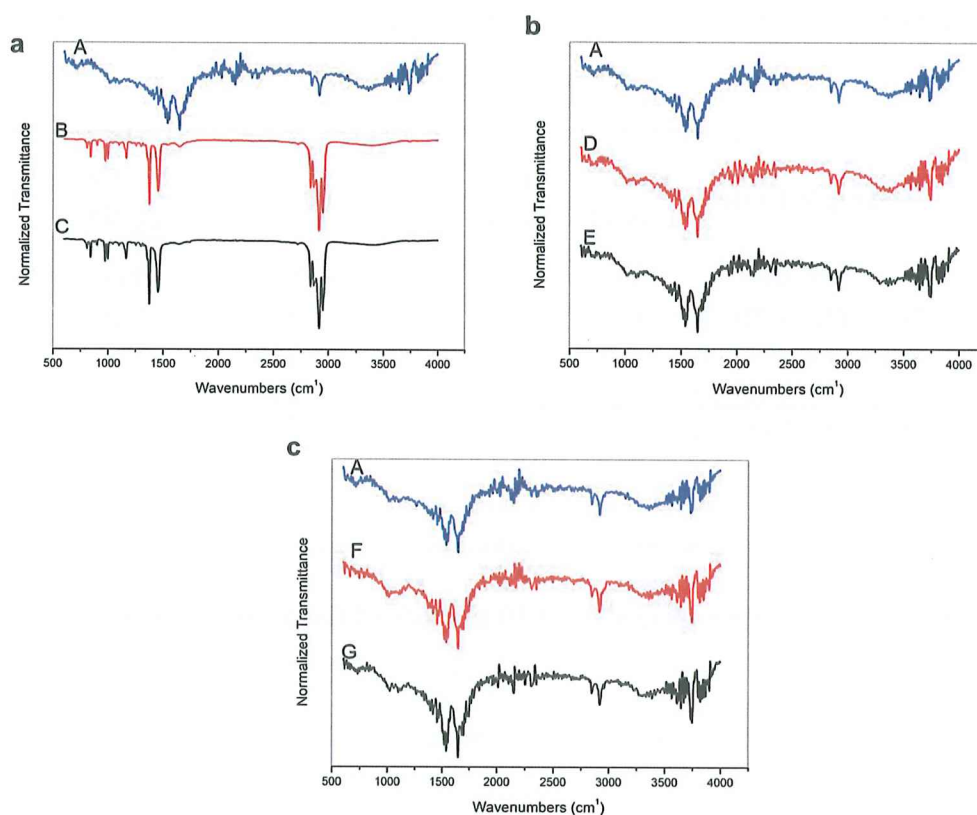


Figure 4.18: A; SJF 30, B; PP 95, C; PP 100, D; ATH 30, E; APP 30, F; 40 ATH, G; 40 APP.

4.9 UL-94 (Vertical Burning) Test Results Analysis

UL-94 (Vertical Burning) test have been carried out to evaluate the flammable behavior of flame-retardant reinforced jute/PP composites. Table 4.8, represents burning classification of vertically oriented test specimens including total combustion time, ignition condition while combustion and dripping behavior of flame-retardant reinforced jute/PP composites.

From Table 4.8, it has been clearly proved that- 'High flammability' is a key concern when 30 wt.% of jute fibers have been infused with polypropylene alone due to two reasons. One is 'high concentration of cellulose of jute' [87] and another one is 'presence of carbon and hydrogen within the chemical structure of PP which is responsible of high flammability of PP'. When ignition of PP occurs, high amount of carbon dioxide and water vapor is to be produced from instant flame propagation [113]. Without any addition of flame-retardant materials, jute/PP composites have been burned out completely (Figure 4.19-A2) after first flame application within 39.4 seconds (T_1^a).

Table 4.8: Details of UL-94 vertical burning testing results of multi-flame retardants reinforced jute-PP composites.

Sample Code	T_1^a (sec)	T_2^b (sec)	Total Time (sec)	Dripping	Cotton Ignition	Rating
SJF 30	39.4	-	39.4	Yes	Yes	NC
30 (ATH_4/ZB_1)	84.9	-	84.9	Yes	Yes	NC
40 (ATH_4/ZB_1)	3.3	153.3	156.6	Yes	Yes	NC
30 (APP_4/ZB_1)	5.5	121	126.5	Yes	Yes	NC
40 (APP_4/ZB_1)	1.3	1.5	2.8	No	No	V-0

^aSample burning time from first 10 seconds of flame application.

