PREPARATION OF MALEATED POLYBUTYLENE ADIPATE-co-TEREPHTALATE AS A COMPATIBILIZER FOR POLYBUTYLENE ADIPATE-co-TEREPHTALATE /THERMOPLASTIC STARCH BLENDS

Nguyen Chau Giang*, Vu Minh Duc
School of Material Science and Engineering - Hanoi University of Science and Technology

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ABSTRACT

Maleated poly(butylene adipate-co-terephthalate) could be utilized as a compatibiliser for a blend of poly (butylene adipate-co-terephthalate) and thermoplastic starch as the maleic anhydride units grafted on the poly (butylene adipate-co-terephthalate) chain can form a strong chemical bond with the hydroxyl group of thermoplastic starch, resulting in a reduction of interfacial tension between these two immiscible phases. This work focuses on investigating the simultaneous impacts of maleated poly(butylene adipate-co-terephthalate) synthesis conditions on grafting reaction efficiency. The grafting reactions were performed with dicumyl peroxide as an initiator with concentration varied from 0.25 to 0.75 wt% at each given maleic anhydride content of 3.0, 5.0 and 6.0 wt%. The molecular structure of the maleated poly(butylene adipate-co-terephthalate) was confirmed using Proton Nuclear Magnetic Resonance, water contact angle measurements and the degree of grafting was assessed using titration. Increasing dicumyl peroxide and maleic anhydride content leads to rising in degree of grafting, but side reactions may occur, which results in branching or chain scission of the poly(butylene adipate-co-terephthalate) chain. The produced maleated poly(butylene adipate-co-terephthalate) was found to be highly effective in improving interfacial adhesion of the poly(butylene adipate-co-terephthalate)/ thermoplastic starch (60/40 wt%) blend.

NGHIÊN CỨU CHẾ TẠO POLY(BUTYLENE ADIPATE-CO-TEREPHTALATE GHÉP MALEIC ANHYDRIDE ỨNG DỤNG LÀM CHẤT TRỢ TƯƠNG HỢP CHO BLEND PBAT VÀ TINH BỘT NHIỆT DẺO

Nguyễn Châu Giang*, Vũ Minh Đức
Trường Vật liệu - Đại học Bách Khoa Hà Nội

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TỪ KHÓA

Poly (butylene adipate-co-terephthalate)
PBAT maleat hóa
Tinh bột nhiệt dẻo
Tinh bột sắn
Polyme blend phân hủy sinh học

TÓM TẮT

Poly (butylene adipate-co-terephthalate) maleat hóa có thể được sử dụng làm chất trợ tương hợp cho blend của poly(butylene adipate-co-terephthalate) và tinh bột nhiệt dẻo do nhóm anhydrit maleic gắn trên mạch phân tử poly(butylene adipate-co-terephthalate) có khả năng tạo liên kết hóa học bền vững với nhóm hydroxyl của tinh bột nhiệt dẻo, từ đó làm giảm sức căng bề mặt giữa hai pha không tương hợp. Nghiên cứu này tập trung khảo sát ảnh hưởng đồng thời của các điều kiện tổng hợp PBAT maleat hóa đến hiệu quả phản ứng ghép. Phản ứng ghép được thực hiện với dicumyl peroxide làm chất khơi mào, thay đổi từ 0,25 đến 0,75 wt% tại các hàm lượng anhydrit maleic khác nhau (3,0; 5,0; 6,0 wt%). Cấu trúc phân tử PBAT maleat hóa được xác nhận bằng phổ cộng hưởng từ hạt nhân, đo góc tiếp xúc với nước và mức độ ghép được xác định bằng phương pháp chuẩn độ. Kết quả cho thấy mức độ ghép tăng theo hàm lượng dicumyl peroxide và anhydrit maleic, tuy nhiên có thể xảy ra phản ứng phụ như phân nhánh hoặc gãy mạch. PBAT maleat hóa chế tạo được đã chứng minh được hiệu quả cao trong cải thiện độ bám dính pha trong blend Poly (butylene adipate-co-terephthalate)/tinh bột nhiệt dẻo (60/40 phần khối lượng).

