**SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING**

**UNIVERSITI SAINS MALAYSIA**

# FABRICATION AND CHARACTERIZATION OF ELECTROSPINNING BIODEGRADABLE FABRIC LAYER FOR FACE MASK APPLICATION

By

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## DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled 'Fabrication and Characterization of Electrospinning Biodegradable Fabric Layer for Face Mask Application'. I also declare that it has not been previously submitted for the award of any degree and diploma or other similar title of this for any other examining body or University.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ASTM | American Standard Testing Measurement |
| Ave | Average |
| BFE | Bacterial Filtration Efficiency |
| COVID | Coronavirus Disease |
| DCM | Dichloromethane |
| DMF | N, N-Dimethylformamide |
| FDA | Food And Drug Administration |
| MP | Microplastic |
| PAN | Polyacrylonitrile |
| PBAT | Polybutylene adipate-co-terephthalate |
| PC | Polycarbonate |
| PCL | Polycaprolactone |
| PE | Polyethylene |
| PEI | Polyethylenimine |
| PES | Polyester |
| PHB | Polyhydroxybutyrate |
| PLA | Polylactic Acid |
| PM | Particulate Matter |
| PP | Polypropylene |
| PPE | Personal Protective Equipment |
| PS | Polystyrene |
| PU | Polyurethane |
| PVA | Poly Vinyl Alcohol |

| | |
|---|---|
| SARS | Severe Acute Respiratory Syndrome |
| SEM | Scanning Electron Microscope |
| St dev | Standard Deviation |
| USD | United States Dollar |
| UTM | Universal Testing Machine |
| WHO | World Health Organizations |

## LIST OF SYMBOLS

| | |
|---|---|
| mL/h | Milli Litre Per Hour |
| cm | Centimetre |
| min | Minutes |
| h | Hour |
| g | Gram |
| nm | Nanometre |
| mm | Millimetre |
| μm | Micrometre |
| % | Percentage |
| < | Less Than |
| m | Metre |
| °C | Degree Celsius |
| wt% | Weight Percent |
| w/v | Weight Per Volume |
| kV | Kilo Volt |
| s | Seconds |
| mm/min | Millimetre Per Minutes |
| n | Number of Fibres |
| g/mol | Gram Per Mol |
| $gm/cm^3$ | Gram Per Centimetre Cube |
| MPa | Mega Pascal |

# PENGHASILAN DAN PENCIRIAN LAPISAN KAIN TERBIODEGRADASIKAN BAGI APLIKASI PELITUP MUKA


## ABSTRAK

Peningkatan penggunaan pelitup muka 3 lapis telah menimbulkan kebimbangan mengenai pencemaran kepada alam sekitar. Pelitup muka 3 lapis semasa yang tidak terbiodegradasikan menjadi tumpuan kerana proses pelupusannya adalah sukar dan lebih banyak pencemaran yang boleh terjadi akibat pembuangan pelitup muka yang tidak betul. Beberapa kajian telah dilakukan untuk memperbaik lapisan penyaringan di dalam pelitup muka secara konsisten dengan menggunakan polimer terbiodegradasikan sebagai alternatif. Kajian ini bertujuan untuk membuat lapisan tikar serat asid polilaktik (PLA), poli (butilena adipat tereftalat) (PBAT) dan PLA / PBAT. Kesan saiz jarum yang berbeza terhadap sifat dan morfologi tikar serat PLA dan PBAT dikenal pasti. Kesan pelarut tunggal dan binari disiasat untuk menghasilkan lapisan tikar gentian PLA / PBAT. Hasil kajian menunjukkan bahawa jarum bersaiz kecil menghasilkan serat berdiameter kecil. Jarum terbaik untuk digunakan dalam pembuatan tikar serat PLA dan PBAT masing-masing adalah 25G dan 23G. Jarum 25G menghasilkan tikar gentian berdiameter terkecil dan padat. Walau bagaimanapun, jarum 21G menghasilkan tikar gentian PLA dengan kekuatan tegangan dan modulus Young tertinggi. Sebaliknya, jarum 25G adalah ukuran jarum terbaik untuk menghasilkan tikar serat PBAT dengan sifat tegangan yang baik. Pelarut binari yang menggunakan DCM dan DMF menghasilkan serat berterusan dan nipis dan menunjukkan sifat tegangan yang lebih tinggi berbanding dengan sistem pelarut tunggal.

# FABRICATION AND CHARACTERIZATION OF ELECTROSPINNING BIODEGRADABLE FABRIC LAYER FOR FACE MASK APPLICATION

## ABSTRACT

The increased use of 3-ply facemasks has raised concerns about the pollution they cause to the environment. The non-biodegradability of current 3-ply facemasks is highlighted because it is difficult to manage the waste and much more pollution is created when facemasks are discarded. Several studies have been conducted in order to improve the filtration layer on a consistent basis by using biodegradable polymers as an alternative. The present study aimed to fabricate polylactic acid (PLA), poly (butylene adipate-co-terephthalate) (PBAT) and PLA/PBAT fibre mat layers. The effect of different needle sizes on the properties and morphology of PLA and PBAT fibre mats was identified by using the Scanning Electron Microscope (SEM) tabletop, ImageJ and Universal Tensile Machine (UTS). In addition, the effect of single and binary solvents was investigated to produce PLA/PBAT fibre mat layer. Results showed that small size needles produced small-diameter fibres. The best needles to use for the fabrication of the PLA and PBAT fibre mats are 25G and 23G, respectively based on the fibre diameter and tensile properties. A 25G needle produced the smallest diameter and compact fibre mat. However, a 21G needle produced a PLA fibre mat with the highest tensile strength and Young's modulus. On the other hand, a 25G needle is the best needle size to produce a PBAT fibre mat with good tensile properties. Binary solvents using DCM and DMF produced continuous and thinner fibres and showed higher tensile properties compared to the single solvent system.