^bSample burning time from second 10 seconds of flame application.

Addition of “30 (ATH_4/ZB_1)” with jute/PP composite, an instant change of burning behavior have been noticed. 115.48% increase in total time (T_1^a increases up to 84.9 seconds) in time for “30 (ATH_4/ZB_1)” have been noticed compared to jute-pp composite (SJF 30). However, dripping and cotton ignition continues (Figure 4.19-B2). The key reason is percentage of incorporation of ATH is less. Amount of ATH should be around 60 wt.% to get optimum flame retardancy but it is expensive [114]. To reduce cost with effective performance of jute-pp composites ZB (a synergist) have been incorporated.

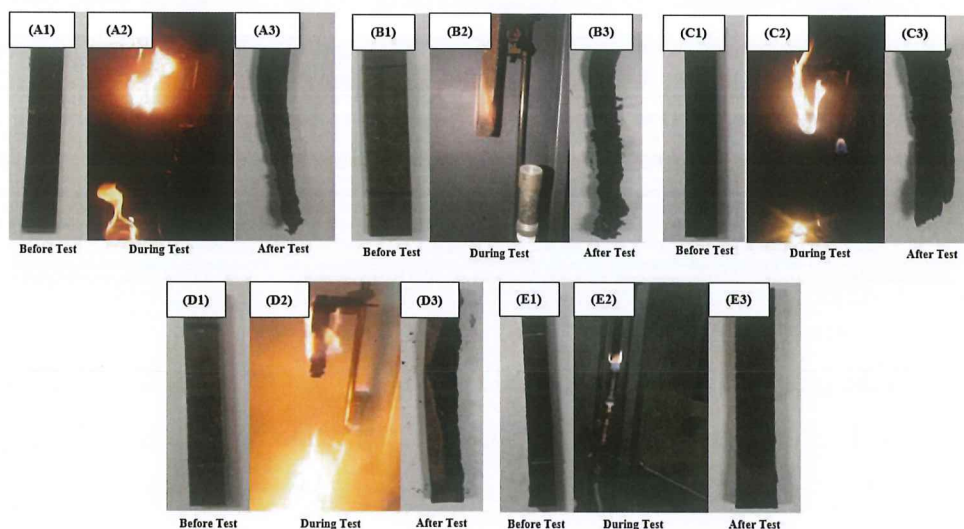


Figure 4.19: Digital photographs of SJF 30 (A1, A2, A3), 30 (ATH_4/ZB_1) (B1, B2, B3), 40 (ATH_4/ZB_1) (C1, C2, C3), 30 (APP_4/ZB_1) (D1, D2, D3) and 40 (APP_4/ZB_1) (E1, E2, E3) of UL-94 (vertical burning) test.

When concentration of ATH is 32 wt.% and ZB is 8 wt.% (“40 (ATH_4/ZB_1)”), total burning time ($T_1^a + T_2^b$) have been increased gradually up to 156.6 seconds (297% increase) compared to SJF 30 (39.4 seconds) where there have been no flame retardant additives involvement. But, dripping and cotton ignition continues here as well (Figure 4.19-C2). Prevention of oxygen penetration occurs [43] within jute/PP composite. Heat sinking behavior of ATH [43] is not so visible with the loading amount of 24 wt.% and 32 wt.%. Such that, slow burning continues even with prolonged sample burning time; dripping and cotton ignition is remarkably visible. Hence, it proves that-concentration level of ATH should be around 60 wt.% as mentioned above.

On the other side, when 30 wt.% flame retardants ('30 (APP_4/ZB_1)') have been incorporated with jute/PP composites total burning time have been increased up to 126.5 seconds within same condition as '30 (ATH_4/ZB_1)'. A complete increase of 221.06% in the performance of 'total burning time' have been noticed but dripping continues (Figure 4.19-D2). From the aspects of continuous dripping and cotton ignition, it can be said that 'concentration of APP' should be increased more (>30 wt.%) [2,48,115] along with ZB to get optimum V-0 level. No dripping and cotton ignition occurs (Figure 4.19-E2 & E3) when loading concentration of APP is 32 wt.% and ZB is 8 wt.%. Reduction of total combustion time (92.89%) have been noticed significantly. A complete V-0 level from UL-94 (Vertical Test) have been achieved. None of the testing samples have been passed V-0 rating except '40 (APP_4/ZB_1)'. Key reason is- To have intumescent behavior and charring effect of APP 'Concentration of APP' should be more than 30 wt.%. Formation of char is significant from component of APP and ZB due to water vapor release. Phosphorus compounds of APP and Boron compounds of ZB have been also noticeable [98] within jute-pp composites in the presence of 40 wt.% flame retardants '40 (APP_4/ZB_1)'. Both of these compounds are greatly responsible for char formation. However, ATH needs more filler loading within jute/PP composites to get optimum flame retardancy. Figure 4.19 and Figure 4.20 shows in depth scenarios and results of UL-94 (Vertical Burning) test.