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* Corresponding author. *Email: giang.nguyenchau@hust.edu.vn*

1. Introduction

Driven by increasing environmental concerns, compostable polymers have attracted large attention as sustainable alternatives to conventional petroleum-based plastics. Among these, poly(butylene adipate-co-terephthalate) (PBAT) is widely used due to its biodegradability, favorable mechanical properties and processability, making it suitable for packaging. In addition, cassava starch, a polymer derived from very abundant and low-cost renewable resources in Vietnam, is another important biodegradable material which is easily turned into thermoplastic form (thermoplastic starch – TPS) for packaging applications. Blending PBAT and TPS brings a promising approach to developing new cost-effective and environmentally friendly materials. However, there is a difficulty that arises when blending PBAT and TPS due to their different polarities, which bring the immiscibility between the two phases in the blend. To address the compatibility issue in polymer blends, the incorporation of compatibilizers is one of the most common solutions to improve interfacial adhesions, and maleic anhydride (MA) grafting onto polymer backbones is a well-established reactive modification technique used to produce effective compatibilizers. References have shown that the use of maleated poly(butylene adipate-co-terephthalate) (PBAT-g-MA) leads to improvement in the tensile strength and modulus, as well as the rheological behavior and morphology of the PBAT/TPS blend 161] – [4] and composites based on PBAT and hydrophilic reinforcements such as cellulose nanocrystal, nanoclay, or rice husk [5] - [7].

Reactive extrusion or melt mixing are common methods employed in many studies to graft MA onto biodegradable polyester like PBAT, poly(butylene succinate) (PBS) [1], polylactic acid (PLA) [2]. According to literature, the reactive extrusion technique utilizing organic peroxides as radical initiators is an effective method to introduce anhydride functional groups onto the polyester backbones. Nabar et al. [1] employed Lupersol 101 as the initiator with concentration varied from 0.0 to 0.5 wt % at 3.0 wt % MA concentration and the MA content ranging from 0.0 to 5.0 wt % at 0.5 wt % peroxide concentration. To facilitate the grafting of MA onto PBAT chains, the other authors utilized proxide benzoyle (PB) in the melt mixer [3] - [6]. The studies conducted by Sreekantan et al. [7] showed that PBAT-g-MA could be obtained by applied dicumyl peroxide (DCP).

Higher grafting efficiency leads to better compatibility of PBAT-g-MA. Therefore, controlling processing conditions is crucial for balancing grafting content and the grafted PBAT properties [2], [8], [9]. Although previous studies have examined the effects of individual production parameters on PBAT-g-MA, the interplay and simultaneous influence of peroxide initiator content, maleic anhydride concentration, and temperature on the grafting efficiency. This is particularly true in the context of its intended application as a compatibilizer for PBAT/TPS films. Therefore, the main objective of this work is to thoroughly investigate the effects of varying peroxide initiator content, maleic anhydride content simultaneously on the degree of grafting, molecular structure, and melting properties of the synthesized PBAT-g-MA. This research also focused on improving the mechanical properties of the biodegradable films by using PBAT-g-MA to replace part of PBAT in the PBAT/TPS blend to boot TPS content up to 40 wt%.

2. Materials and methods

2.1. Materials

PBAT (grade BG1070) was supplied by ANKOR BIOPLASTICS (South Korea), which has an MFI of 2.5 - 4.5 gram per 10 min and a density of 1.25 g/cm^3. Starch from cassava was provided by Hung Duy Co. (Tay Ninh, Vietnam) with moisture content of 11-12%. Glycerol was used as plasticizers for starch was purchased Echo Chem SDN. BHD (Malaysia). Maleic anhydride (MA), dicumin peroxide (DCP) was purchased from Sigma-Aldrich Chemical Co.

2.2. Preparation of PBAT-g-MA

The preparation of PBAT-g-MA was conducted in a Leistritz parallel twin screw extruder with ten heating zones and the L/D of 36. PBAT pellets were mixed with maleic anhydride powder and

dicumyl peroxide in a dough mixer for 10 minutes before extrusion. The DCP concentration was adjusted between 0.25 and 1 wt.% at each MA concentration of 3.0, 5.0 and 6.0 wt%. The temperature profile was 130-150-170-185-185-185-185-185-185-185-185 °C from the feed throat to the die, with a screw speed of 200 rpm. The extrudate was cooled by passing it through a water tank before pelletizing and subsequently dried in an oven at 80 °C for at least 4 hours. Figure 1 illustrates the PBAT-g-MA synthesis procedure.