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Disposable surgical or medical face masks are typically made up of three or four layers of non-woven fabrics created through spun bonding and melt-blowing manufacturing processes, as well as emerging electrospinning or texturized film extrusion technologies (Chellamani et al., 2013; Pan et al., 2020; Chua et al., 2020a). These fabrics are mostly microfibers derived from petroleum-based non-biodegradable polymers such as polypropylene (PP), polyethylene (PE), polyurethane (PU), polystyrene (PS), polycarbonate (PC), and polyacrylonitrile (PAN) (Chua et al., 2020b; Abbasi et al., 2020), can generate many micro-sized particles more easily than bulk plastic wastes and further fragment into nano plastics that disperse into ecosystems. As a result, it is critical to recognise this looming environmental threat and begin planning for long-term solutions to mitigate the effects while meeting the massive mask demand during the COVID-19 pandemic. Due to the increasing environmental awareness and demand for green products, the development of biodegradable/biobased plastics and their composites has been encouraged globally by both the industrial and academic sectors to reduce plastic waste in landfills and conserve petroleum resources and energy.

Because of their non-harmful effects, biodegradable polymers have several advantages for environmental conservation. Poly (lactic acid) (PLA) and poly (butylene adipate-co-terephthalate) (PBAT) are two of the most viable biodegradable plastics that have been commercialised to eventually replace conventional plastics to solve the environmental problems caused by plastic waste. PLA, one of the most prominent eco-friendly biodegradables, is regarded as a versatile substitute for those finite resources due to its cost-effective industrial processes for obtaining its monomers from vegetable resources (Garlotta, 2001; Balla et al., 2021). PLA is biodegradable and has characteristics similar to PP, PE, or PS. It can be produced from already existing manufacturing equipment (those designed and originally used for petrochemical industry plastics). This makes it relatively cost-efficient to produce.

PBAT is more flexible and has a higher elongation at break than most biodegradable polyesters, such as PLA, making it a better choice for packaging films. Despite its strong background in industrial applications, PBAT has received little attention in the medical device field, with only a few articles mentioning the possibility of its use in clinical settings. The main limitations of PBAT for wider industrial and medical applications are its poor thermal and mechanical resistance. The biodegradability of PBAT is determined by its chemical structure and environmental degradation conditions. Although it is necessary, PBAT biodegradability alone will not ensure consumer acceptance of this material. High production costs, as well as low thermophysical and mechanical resistance compared to non-biodegradable polymers, are barriers to the use of this biodegradable material. As a result, the development of a PBAT market will be possible only when production costs are reduced, and their properties are improved.

PBAT is regarded as an excellent candidate for toughening PLA materials through blending while retaining biodegradability. Kumar et al. (2010) fabricated the PLA/PBAT blend using a melt blending technique and discovered that increasing the PBAT loading increased both the impact strength and tensile properties of the PLA matrix, and the incorporation of glycidyl methacrylate (GMA) also increased the impact strength.

The filter media can be manufactured by electrospinning technology. In this technique, high voltage is applied to the polymer solution to form a fine filamentous structure (Ramakrishna et al., 2006). The process can be easily optimized to design filter media as per the required pore size distribution, thickness, and permeability (Tebyetekerwa et al., 2020). The electrospinning method utilizes high-voltage static electricity to form a jet of polymer melt or solution and has the advantages of simple manufacturing equipment, wide application range, controllable process, high production efficiency, and low cost. It can spin fibres of different shapes and orientations as required to meet various performance requirements and is considered one of the most effective means for nanofiber preparation.

Ultrafine fibres have distinct properties such as an extremely high surface-to-volume ratio, diverse surface chemistry, and the ability to form high and interconnected porosity. When compared to conventional micro-fibrous filters, ultrafine fibres filter significantly increases the possibility of particulate matter deposition. These benefits may help to avoid secondary pollution caused by particle shedding/leaking. As a result, the ultrathin fibrous filters can physically block ultra-small particles and viruses while not losing static electricity. As a result, much research is conducted to improve and obtain the optimum processing parameters to achieve the best filtration layer for the face mask.

## 1.2 Problem Statement

The increasing use of facemasks significantly increases the production of facemasks, and it consumes a higher amount of energy. A study by Klemeš et al. (2020) showed that mask production consumes about 10-30 watt-hour ($Wh$) energy and releases 59 g of Carbon dioxide equivalent ($CO_2eq$) greenhouse gas to the environment. Further, ever-increasing uses of face masks also increase landfill and medical waste. Most of these face mask wastes contain either PP and/or PE, PU, PS, PC, or PAN, which add plastic or microplastic pollution to the environment (Akber et al., 2020). This indicates that the current ongoing pandemic increases environmental pollution and negative impact on human and animal health. Therefore, sustainable solutions need to reduce the environmental impacts, while meeting the masked demand.

Therefore, to overcome the problem, other alternative solutions including the reuse, reprocessing, and disinfection of approved disposable masks, and producing biodegradable masks and homemade or non-certified masks are being developed (Rubio-Romero et al., 2020). The usage of biodegradable polymer can help to reduce and eliminate the plastic and pollution problem caused by facemasks. However, the research on the development of the new type of facemask layer using biodegradable polymers such as PLA, PBAT and PLA/PBAT blend by using the electrospinning method is still limited and many areas need to be investigated such as the suitable needle size for the fabrication of PLA and PBAT fibres. Besides, the use of a suitable solvent system either single or binary solvents to fabricate the PLA/PBAT fibre mats is investigated to see the effect on the morphology and tensile properties.

## 1.3 Research Objectives

The main goal of this research is to develop a new middle layer made up of a biodegradable polymer as an alternative to the filtration layer in the current facemask. The specific research objectives are as follows:

I. To investigate the effect of different needles size on the tensile properties of PLA and PBAT fibres.

II. To investigate the effect of different solvent systems on the tensile properties of the PLA/PBAT fibre mats produced by using the electrospinning process.

## 1.4 Thesis Outline

**Chapter 1:** Discusses the development of a disposable facemask, including the facemask's background, problem statement, and objectives.

**Chapter 2:** Discusses the literature review concerning the development of the biodegradable facemask, fabrication of the new filtration facemask layer, classification and performance properties, fabrication of nanofiber mat by electrospinning method, study on the effect of different needle sizes, and characterisation of facemask layer in reference to previous works or studies on this topic.

