Effect of Compatibilizer on the Aging and Degradation Mechanism of the Natural Fiber-Reinforced Thermoplastic Composites

C. Ganesan, M. Chandrasekar, M. S. Nisha, and S. Subha

Abstract Natural fiber-reinforced composites have received significant attention in recent years due to their distinct properties which include reduced carbon dioxide emission, lightweight, reduced tool wear, etc. This chapter provides an outline of the influence of different compatibilizers on the aging characteristics and degradation mechanisms of the natural fiber reinforced thermoplastic composites. Different compatibilization techniques and their effect on various properties which include mechanical, thermal, hygrothermal, chemical has been discussed. It was found that the Compatibilizers play a major role in improving interfacial characteristics and their performance will be decided by the miscibility of its components. The severity of degradation under hygrothermal aging will be lower for composites with compatibilizers and helps in retaining mechanical and thermal properties.

Keywords Compatibilizer · Thermoplastics · Natural fibres · Aging

1 Introduction

Fiber reinforced composites play a substantial role in recent decades in replacing the usage of traditional materials in various industries ranging from aerospace to sports equipments. Even though advanced fibers such as glass, carbon, aramid, etc. exhibit better properties, replacing them with an eco-friendly alternative such as natural fiber has been under investigation for the past few decades. In particular, the use of lignocellulosic fibers as reinforcement in the polymer matrix has benefits such as improved specific strength and specific modulus (Chattopadhyay et al. 2011; Atiqah et al. 2020). The natural fibers are biodegradable which can be obtained

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from renewable sources, and they are abundant in nature and cost-effective as well (Dissanayake et al. 2009; Senthilkumar et al. 2021).

Plant fiber-based composites that have found industrial applications are based on sisal (Huber et al. 2012; Senthilkumar et al. 2020), kenaf (Ochi 2008), bamboo (Deshpande et al. 2000), flax (De Prez et al. 2018; Shahroze et al. 2021) and hemp (Beaugrand and Berzin 2013). Scientists from different countries have developed different natural fibers for applications in automobile parts and other industries (Sun 2018). These applications were further extended to insulation materials such as blowing insulation, for ceiling panels that act as a thermal insulator, sound insulation material and acoustic soundproof (Müssig 2010).

Degradation of bio composite material due to the hydrophilic nature of the natural fiber under various environmental conditions such as temperature, moisture, humidity, various microorganisms, radiations can affect the performance and load-carrying capability of these composites (Tokiwa et al. 2009; Krishnasamy et al. 2019). This drawback can be overcome by the addition of a compatibilizer in thermoplastic-based composites. The upcoming sections focus on explaining the theory of compatibilization and the response of various natural fiber reinforced thermoplastic composites to aging conditions.

2 Theory of Compatibilization

The process of transforming a multiphase polymer solution into a commercially viable component is known as compatibilization. In principle, if a molecule contains a part that is soluble in one phase whilst the other is soluble in the other, it will act as a compatibilizer. As a result, the compatibilizer would be positioned around the interface which minimizes interfacial tension between materials and allows smoother dispersion while blending them with a thermoplastic matrix. Hence, this characteristic provides a degree of stability and leads to improved interfacial bonding (Ajitha and Thomas 2020). The compatibilizers can be classified into non-reactive and reactive types as discussed below.

2.1 Non-reactive Compatibilizers

Compatibilizers of the form AB copolymers are most widely used in this process for immiscible blends of polymer A and polymer B. A further choice is to use a copolymer composed of segments with distinct connections or solubility with one or more of the ingredients. The miscibility of a compatibilizer's corresponding blocks or components in each phase, as well as the compatibilizer's molecular weight, decides its performance. When a compatibilizer is inserted into an immiscible blend, it may switch to the interface between the components, allowing one end of the compatibilizer to connect with one part of the blend while the other end interacts with

the other part of the blend. This behavior of compatibilizers aid in the reduction of scattered phase proportions, thus strengthening the blend composition and enhancing polymeric adhesion with the reinforcement.