Figure 1. *Diagram of PBAT-g-MA preparation*

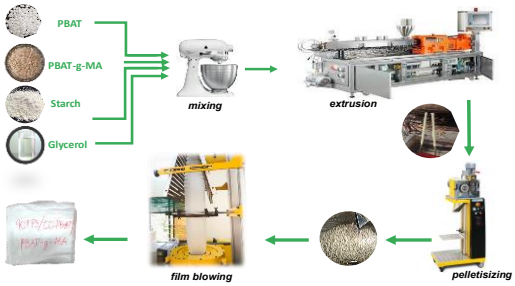

Figure 2. *Diagram of PBAT+PBAT-g-MA/TPS film production*

2.3. Preparation of PBAT+PBAT-g-MA/TPS film

Initially, a mixture of 25 wt.% glycerol to starch weight was combined with cassava starch using a dough mixer operating at 50 rpm for 10 minutes. Compositions of PBAT and PBAT-g-MA at different ratios was introduced to the starch/glycerol mixture, and the mixture was further mixed for more 5 minutes. The PBAT+PBAT-g-MA/TPS (60/40 wt%) blend was prepared in the Leistritz extruder discussed earlier, using a different temperature profile and a screw speed of 300 rpm. The resulting blends were subjected to drying at a temperature of 80 °C for a minimum of 4 hours before the blowing process. The PBAT/starch films were manufactured by blown film extrusion, employing a single screw extruder (Labtech) with a screw diameter of 25 mm and an L/D ratio of 30:1. The extrusion temperature was programmed to a range of 150-160-170-180 °C with a screw speed of 350 rpm. The procedure of PBAT+PBAT-g-MA/TPS film production is illustrated in Figure 2.

2.4. Degree of MA grafting determination

The degree of grafting (Dg) for PBAT-g-MA was determined through neutralized titration [10] and calculated following equation (1). To purify PBAT-g-MA for the titration, the extrudate samples were dissolved in chloroform at a 60 °C, and subsequently precipitated in acetone. The dried and purified MA-*g*-PBAT was dissolved in chloroform, and the solution was titrated by potassium hydroxide in methanol to a phenolphthalein end point.

$$D_g = \frac{(V_1 - V_o) \times C_{KOH} \times 98{,}06 \times 100\%}{2W} \quad (1)$$

Where V_0 and V_1 is the KOH volume [ml] for blank solution and for titration of PBAT-g-MA, respectively. C_{KOH} is the normality (moles per equivalent) of the KOH solution; W is the weight of MA-*g*-PBAT in grams.

Yield of grafting reaction (Eg) was calculated from the following equation (2):

$$\boldsymbol{E_g = \frac{m_2}{m_1}} \text{ x } 100\% \quad (2)$$

Where m_1 is weight of MA reactant (g) and m_2 is the weight (g) of grafted MA calculated from Dg

2.5. NMR

Proton (^{1}H) Nuclear Magnetic Resonance (NMR) spectroscopy was applied on purified samples to study the chemical structure of PBAT-g-MA on a ECX400 – JEOL spectrometer using chloroform as solvent at ambient temperature.

2.6. Contact angle

Water contact angle of PBAT-g-MA was determined using a Phoenix 300 Touch. The purified PBAT-g-MA samples were melt pressed into films, and the contact angle was measured 3 seconds after droplet placement on the films.

2.7. Melt Flow Index (MFI)

A Tinius Olsen Extrusion Plastometer was used to characterize the melt viscosity of PBAT and MA-*g*-PBAT according to the ASTM standard test D-123859. The standard test conditions of 190 °C and a 2.16 kg load were employed.

2.8. Morphology analysis

The micrographs of the PBAT+PBAT-g-MA/starch films were obtained on a JEOL 6400 scanning electric microscope (SEM)

2.9. Mechanical properties of the blends

The tensile test of the biodegradable PBAT+PBAT-g-MA/starch films was carried out according to ASTM D638-14 test method at a strain rate of 200 mm/min using a Lloyd instrument. Ten measurements were performed for the average values of tensile strength and elongation of the films.