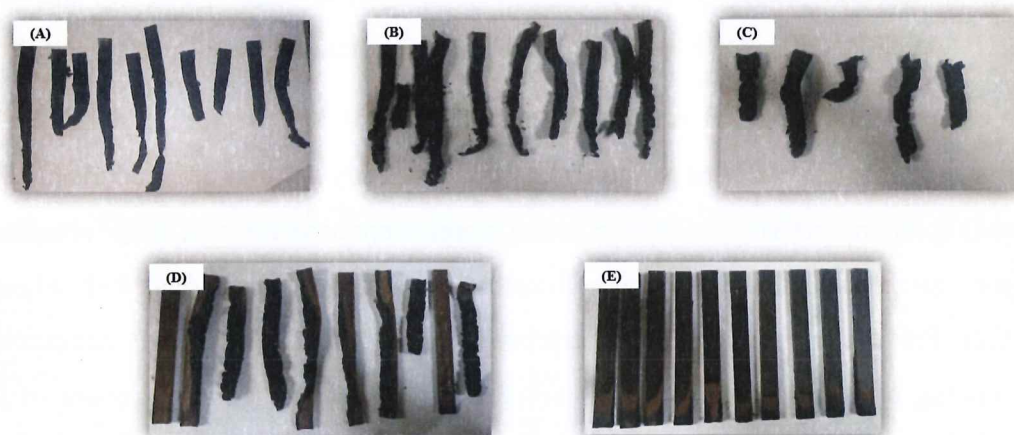


Figure 4.20: Digital photographs of after results of SJF 30 (A1, A2, A3), 30 (ATH_4/ZB_1) (B1, B2, B3), 40 (ATH_4/ZB_1) (C1, C2, C3), 30 (AAPP_4/ZB_1) (D1, D2, D3) and 40 (APP_4/ZB_1) (E1, E2, E3) regarding UL-94 (vertical burning) test.

4.10 UL-94 (Horizontal Burning) Test Results Analysis

UL-94 (Horizontal Burning) test have been carried out to classify material's flammability and to determine average linear burning rate (mm.min^{-1}) of selected test specimens regarding to its thickness accordingly [43,87,113,114].

Table 4.9 represents results of flame-retardant reinforced jute-pp composites based on UL-94 (Horizontal Burning) test where 'average linear burning rate (mm.min^{-1})' and 'flammability rating' have been summarized precisely. From Table 4.9, it is evident that- 'high fiber content of jute and its fast decomposition manner with heat source' [113] is greatly responsible for extreme level of average linear burning rate ($22.96 \text{ mm.min}^{-1}$). Figure 4.21 shows digital photographs of all samples during UL-94 (Horizontal Burning) test.

Table 4.9: Details of UL-94 horizontal burning testing results of multi-flame retardants reinforced jute/PP composites.

Sample Code	Average Linear Burning Rate (mm/min)	Percentage Change	Flame Reach 25 mm	Flame Reach 100 mm	Flammability Rating
SJF 30	22.96	-	Yes	Yes	HB
30 (ATH_4/ZB_1)	11.34	50.60 Decrease	Yes	Yes	HB
40 (ATH_4/ZB_1)	No Burning	-	No	No	HB
30 (APP_4/ZB_1)	7.2	68.64 Decrease	Yes	No	HB
40 (APP_4/ZB_1)	No Burning	-	No	No	HB

Addition of 24 wt.% of ATH with 6 wt.% of ZB drastically reduced the average linear burning rate ($11.34 \text{ mm.min}^{-1}$) of jute/PP composites. A clear decrease of more than 50% have been observed. Interestingly, 'No burning' have been noticed when 32 wt.% of ATH and 8 wt.% of ZB have been incorporated with jute-pp composites. Possible reason is attributed towards testing method. Flame propagation is different in between horizontal and vertical burning test methods. Propagated flame can be an integral part to achieve a feasible result on flame retardancy. Moreover, to get self-extinguishing fire behavior 60 wt.% of ATH alone is needed which is not so feasible economically [115] for jute/PP composites.