**Chapter 3:** Covers the raw materials used in the fabrication of the biodegradable facemask layer, as well as the fabrication method and characterisation method.

**Chapter 4:** Reports on the results and findings of the experimental work performed, as well as information on the relevance of the completion method used in this study.

**Chapter 5:** Discusses the project's findings and recommendations for future research.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Development of Biodegradable Face Mask

Single-use face masks, as the name suggests, are intended to be used once and then discarded. While polypropylene is the most common material used to make face masks, other alternatives include polystyrene, polycarbonate, polyethylene, or polyester (Chellamani et al., 2013). In addition, the face masks contain either aluminum or plastic nose clips, and two elastic cords. Thus, single-use face masks are prevalent among health workers and the public due to their high filtration capacity, lightweight, affordability, convenience, breathability, and disposability. Knicker and Velasco-molina (2021) estimation of the amount of carbon dioxide exclusively produced following biodegradation of single-use polypropylene-based face masks revealed an additional annual contribution of 41 to 68 t year−1 provided 0.1% of masks end up in the soil. Furthermore, they pointed out that the mean residence time for these masks in soil ranged from 2 to 3 days and between 7 and 18 years for easily decomposable and non-easily decomposable mask fragments, respectively. However, a synthesized understanding of face masks in the environment and the interaction between societal drivers and the environment is still lacking.

The term face masks (single-use face masks or surgical masks) refer to a three-layer disposable face mask. Before the COVID-19 pandemic, the last two decades saw a large-scale surge in mask use in response to the SARS (severe acute respiratory syndrome) epidemic of 2003 and increasing air pollution problems in parts of Asia (Sim et al., 2014; Zhang and Mu, 2018). According to certain estimates, the face mask market grew from USD 737 million in 2019 and is expected to hit USD 22,143 million at the close of 2021 (Markets and Markets, 2020).

The current COVID-19 pandemic has somewhat normalized the global use of face masks outside hospital environments in many parts of the world. However, the sight of people wearing face masks in many parts of Asia has always been commonplace; becoming somewhat like an entrenched culture (Joung, 2020). Be it the increasing prevalence of air pollution or the response to limit respiratory infection by adopting single-use masks, industrialization, mass transportation, and mass media seem Data that accounts for how much of the estimated growth is or will be directly linked to single-use face mask production is not readily available. Life cycle assessment estimates of single-use surgical masks indicate that the most severe environmental impact incurred occurs primarily at the raw material procurement stage (Lee et al., 2021). Zooming in further, some estimates indicate that if 1% of face masks were improperly disposed of, it will account for about 40 tons of plastics per month in nature (WWF, 2020). Also, government policies that mandate every citizen to wear face masks when outdoors are playing a key role in mass usage and production of masks. The overall environmental impact can be well understood through the life cycle assessment of face masks. Furthermore, due to environmental processes, littered face masks can Stormwater flooding, rivers, and wind are the main pathways of face masks recovered in marine environments.

Face masks discarded in the environment break down physical and there is evidence that face masks produced by electrospinning are potential sources of microplastics, nanoplastics, and microfibers in the environment (Aragaw, 2020; Chen et al., 2021; Fadare and Okoffo, 2020; Ma et al., 2021). The research was significant in understanding the potential impacts of discarded face masks on the environment. The physical, physiological, and ecotoxicological damage of discarded masks to domestic and wildlife has been recognized by researchers (Hiemstra et al., 2021; Patrício Silva et al., 2021). Because of the increasing presence of face masks in the environment, particularly the marine environment, there is a need to reduce the risk associated with them. Measures such as waste management, waste utilisation, alternative products, and biodegradable face masks have been implemented to reduce the influx of face masks into the environment.

Multiple studies show the great potential for using biodegradable polymers as substitutes for the traditional materials used in the manufacturing of face masks (Das et al., 2020; Majchrzycka, 2014). The development of reusable or substitute fibrous products based on green and biodegradable precursors can be one of the solutions to alleviate this problem (Das et al., 2020; Desai et al., 2009; Neisiany et al., 2020). Also, different combinations of biopolymers are used to achieve higher filtration efficiencies and more functionalities, including polylactic acid/polyhydroxybutyrate (PLA/PHB) (Nicosia et al., 2015), Sericin/Poly Vinyl Alcohol (PVA)/Clay (Cloisite 30B) (Purwar et al., 2016), cellulose/poly(ethylenimine) (PEI) (Tiliket et al., 2011), and Ag nanoparticles. The majority of these studies have utilized quaternary ammonium (Nicosia et al., 2015; Purwar et al., 2016; Tseng et al., 2016), metals nanoparticles (Hiragond et al., 2018), and N-halamines (Demir et al., 2015; Ren et al., 2018); which are all well-known antibacterial agents that have been used for infection control before. Sericin/PVA/Clay (Purwar et al., 2016) have shown high filtration efficiencies along with good antimicrobial activity.

Face mask's comfort and performance depend on various parameters such as the nature of the textile, fiber diameter, special surface treatments and finishes, fitting performance, microclimate, number of layers or thickness (Lee et al., 2020; Sivri, 2018), and their composition (such as the presence of high surface area activated carbons) (Konda et al., 2020). Some nanofiber filter structures, and custom-made 3D printed filters and respirators have been developed to increase the comfort and design fit to increase the wearing time (He et al., 2020; Li and Gong, 2015; Swennen et al., 2020). There are several reports about the fabrication of novel electret filter structures in combination with bulky and open structures that allows for recharging and rejuvenating after disinfection (Hossain et al., 2020; Wang et al., 2020). The possible challenges for introducing biodegradable multifunctional surgical face masks include the limited sources in supplying bio-based substrate for such a huge consumption, the price of the final face mask to be affordable as in universal masking, the toxicity of the different high-performance materials, large-scale manufacturability, and also the durability of such effective materials on a surgical face mask.