2.2 Reactive Compatibilization

Another kind of compatibilization is reactive compatibilization, which occurs when copolymers react in situ to form a solid interface. During the reactive compatibilization process, a copolymer spontaneously forms at the interface between two insoluble polymers, where it is required to regulate morphology. Reactive compatibilization includes many reactions such as (a) transreactions, (b) Reactive graft, block, or lightly cross-linked copolymer formation, (c) ionically bound structure formation and (d) mechanical and chemical mixing. The above-mentioned mechanisms can result in chain breakage and recombination, resulting in copolymer formation. Another effective technique for immiscible blend compatibilization is the use of nanofillers (Saleem and Baker 1990; Markham 1990; Xanthos and Dagli 1991; Song and Baker 1992; Liu et al. 1993; Utracki 2002; Brown 2003).

2.3 Mechanical and Thermal Properties

Hydroxyl and other polar groups in the natural fibers which makes them hydrophilic in are the principal cause of weak wear resistance between the polymer matrix and the fiber. Since the hydrophilic fractions of the fibers have a free hydroxyl group, they have poor wetting characteristics with the hydrophobic matrix, leading to inferior performance of the composites under various mechanical loads (Wu 2009). However, the efficiency of natural fiber composites can be enhanced by the use of compatibilizers and binding agents.

Malleated polypropylene (MAPP) was used to enhance the compatibility between kenaf fiber reinforcement and matrix which improve the impact strength significantly. Because of improved adhesion between fiber and polymer molecules, the crystallization was slower which resulted in reduced crystalline temperature and reduced crystallity for higher molecular weight blends and vice versa for lower molecular weight blends. Because of the improved stress transfer interface caused by the stronger possible covalent bonding between the anhydride group and the hydroxyl groups of the kenaf, MAPP, and PP, the failure strain and tensile strength were higher than the composite without MAPP.

Wu reported that acrylic acid grafted polylactic acid (AA-g-PLA) reinforced with sisal fiber (SF) demonstrated higher tensile strength, durability, and lesser biodegradability than PLA/SF due to the better compatibility between SF and PLA. Maleic anhydride grafted PLA/coir fiber-based composites yielded comparatively better performance than the PLA/coir-based composites (Wu 2009). After exposing the

composites to the bacteria named *Burkholderia cepacia*, it was discovered that the biodegradation intensity of PLA-g-MA/coir was higher than PLA but lower than PLA/coir while the biodegradability improved with the increment in coir fiber wt%. After 21 days, the weight loss percentages of PLA, PLA/coir, and PLA-g-MA/coir is approximately 15, 80, and 75%, respectively.

The temperature modulus, softening temperature and creep properties were improved due to the improved adhesion between the polypropylene matrix and kenaf due to the addition of MAPP (Feng et al. 2001). Thermoplastic elastomer (TPNR) has many advantages in properties like elasticity, damping, characteristics and thermal stability. TPNR is a combination of natural rubber and thermoplastic that can be used for applications including household appliances and automotive parts (Tanrattanakul and Bunkaew 2014; Bhattacharya et al. 2020). Because of the strong chemical interaction between the phases, properties such as tensile strength, hardness, elongation at break, and tear strength were substantially improved when MNR, NR-g-PMMA, and ENR-50 were used as compatibilizers. "The TPNR with ENR-50 and MNR as compatibilizers demonstrated a higher enticing force due to the presence of epoxide and anhydride groups in the ENR and MNR molecules as they interacted with the PBAT molecule" (Kalkornsurapranee et al. 2020).

3 Degradation

3.1 Chemical Degradation

When polymers/fibers are exposed to chemicals such as acids, bases, solvents, and other chemicals, chemical oxidation occurs.

3.2 Thermal Degradation

Thermal degradation is the decay of a polymer's molecular structure caused by temperature change. This happens at the melting temperature of thermoplastic polymers, as the state of the polymer is shifted from solid to liquid. The polymer backbone chain components detach and react with one another, modifying the polymer's properties.