On the other side, addition of 24 wt.% of APP along with 6 wt.% of ZB shows 68.64% decrease in average linear burning rate (7.2 mm.min^{-1}). However, a significant level of melt dripping is noticeable for all of the three samples but have not reached 100 mm mark which signifies that more of APP concentration is to be needed to infuse while compounding.

An optimum way to have self-extinguishing fire properties is to incorporate more than 30 wt.% of APP with jute/PP composite while extrusion. Otherwise, HB classification won't be significant along with flammability rating of V-0 [2]. By incorporation of 32 wt.% of APP with 8 wt.% of ZB, it is inevitable to say that- 'Average linear burning rate is phenomenal'. Prime reason is 'High quality char layer formation' which is greatly influenced by APP and ZB side by side [48].

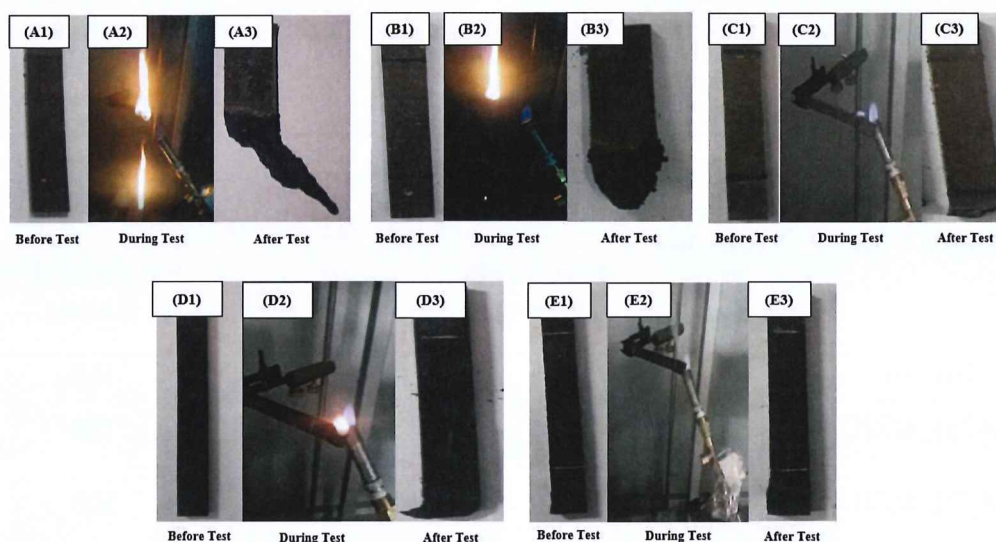


Figure 4.21: Digital photographs of SJF 30 (A1, A2, A3), 30 (ATH_4/ZB_1) (B1, B2, B3), 40 (ATH_4/ZB_1) (C1, C2, C3), 30 (APP_4/ZB_1) (D1, D2, D3) and 40 (APP_4/ZB_1) (E1, E2, E3) during UL-94 (horizontal burning) test.

Penetration of heat and oxygen have been blocked by these two prominent type halogen free flame retardants during combustion period [2]. They are responsible to generate a good thermal barrier [114] towards jute/PP composites. Such that, propagation of flame has not been even crossed initial mark of 25 mm during ignition.

4.11 Limiting Oxygen Index (LOI) Test Results Analysis

The limiting oxygen index (LOI) value is used to determine self-extinguishment behavior and flame-retardant properties of polymer materials where minimum oxygen concentration (vol %) level is being mainly focused [113]. The higher the LOI value, the lesser the chance to burn. As we know, air is having 21 vol% oxygen which refers to 'Any material with the value of LOI, less than 21% signifies as 'fully combustible/burnable'' [114]. From Table 4.12, the LOI value of jute/PP composites is only 16.4% which is below 21%, therefore, jute-pp composites are fully flammable without any modification with flame-retardant (FR) materials. Some key reasons of extreme low LOI value of jute/PP composites are as follows:

- ✓ Extrusion temperature is critical. Because, it is directly co-related with thermal stability of jute fiber. The thermal stability is largely depending on decomposition temperature of cellulose (350⁰ C), hemicellulose (200⁰ C) and lignin (200 ⁰C-600 ⁰C) of jute fibers [115].
- ✓ Amount of 'Cellulose' within jute fiber is higher (59-71.5 wt.%) [2]. It is greatly responsible for creating volatile flammables named as 'Levoglucosan' [43].
- ✓ Presence of 'aromatic rings' of 'Lignin' that creates an extreme 'resistance of oxidation' [43].
- ✓ Since jute fiber is having less amount of lignin (13.6-18 wt.%) compared to cellulose (59-71.5 wt.%) and hemicellulose (13.6-20.4 wt.%) [2] there is an extreme chance to have more thermal degradation at low temperature due to degradation of aromatic and aliphatic C-C bonds leads towards depolymerization. However, it creates char formation within jute/PP natural fiber polymer composites [43].