3. Results and discussion

3.1. Characterizations of PBAT-g-MA

3.1.1. Degree of maleation identification

In several prior investigations on the radical grafting reaction of MA onto polyester, PBAT-g-MA was produced by varying either the MA or the initiator concentration [1], [6], [8]. In this study, the influence of MA content ranging from 3 to 6 wt.% and initiator content ranging from 0.25 to 1 wt.% on degree of maleation were studied concurrently. The results show that the graft cntent of the compatibilizers depends on both monomer and initiator concentration, as shown in Figure 3. From Figure 3, it can be observed that increasing the DCP content as well as MA content results in more MA grafting onto the PBAT backbone. At MA content of 5%, the grafting content of MA onto PBAT steadily rose and reached a saturation value of 1.96% when the DCP content was raised from 0.25 to 0.75%. Futher raising the MA content to 6% did not result in an improvement in grafting levels, but caused a considerable increase in the MFI of PBAT-g-MA compared to native PBAT. As can be seen from Figure 4, at a given initiator content, higher MA concentrations resulted in higher MFIs. As an instance, at 0.25 wt.% DCP when MA was raised from 3 to 6 wt.%, the MFI of the grafting product increased from 7.07 to 11.9 g/10min, which was three to four times greater than that of the original PBAT. To this extent, when the initiator amount was held constant at 0.5%, an increase in MFIs from 3.8 to 3.9 and 8.43 was observed as the MA content rose from 3% to 5% and 6%, respectively. As we all know, the melt flow index has a correlation to the molecular weight and viscosity of the polymer and is used to compare polymers of the same grade or batch. A low MFI polymer has a higher molecular weight than its high MFI counterpart [11]. Thus, the increase in MFI of grafted samples corresponding with rising MA content, reveals there was a drop in molecular weight of PBAT during maleated reaction process, which could be attributed to beta-scission occurring along the polymer backbone during the free-radical reaction, as reported in some previous studies [1], [8].

It is interesting to note that, contrary to the trend of MA, declines in MFI of grafted products were seen at increased DCP concentration. When PBAT-g-MA was prepared with MA content of 3 wt.%, increasing the concentration of DCP initiator from 0.25 to 0.75 wt.% resulted in a considerable decrease in MFI value from 7.07 to 1.54 g/10 min. As the MA content was kept at 5

wt.%, the grafted product's MFI sharply dropped from 8.53 to 1.14 g/10 min. The decline in MFI can be attributed to a side reaction, namely a branching reaction, occurred favourably during the maleation process due to a high concentration of the free radical initiator and generated larger molecular chains. This branching reaction is likely dominant at high DCP content above 0.5wt.% and low MA content of less than 5 wt.% because of large concentrations of free radicals generated from DCP initiator. On the contrary, at high MA amounts, larger than 5 wt. % and lower DCP concentration, the dominant side reaction would be beta scission, which causes the PBAT molecule chains to break up, as evidenced by increasing MFI value. These results were also consistent with the findings documented by Nabar et al. [8]. Nevertheless, when the MA content was increased up to 6 wt.%, the reduction in MFI was not substantial despite an increase in DCP quantity.