## 2.2 Biodegradable Polymer as Filtration Layer in Face Mask

Achieving a mask with higher capacity, optimal comfort, as well as high efficiency in eliminating bioaerosol's optimal filtration of airborne particles has always been one of the goals of studies conducted in this field. For this purpose, the factors affecting the determination of the final mask quality have been focused on increasing and improving the efficiency of the mask in the centre of attention. In general, the filtering ability of the mask is influenced by the specifications of the mask filter and external factors. Mask filter specifications include the inherent properties of the materials used in the mask, such as the chemical composition of the filter, and characteristics such as the thickness and packing density of the fibres in the filter.

Due to the conditions of the mask and the high humidity and temperature created during the respiration cycle, it will lead to the formation of steam in the mask, and this process will accelerate the mechanism of penetration and faster spread of microorganisms to the inner parts of the mask. To avoid such anomalies, it is vital to analyse the transmission mechanisms of the bio-particles and particles in the mask and to monitor the design and use of the mask following such mechanisms. This review was conducted to investigate the mechanisms of particle filtration by the mask and examine the parameters affecting such as face velocity or airflow, the steady or unsteady pattern of flow, the charge state of the particle, frequency of the respiration, relative humidity, and temperature, and loading time the efficient mask filtration.

The effectiveness of the filter depends on the size of the particle; for example, in the range of particle sizes of inferior and higher than 300 nm, the values of efficiency are stated in the interval of 5–80% and 5–95% for single layer, respectively. It has been shown that some physicochemical parameters like particle structure, the potential of the aggregation of the particles, and coating of the surface of the particles affect the behaviour of the nano-sized particles. For sub-micrometre particles filtered by mechanical filters, interception and diffusion are the most dominant mechanisms. The particle form often impacts entry into the masks; for example, rode-shaped particles have less potential to penetrate, and the value is about half of the spherical particles. It should be mentioned that penetration is strengthened by increasing flow rate, and differences in flow rate do not express more risk of infection because the actual infectious or lethal dose one takes in, would also be comparable to the total breathing flow rate. It has been mentioned that during hard work, the inhalation rate can exceed 350L/min, besides it was represented that at both constant and cyclic conditions, the penetration of particles is increased due to shorter residence time through filters. The results presented that by considering a low flow rate, the efficiency of filtration is increased because primarily the time to remove sub-micrometre particles by the electrostatically charged fibres of the respirator has been expanded. Filters utilized in respirators and medical masks should permit the user to breathe, and they should be able to prevent clogging of the pore while still allowing air to flow inside the filter. Respirator and medical mask filters are commonly made of mats of nonwoven fibrous materials, like wool felt, fiberglass paper, or polypropylene.

A theoretical model is proposed to examine different parameters like face velocity, fibre diameter, packing density, filter thickness, and fibre charge density that impress the filtration characteristics of filters used for respiratory protection. However, the efficiency of the respiratory masks is in the nanometric size, where it changes for different types of respiratory masks. Different mechanisms were influential in the penetration of the particles through the masks. The studies show that for the particle sizes above 0.5 μm, gravity, inertia, and interception are the dominant mechanisms, but for the particles lower than the 0.2 μm diffusion mechanism becomes dominant. Besides, the electrostatic mechanism is less affected by the particle size, where it shows efficiency up to about 300 nm but is more affected by the flow rate. However, the other parameters are less effective, but by increasing the humidity, temperature, frequency, and changing the flow rate to the cyclic flow, the filtration efficiency decreases, whereas by increasing the loading time, it increases.

Similarly, according to Adanur and Jayswal (2020), despite the large size difference of particulates, the mask did not show any significant difference in its performance; therefore, it can be interpreted that N95 respirators are effective in filtering out the SARS-Cov-2 virus that causes COVID-19. The filtration efficacy of masks depends on fibre diameter and pore size. As the fibre diameter gets smaller, the pore size also becomes smaller; as a result, the uniform distribution of fibres per unit area is improved, which increases the filtration efficiency. When particulates pass through the pores of electrostatically charged filters, they get attracted by the fibres due to the electrostatic charge (ESC) difference between the particulates and fibres. Thus, the combination of these two mechanisms in N95 respirators allows them to show higher filter qualities than mechanical filters made of the same materials but without fibre charge.

The majority of disposable masks are made of polymers such as polypropylene, polyurethane, polycarbonate, polyacrylonitrile, polyethylene, polystyrene, or polyester, whereas masks with filtration classes FFP2 and FFP3 are typically made of polypropylene nonwoven fabric. The process employs the melt blowing technique, in which melted polymer is extruded from small nozzles to form micro-and nanofibers, resulting in high filtration efficiencies. Recent research has found strong evidence of the environmental impact of PPE such as disposable masks, pointing them out as a potential source of (micro-)plastic. When they reach the sea, oceans, and other bodies of water, they break down into smaller elements (some with diameters less than 5 mm) due to a variety of external factors. Hence, there is a need to replace non-biodegradable materials with environmentally friendly polymers.

Biodegradable polymers are the polymers that have piqued the interest of researchers in the development of a new facemask to address the current issue. Biodegradable polymers are polymers that contain monomers that are linked to one another via realistic gatherings and have shaky spine connections. **Figure 2.1** depicts the classification of biodegradable polymers. They are broken down into naturally appropriate atoms, which are then metabolised and eliminated from the body through normal metabolic pathways (Nair & Laurencin, 2005). **Table 2.1** commonly used mask materials and their products and properties (Ogbuoji et.al, 2021).