To increase flame retardancy of jute/PP composites within an economic perspective, combination of APP and ZB is more beneficial compared to the combination of ATH and ZB. But it is possible to reduce burning of jute/PP composite materials by incorporating ZB with ATH. However, slow burning continues with both jute/PP composites even with 30 wt.% and 40 wt.% loading of ''30 (ATH_4/ZB_1)''.

Table 4.10: Limiting oxygen index (LOI) data of flame retardant based jute-PP composites.

Sample Code	LOI (%)	% Increase	Classification [1]
SJF 30	16.4	-	Flammable (LOI<20.95)
30 (ATH_4/ZB_1)	21.9	33.54	Slow Burning (21<LOI<28)
40 (ATH_4/ZB_1)	26	58.54	Slow Burning (21<LOI<28)
30 (APP_4/ZB_1)	25	52.44	Slow Burning (21<LOI<28)
40 (APP_4/ZB_1)	28.8	75.61	Self-Extinguishing (28<LOI<100)

But LOI value have been increased to 21.9% (percentage of increase 33.54%) with 30 wt.% loading “30 (ATH_4/ZB_1)” of flame retardants compared to without any loading of flame retardants. When loading level of flame retardants “40 (ATH_4/ZB_1)” have been increased LOI value have been remarkably increased up to 26% (percentage of increase 58.53%) but slow burning continues even with high amount of LOI value. The key reason is attributed towards slow dilution process of combustible gases which causes release of temperatures [48].

To bring jute/PP composites Flame retardancy of PP can be improved by using intumescent flame-retardant like APP along with ZB [98]. When APP and ZB have been used with jute-PP composites at loading level of 30 wt.% “30 (APP_4/ZB_1)” and 40 wt.% “40 (ATH_4/ZB_1)” a noticeable synergetic effect can be noticeable. However, slow burning continues even with higher LOI value 25% compared to “30 (ATH_4/ZB_1)” for the formulation of “30 (APP_4/ZB_1)”. Self-extinguished jute-pp composites have been noticed with 40 wt.% loading of flame retardants “40 (APP_4/ZB_1)”. LOI value reached up to 28.8%. A clear increase of percentage of 75.60% compared to neat jute-PP composite. Reasons are as follows:

- ✓ APP works as an intumescent filler to create thermal barrier [43].
- ✓ APP can create a char formation that helps to close the immediate expansion of heat and mass transfer of jute/PP composites. But smoke generation is a drawback [44].

- ✓ Decomposition temperature of APP ($\approx 250^{\circ}\text{C}$) [45] and ZB ($\approx 290^{\circ}\text{C}$) are different. Decomposition hardly occurs at elevated temperatures when APP and ZB flame retardants are together [47] while extrusion.
- ✓ Compared to ATH, APP is more effective flame retardant to make self-extinguished jute-PP composites with low level of loading along with ZB.

Figure 4.22 shows experimentation of limiting oxygen index (LOI) test.