3.3 Mechanical Degradation

Mechanical degradation refers to the macroscopic changes that happen in polymer materials as a outcome of compression, stress, and shear forces.

3.4 Photodegradation

Photodegradation happens as polymers undergo physical and chemical modifications as a result of ultraviolet or visible light exposure, causing Norrish and/or crosslinking reactions.

3.5 Hygrothermal Degradation

Hygrothermal deterioration refers to the significant loss of a material's weight and mechanical properties caused by moisture and temperature (Balakrishnan et al. 2011).

4 Aging Effects

Inácio et al. (2018) investigated the effect of maleic anhydride grafted polypropylene on the physical and mechanical properties of the bamboo/recycled talc/ethylene-propylene-diene monomer composites subjected to aging in a hot air chamber at 90 °C for 7 days. From their results, it can be observed that the addition of MAPP had an encouraging effect on the tensile strength, fatigue life and flexural properties while the impact energy, tensile modulus and strain at break from the tensile test showed a decreasing trend for the aged composite specimens. The reduction in impact energy was believed to be due to the weakening of the bond between the polymeric chains and the compatibilizer. Furthermore, incorporation of MAPP resulted in a marginal increase in thermal degradation temperature of the aged composites as indicated from their TGA results (Inácio et al. 2018).

Pinewood waste/Low-density polyethylene scrap waste-based composites incorporated with 2.5 wt% of maleic anhydride grafted polyethylene were subjected to accelerated weathering for 48, 168, 672, 720, 1344 and 4032 h. Their results indicate that deterioration in modulus of elasticity, ultimate tensile strength and elongation deteriorated at 4032 h compared to 720 h for the pure polymer without the reinforcement. The decline in tensile properties under the accelerated aging was caused by the formation of microcracks in the aged specimens due to the residual stress generated within the polymer and chemical changes within the polymer due to aging. The pinewood fibers in the composite absorbed the incident UV irradiation such that the severity on the composite due to aging was lower compared to the pure polymer (Moreno et al. 2018).

Badji et al. investigated the effects of natural weathering under climatic conditions and natural weathering under glass by subjecting the hemp/PP/MAPP in proportions of 10/87.3/2.7 and 30/67.1/2.1 in terms of wt% for 12 months. According to them, Young's modulus decreased with the aging time for both the pure polymer and composite specimens. However, composites exhibited superior elastic modulus

over the pure polymer irrespective of aging since hemp fibers imparted stiffening effect to the composite. Among the aging type, the composite specimens exposed to glass weathering showed more variation in elastic modulus than the naturally weathered specimens. The composites reinforced with hemp fibers showed yellowing due to aging over the pure PP which was also confirmed by the higher value of lightness evolution measured for composites than the pure PP. Higher color variation in composites was mainly due to oxidation occurring in hemp fiber exposed to weathering. Lightness evolution reached saturation and remained constant after 9 and 12 months of exposure under natural weathering and under glass weathering. This is due to the formation of holocellulose on the fiber surface over the increased exposure time. The composites exposed to natural weather under climatic conditions exhibited microcracks as well as hollow spots indicating fiber removal compared to the microcracks in the exposure time. Moreover, average surface roughness increased with the exposure time for the weathered specimens and the surface roughness values were comparatively higher for naturally weathered specimens than the under glass weathering (Badji et al. 2018).

Hydrothermal aging characteristics of the sisal/polylactide composites incorporated with 2.5 wt% maleic anhydrides (MAH) and 0.3 wt% of dicumyl peroxide (DCP) was investigated by immersing the composite specimens in 65, 75 and 85 °C hot water until saturation. The composites added with MAH and DCP had a smooth surface (Fig. 1b) compared to the rough surface for the control specimen without the compatibilizer (Fig. 1a). The composites incorporated with MAH and DCP also showed reduced water uptake. The severity of the degradation under hydrothermal aging was lower for the composites incorporated with compatibilizer (Fig. 1d) as could be noted from the lesser number of microcracks over the composites without compatibilizer (Fig. 1c) (Gil-Castell et al. 2014).