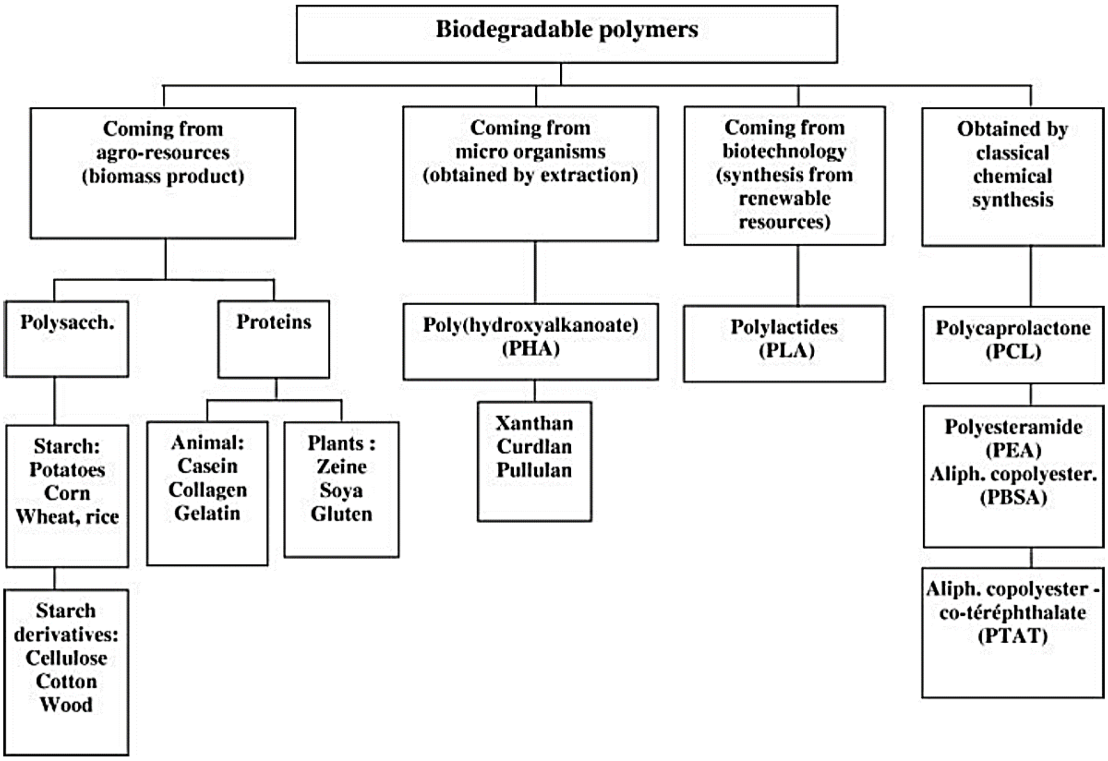


**Figure 2.1:** Classification of biodegradable polymers (Dinesh, 2020).

Polylactic acid (PLA) is a promising candidate for the development of the medical face mask filter layer, according to **Table 2.1**, due to its excellent properties. PLA has biodegradable properties and thermoplastic behaviour. PLA's polymeric backbone is composed of lactic acid monomers. PLA monomers are also known as cyclic diester lactide (CDL) or 2-hydroxypropionic acid (HPA) (Destefano et al., 2020). According to Raju et al. (2020), PLA is the most promising biodegradable polymer due to its exceptional tensile properties, making it ideal for biomedical applications. It is also biodegradable and biocompatible, making it ideal for use as a bioresorbable polymer in a variety of biomedical applications. Bio-based PLA can be thermoplastically transformed into highly transparent products to replace traditional disposable products while also helping to reduce the scarcity of petroleum resources (Fu et al., 2020a).

**Table 2.1:** Commonly used mask materials and their products and properties (Ogbuoji et al, 2021).

| Polymers | | Products | Properties |
|---|---|---|---|
| **Polyolefin** | Polypropylene (PP) | Nonwoven melt-blown and spun-bond fibers | • higher mechanical strength and is less expensive than PE<br>• abrasion resistance<br>• uniform micropore distribution<br>• modifiable inherent hydrophobicity<br>• ability to filter dry particulates<br>• high chemical (alkali and acid) resistance<br>• ease of processing<br>• recyclability |
| | Polyethylene (PE) | Meltblown nonwoven fibres | • can be made into high-density PE, low-density PE, and linear low-density PE<br>• good chemical resistance<br>• lightweight<br>• hydrophobic<br>• easier to extrude than PP due to the high shear sensitivity and higher melting temperature of PP. |
| **Polyesters** | Polyethylene terephthalate (PET) | Spunbond nonwoven fibers | • higher tensile modulus, strength, and heat stability<br>• less cost-effective than PP<br>• more difficult to recycle |
| **Polyamide** | Nylon 6 and 6–6 | Spunbond nonwoven fabrics | • Fibre lightness and high melting temperature (260 °C)<br>• unsuitable for face masks due to water absorption |
| **Cellulose Acetate (CA)** | | Electrospun nanofibrous membranes | • High filtration efficacy<br>• low thickness<br>• hydrophobic<br>• low production cost<br>• biodegradable<br>• high water stability, but soluble in organic solvents |
| **Poly- (vinyl alcohol) (PVA** | | Nanofibrous membranes | • Lightweight<br>• Biodegradable<br>• cost-effective<br>• washable, and reusable |
| **Polylactic Acid (PLA)** | | Nanofibrous membranes | • Biodegradable<br>• cost-effective<br>• favourable mechanical properties<br>• filtration efficiency of 99.99% |

**Table 2.1:** Continued.

| | | |
|---|---|---|
| **Polytetrafluoroethylene (PTFE)** | Air filter membranes | • Lightweight<br>• Hydrophobic<br>• great chemical stability<br>• high surface fracture toughness<br>• high heat resistance<br>• strong C-C and C-F bonds cause the PTEF membrane to be extensively utilized as an air filter membrane<br>• high filtration<br>• a fine particle rejection rate of greater than 99.99% |
| **Polyacrylonitrile (PAN)** | Waterproof membranes | • High cost<br>• significant variations in fiber diameters and mat morphologies<br>• chemical and thermal stabilizations |

### 2.2.1 Polylactic Acid (PLA)

Poly (lactic acid) (PLA, [CH($CH_3$) COO] n) is a synthetic biodegradable polymer that can be produced from annually renewable resources via fermentation and polymerization processes. Nanofibers are used in filtration processes, among other things. Their useful filtration properties are due to their large surface area and small pores between the fibres. One method for producing nanofibers is electrospinning, which is known for its ease of use. PLA can also be processed by injection moulding, film extrusion, blow moulding, thermoforming, and film-forming (Rasal et al., 2010). It is more thermally processible than other bioplastics such as polyhydroxyalkanoates (PHA), polyethylene glycol (PEG), and poly(-caprolactone) (PCL). The glass transition temperature ($T_g$) and melting temperature ($T_m$) values of PLA are compared to those of other polymers in **Figure 2.2.** PLA, as demonstrated, has a relatively high $T_g$ and a low $T_m$ when compared to other thermoplastics (Auras et al., 2004). PLA shares characteristics with cellophane, PS, oriented polypropylene (OPP), and oriented polyethylene. PLA has tensile strength and elastic modulus that are comparable to PET. A PLA bottle is similar to a traditional PET bottle. When compared to the production of a PET bottle, it produces 44 percent less carbon dioxide and consumes 36% less energy (Auras et al., 2004).