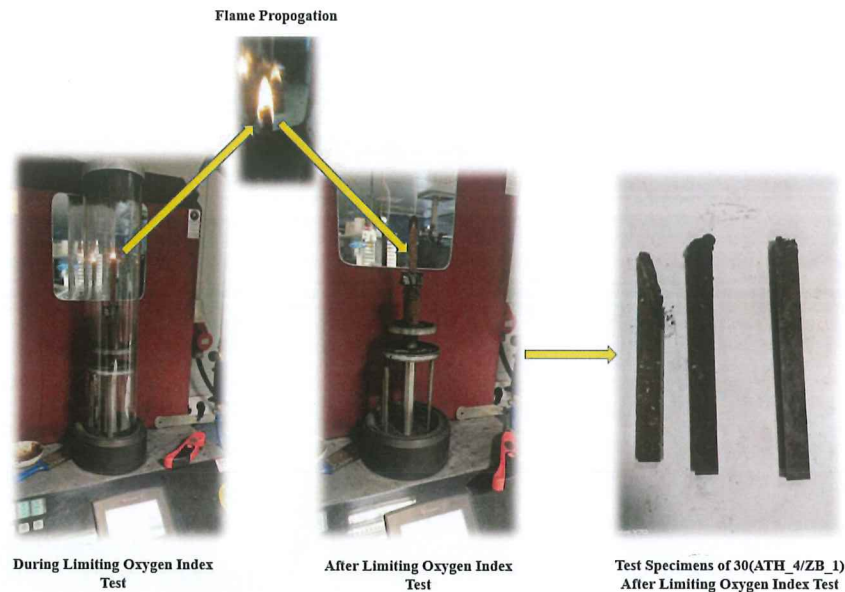


Figure 4.22: Limiting oxygen index (LOI) experimentation of flame retardant jute/PP composites.

4.12 Cone Calorimetry Test Results Analysis

To evaluate the fire performance of jute/PP composites based on flame retardants, the sample “(40 (APP_4/ZB_1))” have been used to characterize based on several parameters obtained from cone calorimetry test. Table 4.11 represents thermal properties of “(40 (APP_4/ZB_1))”, Table 4.12 represents smoke properties of “(40 (APP_4/ZB_1))” and Figure 4.23 represents extensive data over time regarding heat release rate (HRR), total heat release (THR), total smoke production (TSP) and smoke production rate (SPR) of “(40 (APP_4/ZB_1))”. From Figure 4.23(a), time to ignition (TTI) have been observed at 43s (first peak of HRR curve). After 240s, HRR curve has been started to decrease at a constant rate. The time to flame out (TFO) has been observed at 1121s precisely.

Peak heat release rate (pHRR) has been achieved up to 195.37 kW.m^{-2} . With an average heat release rate (Av-HRR) of 80.09 kW.m^{-2} , total heat release (THR) has been reached up to 86.4 MJ.m^{-2} (Table 4.11). From Figure 4.23(b), it is very clear to say that- value of THR increases by time. Until 300s, THR value reached up to 45.11 MJ.m^{-2} from initial stage. When duration has been prolonged up to 1200s, THR value reached up to 87.61 MJ.m^{-2} .

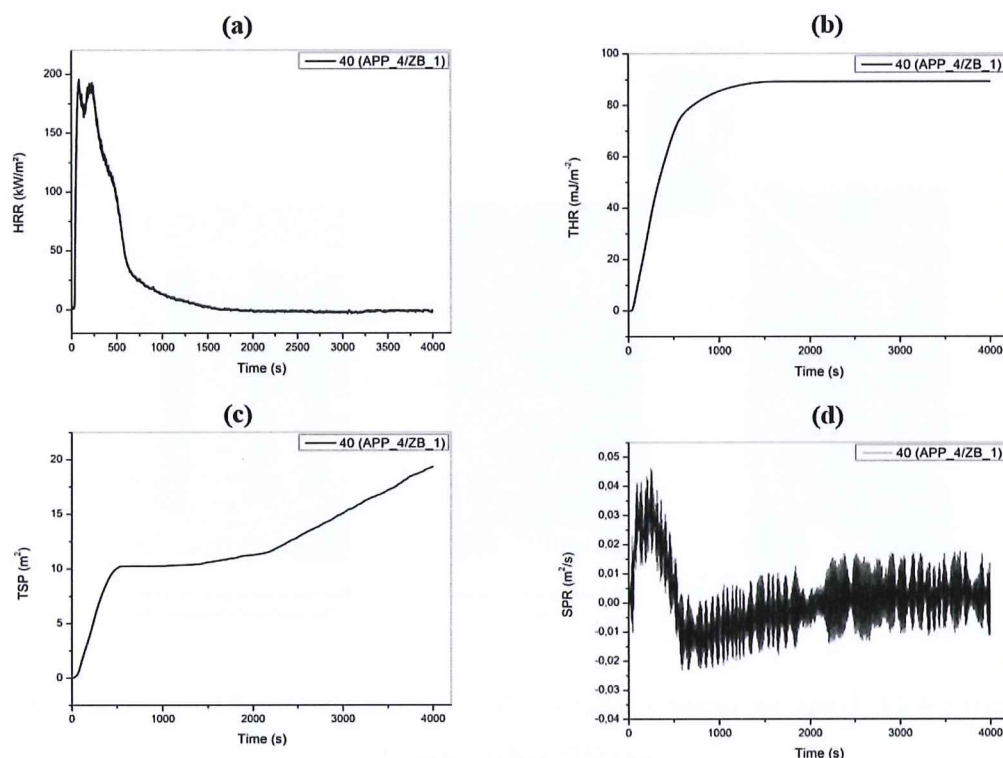


Figure 4.23: The curves of a) heat release rate (HRR) b) total heat release (THR), c) total smoke production (TSP) and d) smoke production rate (SPR) of 40(APP_4/ZB_1) according to cone calorimetry test.