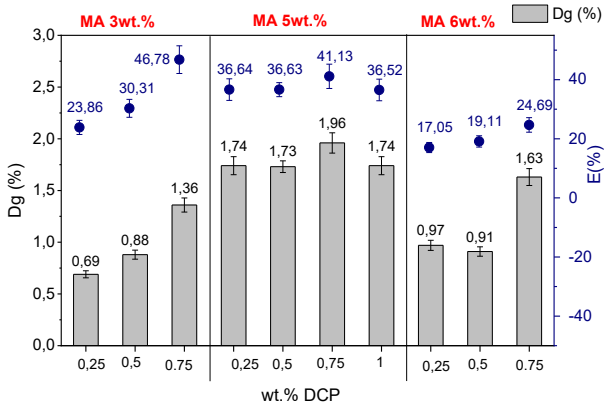

Figure 3. *Degree of grafting at different MA free radical initiator content*

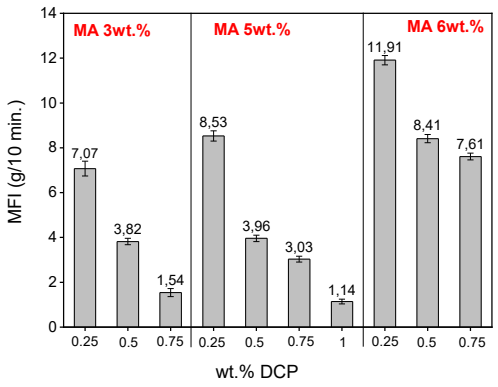

Figure 4. *MFIs of PBAT-g-MA at different MA and free radical initiator amount*

Thus, there is always competition between two opposing side reactions during the twin-screw extrusion process: branching, which increases the molecular weight of the polymer chains, and chain scission, which reduces it. The dominant reaction depends on the processing conditions. The most optimal condition for PBAT maleation is a DCP content of 0.75% by weight and an MA of 0.5% by weight.

3.1.2. Contact angle

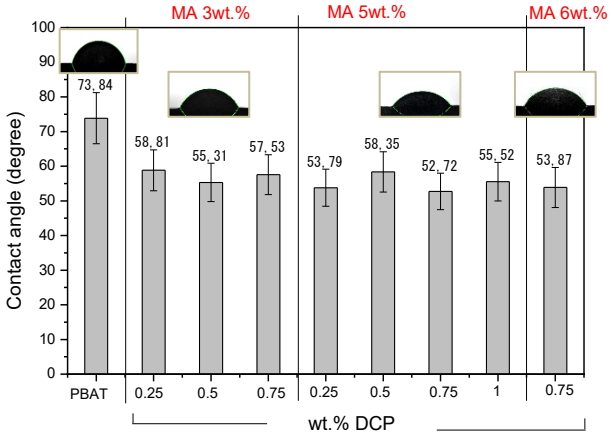

Figure 5. *Contact angle of PBAT-g-MA upon different content of MA and DCP compared to PBAT*

The MA molecule, being polar, when grafted will introduce more hydrophilic functional groups to the PBAT surface, which lower the contact angle. Therefore, in this work, contact angle was not only used to examine hydrophilicity of grafted products, but also to confirm grafted reaction of MA on PBAT by reactive extrusion. The changes in the water contact angle of PBAT-g-MA at

varying concentrations of both MA and DCP in comparison to neat PBAT are illustrated in Figure 5. The water contact angle of PBAT is 73.84 degrees, indicating its relatively hydrophobic nature. Complying with the maleation reaction, a notable drop in the contact angle is observed on the surface of all grafted samples, ranging from 52 to 59 degrees. These findings demonstrate that the grafting reactions of MA onto the PBAT backbone were successful, resulting in increased hydrophilicity of the PBAT-g-MA.