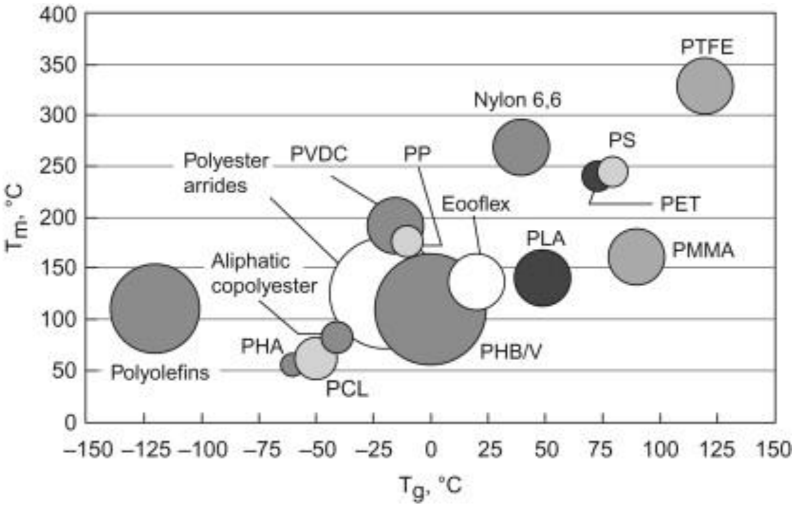


**Figure 2.2:** Comparison of $T_g$ and $T_m$ of polylactic acid (PLA) with other thermoplastics (Lim et al., 2008).

PLA, on the other hand, is a very brittle material with less than 10% elongation at break. Because of its low deformation at the break, high modulus, and hydrophilic properties, it is primarily used in rigid thermoformed packaging (Okamoto et al., 2009; Pillin et al., 2006). Another significant limitation of PLA in comparison to polyolefin is that it has poor gas barrier properties (Lehermeier et al., 2001). Furthermore, the low melt strength of PLA limits melts processing. PLA processing requires a high melt strength, such as film and sheet extrusion, blown film, and foam (Lim et al., 2008).

### 2.2.2 Polybutylene Adipate Terephthalate (PBAT)

There is a growing interest in designing new biodegradable polymers, which are the foundation of biodegradable plastics, to solve environmental problems and meet market demand. Polyesters are a particularly interesting group of polymers to study when developing biodegradable polymers. On the one hand, aliphatic polyesters are easily biodegradable due to their ester bonds in the soft chain, which are hydrolysis sensitive. Unfortunately, aliphatic polyesters such as polycaprolactone (PCL) and poly-hydroxybutyrate (PHB) exhibit poor mechanical and thermal properties. Aromatic polyesters, on the other hand, such as polyethylene terephthalate (PET) and polybutylene terephthalate (PBT), have very good physical properties but high resistance to microorganism attack.

As a result, some aliphatic-aromatic co-polyesters consisting of aliphatic and aromatic units have been synthesised and researched to design new polyesters with both satisfactory mechanical properties and desirable biodegradability. Among numerous aliphatic-aromatic co-polyesters, PBAT is the most promising and popular, with potential development prospects in a wide range of applications. It is produced by poly-condensation of butanediol (BDO), adipic acid (AA), and terephthalic acid (PTA). It has proven to be the most appropriate combination in terms of excellent properties and biodegradability. **Table 2.2** lists the commercially available aliphatic-aromatic co-polyester PBATs.

**Table 2.2:** Major commercially available Co-polyester PBAT (Jian et.al, 2020).

| Company | Country | Brand Name | Capacity (t/y) |
|---|---|---|---|
| **BASF** | Germany | ECOFLEX® | 60,000 |
| **KINGFA** | China | ECOPOND® | 50,000 |
| **NOVAMONT** | Italy | Origo-Bi® | 40,000 |
| **TUNHE** | China | - | 30,000 |
| **XINFU** | China | - | 20,000 |
| **JINHUI** | China | ECOWORD® | 20,000 |

Since aromatic polyesters PET or PBT was discovered to be resistant to hydrolysis under mild conditions and to direct attack by microorganisms, many attempts were made to increase their hydrolytic susceptibility and biological degradability by incorporating aliphatic components into the aromatic polyester chains. Witt et al. (1995) reported for the first time that the co-polyester PBAT was degraded in a compost simulation test at 60 °C up to a PTA content of about 50 mol %. The significant reduction in the weight-average molar masses of the residual materials compared to the initial molar masses indicated that biological decomposition at the surface and significant chemical hydrolysis occur within the co-polyesters.

One year later, Witt et al. (1996) published new data showing that the biodegradation rate of PBAT is affected by the amount of PTA in the polymer. Even though the biological degradation rate decreases continuously as the PTA fraction in the copolymer increases, at a content of about 50 mol percent PTA, it can be estimated that the degradation rate is still satisfied that such materials will be suitable for composting. The effect of aromatic sequences in PBAT on biodegradation was also investigated. The findings show that even longer aromatic oligomers may be biodegradable in compost at high temperatures due to chemical hydrolysis, but oligomers containing one or two terephthalates degrade quickly and easily.

Various aliphatic and aromatic oligomers could be determined and identified in an artificial high accumulation of degradation experiments on PBAT, but at the end of the experiments, only the monomers PTA, AA, and BDO were observed. The analytical methods used detected no other ester compounds that could not be related to medium components. It was demonstrated that all monomers were easily metabolised by the microbial compost population by inoculating the medium containing the degradation products with a mixed culture from compost. Organic Waster System (OWS), an international authoritative testing institution, conducted compostable testing on PBAT following standards En 13432 and ASTM D6400. **Figure 2.3** and **Figure 2.4** depict some of the available test results. In a general conclusion, material PBAT meets all of the evaluation criteria for material characteristics, biodegradation, disintegration, and compost quality outlined in these standards. As a result, PBAT can be considered fully compostable. PBAT has also received authoritative compostable certificates from Australia TUV (Belgium), DIN-CERTCO (Germany), and BPI (USA).