From Table 4.14 and Table 4.15, it can be clearly observed that- application of jute fiber to reduce peak heat release rate have been significantly fruitful. Key reason is a formation of a carbonaceous char layer by the presence of lignin and cellulose of jute fiber. Propagation of fires get prolonged by a dilution of heat conduction. In addition to that char layer of APP have shown heat and smoke insulation impact over jute-PP composite by acting as a thermal protective shield. Such that peak heat release rate has been very significantly less compared to FRs-based literatures based on PP and jute-PP. Total smoke production (TSP) have been observed up to 10.1 m^2 with an average smoke production rat (Av-SPR) of $0.0038 \text{ m}^2.\text{s}^{-1}$.

Specific extinction area (SEA) has been observed up to $125.48 \text{ m}^2.\text{kg}^{-1}$ which signifies towards the reduction of fuel gas density while having combustion. Moreover, declining of CO and CO₂ minimize the possibility to have poisoning and suffocation while fire ignition occurs. In this way, flame retardancy can be improved remarkably.

Table 4.11: Thermal properties of “40(APP_4/ZB_1)” according to cone-calorimetry test.

TTI (s)	PHRR (kW.m ⁻²)	T _{PHRR} (s)	THR (MJ.m ⁻²)	A _{THR} (KW.m ⁻²)	Av-EHC (MJ.kg ⁻¹)	T _{EHC} (s)
43	195.37	87	86.42	80.09	23.47	44

Table 4.12: Smoke properties of “40(APP_4/ZB_1)” according to cone-calorimetry test.

TSR (m ² .m ⁻²)	TSP (m ²)	Av- SPR (m ² .s ⁻¹)	Av-SEA (m ² .kg ⁻¹)	Av-CO yields (kg.kg ⁻¹)	Av-CO ₂ yields (kg.kg ⁻¹)	CO/CO ₂ ratio
1145.17	10.1	0.0038	125.48	0.1037	1.68	0.0617

Table 4.13: Other properties of “40 (APP_4/ZB_1)” according to cone-calorimetry test.

Surface Area (cm ²)	MARHE (kW.m ⁻²)	Mass (initial) (g)	Mass (final) (g)	Av- MLR (g.s ⁻¹ .m ⁻²)	Mass Lost (g.m ⁻²)	Total Oxygen Consumed (g)	Fuel load (MJ.kg ⁻¹)
88.4	150.4	50.1	17.4	3.41	3682.5	56.5	15.25

Table 4.14: Comparison list of “40 (APP_4/ZB_1)” with FRs based PP literatures.

Composition	Wt. %	TTI (s)	pHRR (kW.m ⁻²)	THR (kW.m ⁻²)	LOI (%)	UL-94	Ref.
Ammonium Polyphosphate (APP)	35	11	435.9	83.9	20.4	NR	[116]
Ammonium Polyphosphate (APP)	40	30	440	186	20.8	NR	[117]
Pentaerythritol triacrylate microencapsulated APP (mc-APP)	40	32	214	183	30.5	V-0	[117]
Pentaerythritol phosphate (PEPA)	40	35	776	81	-	-	[118]
Melamine phosphate (MP)	40	39	296	78	-	-	[118]
Aluminum trihydroxide (ATH)	40	28	467	70	-	-	[119]
Magnesium dihydroxide (MDH)	40	46	548	99.1	23.3	NR	[120]
APP/ZB	40	43	195.37	86.42	28.8	V-0	Current Study

Table 15: Comparison list of “40 (APP_4/ZB_1)” with FRs based jute-PP literatures.