Hamour et al. exposed acetylated alfa fiber/PP composites added with 5 wt% MAH to hydrothermal aging at a temperature of 65 °C under a relative humidity of 75% up to 1000 h. Both the onset temperature and residue % obtained from the thermogravimetric analysis was found to decline for the aged specimens. Similarly, crystallinity index (CI) calculated from the X-ray diffraction spectra and young's modulus also decreased for the aged specimens compared to the pristine. The drop in CI was believed to be due to the chain scission which can lead to reduced molecular weight representing an increase in the rigidity of the polymer while the decrease in modulus of elasticity was caused by the plasticization effect of MAH. The plasticization effect for aged composites with MAH was also visible from the lower storage modulus obtained from the dynamic mechanical analysis (Hamour et al. 2015).

Hemp/high-density polyethylene (HDPE) composites added with MAH grafted polyethylene were exposed to water aging and accelerated aging (moisture, temperature and UV radiation). The composite specimens exposed to accelerated aging showed higher tensile strength and modulus of elasticity than the unexposed specimens. In the case of the water aging, the ultimate tensile strength of the composite did not change much while there was a drop in young's modulus. The fiber/matrix interface was severely affected for water aging despite the occurrence of coupling

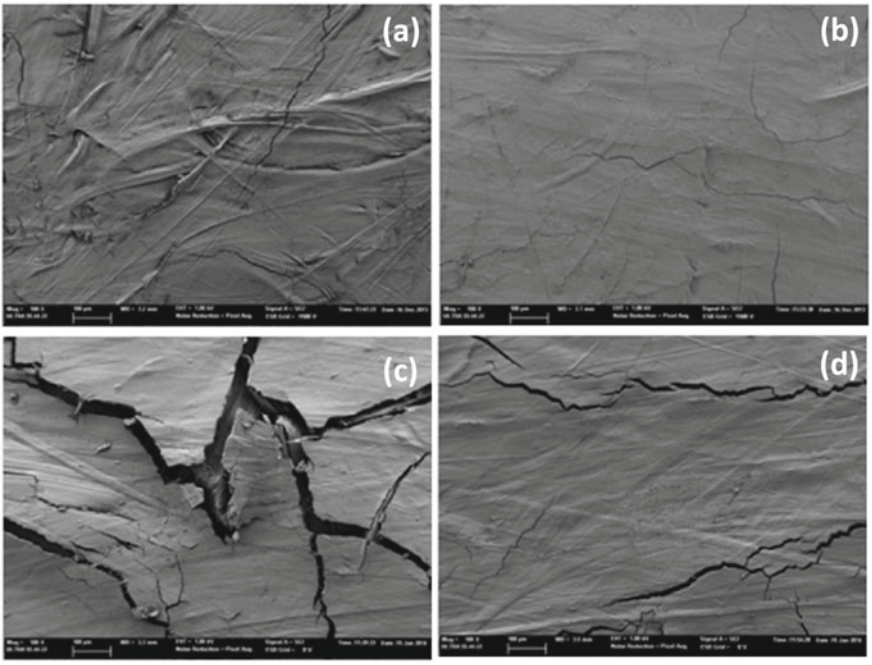

Fig. 1 Sisal/polylactide composites **a** Smooth surface on dry specimen without compatibilizer, **b** Rough surface on the dry specimen incorporated with MAH and DCP, **c** Microcracks in hydrothermally aged specimens without compatibilizer and **d** Microcracks in hydrothermally aged specimens with MAH and DCP (Re-used with permission from Elsevier, License number 5053070771941) (Gil-Castell et al. 2014)

agent compared to the accelerated aging in which the fiber/matrix interface remained intact due to the presence of coupling agent (Sergi et al. 2019).