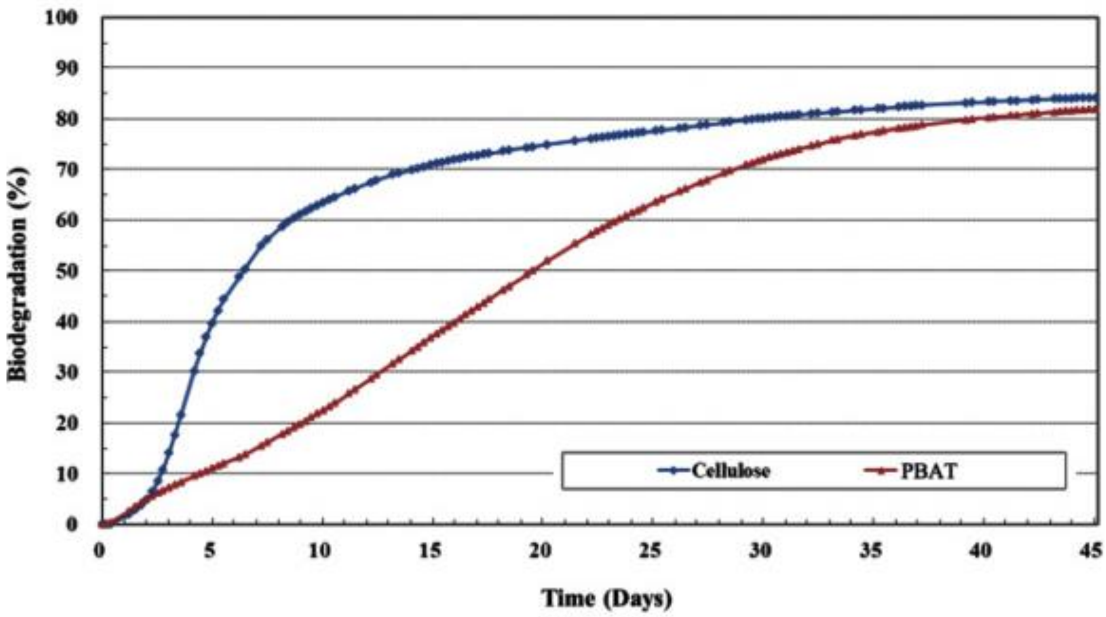


**Figure 2.3:** PBAT biodegradation under standard test conditions (Jian et.al, 2020).

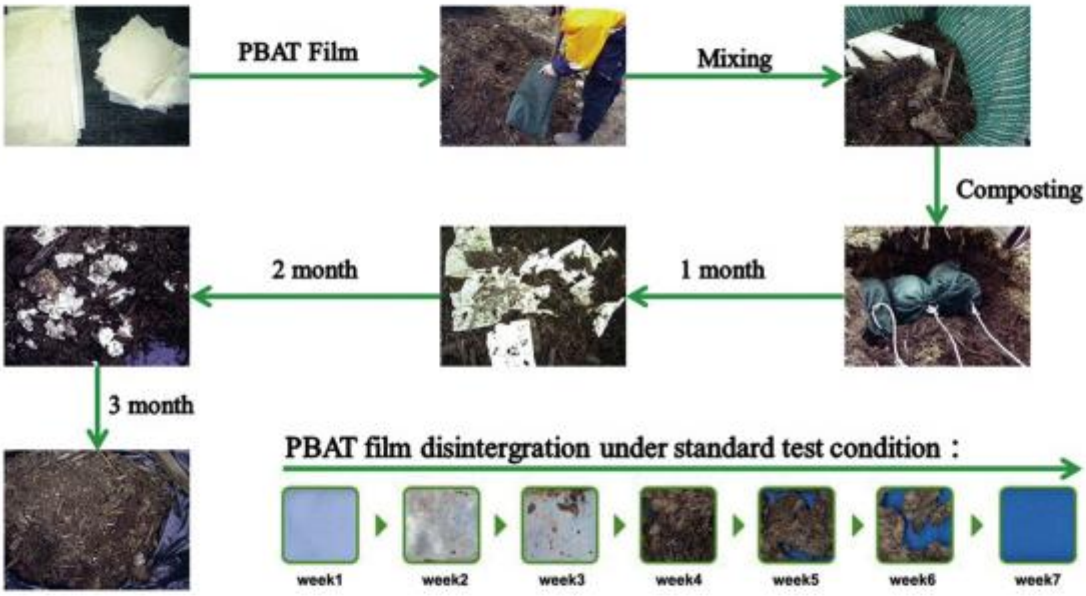


**Figure 2.4:** PBAT film compost under industry composting (Jian et.al, 2020).

PBAT has excellent mechanical properties due to the aromatic unit in the molecule chain, as well as good biodegradability due to the aliphatic unit in the molecule chain. The mechanical properties of PBAT are more flexible than those of most biodegradable polyesters, such as PLA and poly (butylene-co-succinate) (PBS) and are similar to those of low-density PE (LDPE). Because of these mechanical properties, PBAT is a very promising biodegradable material with many potential applications. The mechanical properties of PBAT have been influenced by the composition and molecular weight of the monomers. On the one hand, Lee et al. (1999) and Herrera et al. (2002) report that Young's modulus increases with terephthalate unit content while elongation at break decreases. Tensile strength, on the other hand, increases while elongation at break decreases as molecular weight increases. Based on the findings of the study, the mechanical properties of PBAT can be tailored based on the process variables chosen, such as reactor pressure and temperature, because reaction variables affect the molecular weight of PBAT.

As an example, **Table 2.3** shows the typical KINGFA values for the mechanical properties of PBAT. PBAT has comparable mechanical properties to LDPE. Tensile strength is 21 MPa, elongation at break is 670%, flexural strength is 7.5 MPa and flexural modulus is 126 MPa. The melt flow index under 2.16 kg at 190 °C is around 4, making it ideal for blowing film applications.