Flame Retardant Composite	Composition (wt.%)	LOI (%)	Horizontal Burning (mm/min)	PHRR (Peak Heat Release Rate (kW/m ²))	Time to Ignition (s)	Total Smoke Production (m ²)	Effective Heat Combustion (MJ/kg)	Total Heat Release (MJ/m ²)	Burning Level (UL-94)	Ref.
Ammonium polyphosphate/Expandable graphite/Jute/PP	2/3/75/20	30.6	19.5 (s) (HB)	240	21	3.2	27.15	39.8	-	[58]
B-cyclodextrin/Ammonium polyphosphate/Melamine/Jute/PP	5/10/5/40/40	27.8	37 (s) (HB)	312	18	6.5	25.1	43.3	-	[59]
Diammonium phosphates/Jute/PP	5/60/35	-	19 (HB)	-	-	-	-	-	-	[60]
B-cyclodextrin/Ammonium polyphosphate/Jute/PP	6.67/13.33/40/40	-	-	167 (Average)	-	6.6	-	-	-	[61]
Jute fiber/PP fiber/Ammonium polyphosphate/PP Powder	35/35/10/20	25.7	10.09 (HB)	-	-	-	-	-	-	[62]
Bisphenol A epoxy acrylate (BAEA)/2-hydroxyethyl methacrylic acid phosphate (PM-2)-polyurethane acrylate (PUA)/Jute/PP	BAEA 50 (mass%) PM-2:50 (mass%)	26.1	8.54 (HB)	341.65	7	-	9.2	58.34	-	[63]
Jute/PP/PVC/Aluminum trihydrate (ATH) / Zinc Borate with Antimony trioxide (ZB-AT)	50/16.5/13.5/10/7.5/2.5	-	220 (HB)	-	-	-	-	-	-	[64]
Jute/PP	42/58	18.7	27(HB)	675	33	1121	31.7	95.5	121.2 minute (VB)	[65]
Jute/MA-g-PP/PP/APP/ZB	30/5/25/32/8	28.8	No Burning	195.37	43	10.1	23.47	86.42	V-0	Current Study

5. CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

- ✓ Compared to LJF, SJF have been showed better mechanical properties in terms of tensile strength (Tensile strength: 37.32 ± 0.75 MPa, Young modulus: 1811 ± 81 MPa) and strain: 5.50 ± 0.14 %) and flexural strength (Flexural strength: 34.68 ± 1.70 MPa, Flexural modulus: 1181 ± 95 MPa and strain: 10.49 ± 1.26 %) at 20 wt.% of jute fiber loading.
- ✓ Up to 60 wt.% of loading of SJF tensile strength have been noticeably decreased to 19.50 ± 2.04 MPa. However flexural strength value has been nearly similar (37.77 ± 1.23 MPa) until 40 wt.% of loading of SJF (38.98 ± 3.45 MPa).
- ✓ A 30 wt.% of loading of short jute fiber have been consistent for both tensile and flexural properties.
- ✓ Inclusion of 40 wt.% of flame retardant fillers of APP and ZB at a ratio of 80:20 have been achieved performed excellent compared to others. Tensile strength value of 34.18 ± 2.02 MPa, tensile modulus value of 4180 ± 174 MPa and strain value of 1.52 ± 0.36 % have been observed for “40 (APP_4/ZB_1)”. On the other hand, flexural properties have been also remarkable (Flexural strength: 69.31 ± 6.30 MPa, strain: 2.01 ± 0.19 % and flexural modulus: 6499 ± 706 MPa).
- ✓ Izod impact strength value has been decreased, density and wear rate have been increased noticeably by inclusion of high loading (40 wt.%) of flame retardants.
- ✓ ATH/ZB formulations have not been significant in terms of achieving V-0 level flame retardancy however a ‘no burning effect’ have been noticed during horizontal burning test which signifies that- testing conditions, flame ignition time might change the burning behavior of composites.

- ✓ APP/ZB formulation (“40 (APP_4/ZB_1)”) have been significant in terms of achieving V-0 level flame retardancy. This sample have passed both version of UL-94 tests criteria.
- ✓ A maximum limit oxygen index value of 28.8% have been achieved for “40 (APP_4/ZB_1)” which lies under the classification of self-extinguishing.
- ✓ From the cone calorimetry test result of the sample of “40 (APP_4/ZB_1)”, peak heat release rate has been achieved up to 197.5 kW/m² with total smoke production value of 10.5 m². Total heat release has been observed up to 86.42 kW/m². Effective hear of combustion value have been achieved up to 23.47 MJ/kg.

5.2 Recommendations

- To improve mechanical performance of jute- composites, surface treatment of jute fibers can be implemented via alkali or silane treatment.
- To improve flame retardancy synthesise of APP or ZB particles can be implemented which can reduce the peak heat release rate and total smoke production and increase LOI value by creating a char layer towards jute-pp composite surface.
- Nanomaterials as a filler can be infused to current formulation at a very low level such that heat release rate significantly can be reduced and it might decrease the wt.% of FR filler. In this way, infusion of jute fiber or PP matrix can be increased.

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