According to Fajardo et al., Bamboo/PP composites infused with 3 wt% MAPP and 3 wt% of citric acid exhibited better thermal stability and a lower drop in tensile properties than the composites without compatibilizer exposed to the natural climatic weathering for 1 year. The increased retention in tensile properties for composites with MAPP and citric acid is mainly due to the better compatibility between the fiber and matrix (Fajardo Cabrera de Lima et al. 2020). In their study by Bajwa et al., oak wood fiber/cotton burr and stem/guayule bagasse reinforced HDPE with 1 wt% MAPE were exposed to accelerated weathering under UV irradiation and condensation cycles for 2200 h. Flexural strength, flexural rigidity and impact strength were found to decrease while the compressive strength improved for the aged specimens. However, the addition of MAPE helped retain the mechanical properties better than the composites without MAPE. The degradation in the fiber-matrix interface was lower due to the occurrence of coupling agent according to the study (Bajwa et al. 2015).

5 Applications

Natural fiber-reinforced composites are in higher demand for various applications that concern environmental sustainability. The market growth of natural fiber composites from 2014 to 2020 is almost 28% (Rwawiire et al. 2015). The automobile product such as door panels, roof, seat backboard, dashboard and trunks are being manufactured using natural fiber-reinforced composites. According to this literature (Liu and Dai 2007), the annual requirement for natural fibers exceeds 45,000 tons every year. The major reason for using natural fiber for the automobile is their biodegradability and easier recycling of material (Wilson 2017). According to the European community's 2000/53/EC "End of life vehicles" program, 95% of automobile products must be recyclable. Similarly, the Japanese government has also recommended that 95% of automobile products should be recyclable (Sun 2018). All automobile manufacture should comply with this requirement to sustain themselves in the market.

A product like outdoor bed boards, park benches, fences, indoor decors and building templates are manufactured based on wood-plastic composite. Such applications are produced on large scale by China-based manufactures and later, many countries like North America, Germany and India have elevated the production of natural fiber-reinforced composites (Dammer et al. 2017). Besides that, natural fibers have applications in storage and transportation containers, office and household goods.

6 Conclusion

Compatibilization in the natural fiber composites plays a fine role in improving their properties particularly the interfacial characteristics in addition to their positive contribution in improving the quality of the environment. This chapter discussed numerous compatibilization techniques and their effects on mechanical, thermal, hygrothermal, and chemical properties. Some of the important findings are summarised as follows.

- Compatibilizer allows smoother dispersion while blending with a thermoplastic matrix and provides a degree of stability which leads to improved interfacial bonding.
- The compatibilizer's performance will be decided by the miscibility of its corresponding components in each phase, as well as its molecular weight.
- The principal cause of poor wear resistance between the polymer matrix and the fiber is the Hydroxyl and the other polar groups present in the natural fibers which makes them hydrophilic.
- Composite with MAPP has higher failure strain and tensile strength than the composite without MAPP due to the improved stress transfer interface caused by the stronger possible covalent bonding between the anhydride group and the hydroxyl groups of the kenaf, MAPP, and PP.

- The temperature modulus, softening temperature and creep properties were improved due to the improved adhesion between the polypropylene matrix and kenaf due to the addition of MAPP.
- The incorporation of MAPP resulted in a marginal increase in thermal degradation temperature of the aged composites.
- The severity of the degradation under hydrothermal aging was lower for the composites incorporated with compatibilizer over the composites without compatibilizer
- Flexural strength, flexural rigidity and impact strength were found to decrease while the compressive strength improved for the aged specimens. However, the addition of MAPE helped retain the mechanical properties better than the composites without MAPE

Recent developments in natural fibers with compatibilizers can be used in a variety of industries, including biomedicine, agriculture, aerospace, manufacturing, automotive, construction and electrical applications. Many natural-based fibers can be designed to have enhanced mechanical and thermal properties, as well as to be deliberately or accidentally degraded under suitable environmental conditions.

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