**Table 2.3:** The mechanical properties of PBAT (Jian et al., 2020).

| Properties | Test method | Test Condition | Units | PBAT |
|---|---|---|---|---|
| **Mechanical Properties** | | | | |
| **Tensile Strength** | ASTM D638 | 50 mm/min | MPa | 21 |
| **Elongation at break** | ASTM D638 | 50 mm/min | % | 670 |
| **Flexural Strength** | ASTM D790 | 2 mm/min | MPa | 7.5 |
| **Flexural Modulus** | ASTM D790 | 2 mm/min | MPa | 126 |
| **Thermal Properties** | | | | |
| **Melting Point** | DSC | 10 °C/min | °C | 115-125 |
| **Crystallization Point** | DSC | 10 °C/min | °C | 60 |
| **5% weight loss temperature** | TG | 20 °C/min | °C | 350 |
| **Heat Distortion Temp.** | ASTM D648 | 1.82 MPa, 6.4 mm | °C | 55 |
| **Other Properties** | | | | |
| **Melt Flow Index** | ASTM D1238 | 190 °C, 2.16 Kg | g/10min | 4.0 |
| **Specific Gravity** | ASTM D792 | 23 °C | $g/cm^3$ | 1.22 |

## 2.3 PBAT Based Blends

Pure PBAT properties are insufficient for consumer acceptance due to higher production costs or lower mechanical properties when compared to conventional plastics. As a result, the development of a PBAT market will be possible only if production costs are reduced or their properties are improved. The addition of low-cost materials (such as starch) and reinforcing materials (such as PLA) is an effective way to reduce the final price and improve the properties while maintaining the composites' biodegradability. In the last ten years, commercial series of PBAT-based composites have been developed. PBAT-based products met international compostability standards and were awarded compost certificates. These products can be processed directly on conventional plastic equipment, making them the ideal solution for preparing the same application products as conventional plastics. As a result of their high quality, satisfactory performance, and low cost, PBAT-based products are widely used in a variety of applications, including packaging, mulch film, and cutlery. **Table 2.4** shows the typical mechanical properties of KINGFA's starch-PBAT-based product and PLA-PBAT-based product. The primary information about starch-PBAT-based blends and PLA-PBAT-based blends is presented here.

**Table 2.4:** The mechanical properties of PBAT-based products (Jian et al., 2020).

| Properties | Test Method | Units | Starch/PBAT | PLA/PBAT |
|---|---|---|---|---|
| **Mechanical Properties** | A film with a thickness of 18 um | | | |
| **Tensile Strength/MD** | ISO 527 | MPa | 20.3 | 22.4 |
| **Tensile Strength/TD** | ISO 527 | MPa | 17.8 | 29.4 |
| **Elongation at break/MD** | ISO 527 | % | 287 | 258 |
| **Elongation at break/TD** | ISO 527 | % | 532 | 241 |
| **Tear Strength/MD** | ASTM D6382 | MPa | 3250 | 1590 |
| **Tear Strength/TD** | ASTM D6382 | MPa | 2840 | 2175 |
| **Other Properties** | | | | |
| **Melt Flow Index** | ASTM D1238 | g/10min | 3.5 | 4.6 |
| **Specific Gravity** | ASTM D792 | $g/cm^3$ | 1.24 | 1.22 |

### 2.3.1 Starch-PBAT Based Blends

Starch is a completely biodegradable polysaccharide that is bio synthesised by numerous plants and is one of the most abundant renewable resources. Starch is made up of carbon, hydrogen, and oxygen in proportions 6:10:5 [C6H10O5]. As a result, starch is a carbohydrate organic compound. Starch is a glucose polymer that contains linkages between the glucose units formed during condensation. It is made up of two types of molecules: linear amylose and the branched amylopectin. Amylose is primarily a linear α-D-(1–4) whereas amylopectin is a highly branched -α-D-(1–4) glucan, with α-D-(1–6) linkages at the branch tips. In general, starch contains 20–25% amylose and 75–80% amylopectin. The amount of amylose or amylopectin in starch, however, varies depending on the plant source. Starch contains lipids and proteins, but only in trace amounts. Semi-crystalline starch is defined as having a crystallinity between 20 and 40%. Amylose and amylopectin branching points are found in the amorphous region of starch.

The term "native starch" refers to starch it occurs in plants such as maize, potatoes, and wheat, among others. Because of the strong intermolecular and intermolecular hydrogen bonding in the amylose and amylopectin macromolecular chains, it has a low thermal decomposition temperature and a high melting point. However, under the combined conditions of temperature and shear, native starch can be converted into thermoplastic starch (TPS) similar to most conventional synthetic thermoplastic polymers. Various physical and chemical reactions occur during the thermal conversion of native starch to TPS. Some parameters influence the final TPS products during the extrusion process. These parameters include screw speed, feed moisture content, barrel temperature, die diameter, energy input, die pressure, and so on. Plasticizers are also important in achieving successful TPS. Some basic requirements must be met by an appropriate plasticizer. To begin, a suitable plasticizer would be a small molecule similar to the D-glucan units in starch. The second important requirement for a plasticizer is that it has a high enough boiling point to prevent it from evaporating out of the material during processing and drying. Finally, only a small amount of plasticizer is required. Water and glycerol have traditionally been regarded as the most effective plasticizers due to their small size, ease of insertion, and positioning within three-dimensional starch networks.

PBAT is a biodegradable and flexible material that is used in film extrusion and extrusion coating. Because of its high toughness and biodegradability, PBAT is a promising candidate for TPS toughening. Raquez et al. (2008) synthesised thermoplastic starch with maleic anhydride as a compatibilizer and melting blended it with PBAT. It was discovered that at lower polyester content, no significant compatibilized hydrophobic TPS granule was observed, and hydrophilic blending reaction occurred more likely due to a phase morphology inversion between both components. Ren et al. (2009) used a one-step extrusion process to create binary and ternary blends of TPS, PLA, and PBAT. To