3.1.3. NMR

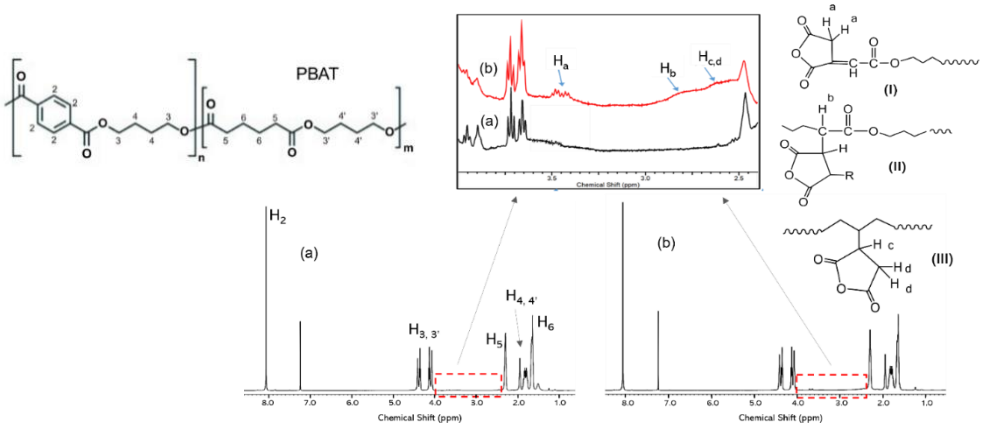

Figure 6. *^{1}H-NMR spectra of (a) pure PBAT, (b) PBAT-g-MA with D_g of 1.96*

The ^{1}H NMR spectroscopy was utilized as a primary method to confirm the chemical structures of materials, providing the most sensitive means for observing the graft. The ^{1}H NMR spectrum for neat PBAT and PBAT-g-MA having grafting degree of 1,96% are showed in Figure 6. The spectrum of the starting PBAT exhibits characteristic signals consistent with its copolyester structure. The resonances at 1.65 ppm and 1.81 - 1.96 ppm were respectively assigned to the methylene protons in the butanediol and adipate units (H_4, $H_{4'}$, H_6). The resonance peaks observed at 2.31corresponding to the outer methylene protons in the acid adipic units (H_5) while the peaks showing at 4.08 - 4.14 ppm and 4.36 - 4.42 ppm correspond to $H_{3'}$, H_3 and H_5 prontons, respectively [1], [11] The prominent signal H_2 protons appearing at 8.08 ppm indicates phenylene groups in terephalic acid units of PBAT. However, compared to the PBAT spectra, differences in the PBAT-g-MA spectrum (Figure 6b) are observed in region 2.5 to 3.6 ppm [8]. The free radical grafting mechanisms of MA onto PBAT backbone has been proposed by some previous authors following by some new structures of copolymers obtained [1], [5], [13], [14] as depicts in Figure 6. An additional signal appearing around δ 3,4 – 3.5 ppm is attributed to H_a proton of MA unit grafted on PBAT backbone of the structure I. The resonance at 2.8 – 2.9 ppm is attributed to H_b proton of α-carbon attached to MA unit of the structure II. Furthermore, the new peak was also found at 2.7 ppm in PBAT-g-MA speatra is assigned to the H_d protons in the structure III, evidenced that MA can abstract a proton from non α-carbon of terephataled and butylene segment of PBAT molecule. This finding is consistent with the report of Rahimi et al. [5]. These results suggest that the MA was grafted onto PBAT backbone.

3.2. Blend PBAT+PBAT-g-MA/TPS

The poor interfacial adhesion of blend PBAT/TPS has significantly limited the loading of TPS to just around 20–30 wt% while maintaining acceptable mechanical properties [14]. Figure 7 illustrates the mechanical properties of the PBAT+PBAT-g-MA/TPS (60/40 wt%) films with varying PBAT-g-MA, while their MFIs are shown in Figure 8. Without the addition of PBAT-g-MA, the blend was not able to be blown into film at this high level of TPS content. Therefore, no data for the uncompatibilized blend is shown in Figure 8. As can be depicted from Figure 8, rising

in PBAT-g-MA loading from 2 to 8 wt% results in notable improvements in both tensile strength and elongation of the blend films. The blend achieved a remarkable tensile strength of 19 MPa and an elongation of 240% at PBAT-g-MA loading of 4 wt%. At 6 wt% it reached a saturation point of 28,47 MPa and 330% respectively, which are about equivalent as the tensile strength of native PBAT. This enhancement is attributed to the action of PBAT-g-MA, which formed strong ester bonds with hydroxyl groups of TPS phases, effectively reducing interfacial tension. The uncompatibilized blend PBAT/TPS was difficult to melt flowing continuously, so its MFI was not measured. However, adding PBAT-g-MA significantly improved the blend's melt flowability, resulting in an increase of MFI from 4.12 to 5.34 g/10 min, as can be seen in Figure 9. For blown film applications, polymers typically require a relatively low MFI, generally ranging between 0.2 to 7 g/10 min to ensure stable film formation and consistent thickness [15]. The MFI values of the compatibilized blends are well within these acceptable levels. As a result, the inclusion of PBAT-g-MA not only improves mechanical qualities but also influences the melt flow behaviour, making it ideal for film-blowing process.

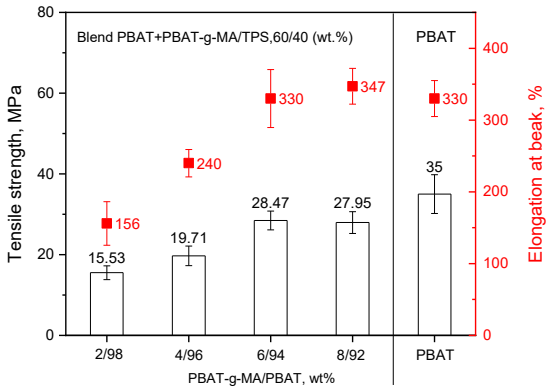

Figure 7. *Mechanical properties of the blends*

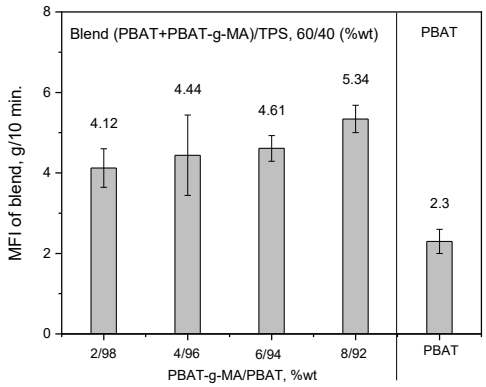

Figure 8. *Melting index of the blends*

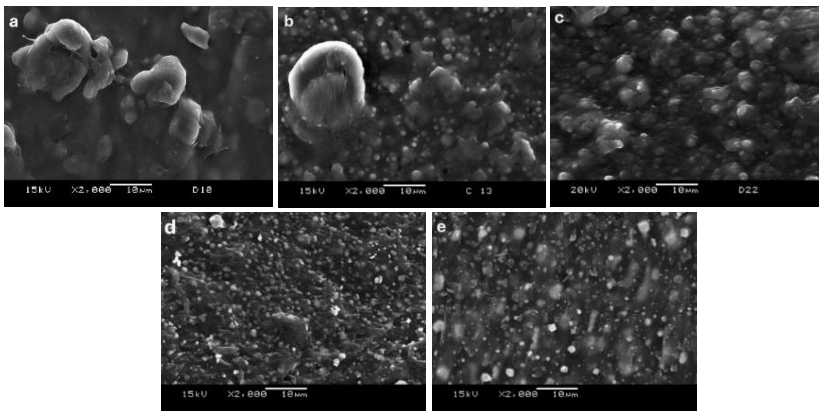

Figure 9. *SEM images of the blend with varying PBAT-g-MA/ PBAT ratio by weight, a) without compatibilizer, b) 2/98, c) 4/96, d) 6/94 and e) 8/92*

SEM images of the blend film surfaces are shown in Figure 9, which further verifies the compatibilization role of PBAT-g-MA in the blend. Figure 9a clearly shows that TPS dispersed in large sizes and irregular shapes in the PBAT matrix indicating severe immiscibility between the two phases of the blend. When PBAT-g-MA in the blend with concentration rose from 2 to 8 wt%, significant morphological refinement was seen. At a modest PBAT-g-MA loading of 2 wt% (Figure 9b), the TPS particles were shown to reduce in size and improve dispersion, although some big particles of several tens of micrometres were still present throughout the PBAT matrix. At 6

wt% (Figure 9c) and 8 wt% (Figure 9d), a very fine and highly scattered morphology is observed. These can be explained by the reactivity of MA groups on the PBAT backbone with hydroxyl groups on the TPS, which forms strong ester linkages at the surface and reduces interfacial tension, preventing phase separation. This improvement explains the better mechanical qualities depicted in Figure 7, as it allows stress to be transferred more effectively.

4. Conclusion

The grafting of MA onto PBAT chain was achieved by reactive extrusion using DCP as initiator. The main findings reveal that increasing MA concentration improves yield of grafting reaction but promotes chain scission of PBAT, resulting in a lower MFI. In contrast, raising the radical initiator concentration at a given amount of MA causes branching reactions to occur to varying degrees, resulting in larger molecular chains. The NMR spectroscopy and water contact angle studies confirmed the grafting of the MA onto the PBAT chains, resulting in a more hydrophilic PBAT. The most suitable condition for synthesizing PBAT-g-MA with a degree of MA grafting on the PBAT backbone as high as 1.96% is 5 wt% MA and 0.75 wt% DCP at 185 °C. This grafted MA degree was sufficient to successfully compatibilize PBAT and TPS at a high loading of 40%, giving a biodegradable blend film with tensile characteristics comparable to native PBAT.

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