The effect of different epoxidised vegetable oils on injection-moulded starch based thermoplastic polymer filled with almond shell powder

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Abstract: This article is focused on the development and characterization of biodegradable and eco-friendly biocomposites, based on starch thermoplastic polymer (TPS) filled with 20 wt% almond shell powder (ASP). Three epoxidised vegetable oils (EVOs) were added at 5, 10 and 20 parts per hundred resin (phr) of TPS/ASP biocomposite, as additive, to study the effect in the properties of injected biodegradable parts: epoxidised soybean oil (ESBO), epoxidised linseed oil (ELO) and epoxidised corn oil (ECO). The biocomposites are prepared in a twin-screw extruder and characterized in terms of their mechanical (tensile and Charpy impact tests) and thermal properties (differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA)). In addition, disintegration test were evaluated. Results showed the addition of EVOs successfully reduced the stiffness and brittleness produced by the incorporation of almond shell, whereas thermal stability increased. The addition of EVOs had a stabilising effect on the disintegration of the biocomposites. The most optimal performance was attained for biocomposites with ELO or ESBO between 10-20 phr.

Keywords: Almond shell, Biocomposite, Epoxidised vegetable oils, Starch polymer, Epoxidised soybean oil, Epoxidised linseed oil, Epoxidised corn oil.

1. Introduction
Nowadays, biocomposites made of biopolymer matrix and natural waste represent an interesting sustainable solution to replace conventional polymers in a wide variety of sectors, such as consumer goods, toys, automotive, building, construction and packaging. However, there are some drawbacks related to the incorporation of lignocellulosic fillers into polymers, for example, poor interfacial adhesion between filler-matrix and an increase of stiffness and brittleness.

In previous studies [1,2], it was shown that the incorporation of almond shell into the polymeric matrixes produces convenient aesthetic changes. Nevertheless, it also modifies the mechanical properties of the material by increasing its stiffness and reducing its deformation capacity and impact resistance. This effect has also been observed in other works in which almond shells have been incorporated into other biodegradable polymeric matrix such as PLA [3,4] or PBS [5-7].

This loss of mechanical properties can compromise the use of these materials in some applications. Therefore, plasticiser additives are used to mitigate these effects. Currently, the plasticiser sector for use in plastics faces several problems: the restriction on the use of phthalate-type compounds due to their...
toxicity to humans and low degradability in the environment, in addition to the social stigma which now surrounds fossil fuels. For all that, the chemical sector is focused on replacing plasticisers of non-renewable origin with compounds of renewable origin that have low toxicity and low migration levels [8]. An interesting alternative to conventional petroleum-derived plasticisers, are vegetable oils. Several studies have developed biocomposites based on biodegradable thermoplastic matrixes and natural fibres in which different vegetable oils have been added to improve the processability, mechanical ductile properties of biopolymer/lignocellulosic and the matrix/fibre compatibility [3,4,6,9,10].

The main objective of the present study has been to develop and characterise composites based on a commercial starch-based thermoplastic matrix, Mater-Bi®, with almond shells incorporating different EVOs: ESBO, ELO and ECO to obtain an eco-friendly biocomposite with high ecological performance and balanced mechanical and thermal properties.

2. Materials and Methodology

2.1. Materials
A commercially available starch-based polymer, Mater-Bi DI01A of Novamont, was used in this study. This bio-based and biodegradable polymer has a melt flow index (MFI) of 35 g/10 min (190 °C/2.16 kg) and a density of 1200 kg/m³ (data provided by Novamont). This reference was selected for their properties that are similar to polypropylene (PP).

A commercial mixture almond shell was used for this study (Figure1(a)). The mixture was provided by Hermen Europe, S.L (Spain) in the form of powder. Figure 1(b) shows the predominant particle size was approximately between 0.08-0.125 mm. Table 1 shows the content of fixed carbon, volatile matter, humidity, ash and the chemical composition of the main components: cellulose, hemicellulose and lignin determined by thermogravimetric analysis (TGA) in previous work [2].

ELO and ESBO was supplied by Traquisa S.L and ECO prepared by Technological Institute of Materials (ITM, Universitat Politècnica de València).

| Moisture (%) | Volatile matter (%) | Fixed carbon (%) | Ash (%) | Hemicellulose (%) | Cellulose (%) | Lignin (%) |
|--------------|---------------------|-----------------|--------|------------------|---------------|-----------|
| 7.4          | 64.5                | 19.4            | 8.7    | 37               | 27            | 36        |

Figure 1. (a) Commercial mixture powder. (b) Scanning electron microscope (SEM) image of almond shell powder (ASP) (∗200 magnification and a scale marker of 100 µm).
2.2. Experimental Procedure

2.2.1. Preparation of biocomposites testing samples. Prior to processing, TPS and ASP were dried separately for 4 h at 60 °C and 24 h at 105 °C, respectively, in a Pol-Eko SLW 115STD INOX air-circulating oven, to minimize its moisture and to avoid hydrolytic reactions [11].

Composites of starch-based polymer and ASP were developed using a BERSTORFF ZE26X44D-BASIC co-rotating twin-screw extruder (26:44 L/D). Before feeding the material into the extruder, a manual pre-mixing of the different components, Mater-Bi DI01A/almond shells/epoxidised vegetable oil, was carried out and fed into the extruder through the main hopper. The ratio of TPS/ASP was set in all cases at 20 wt% since this content has shown balanced mechanical and appealing aesthetics properties in a previous study dealing with TPS biocomposites [1]. ELO, ESBO and ECO were added at 5, 10 and 20 parts per hundred resin (phr) of biocomposite. The temperature profile was set as follows: 130–185–185–185 °C (from feeding zone to die). A rotating speed was 80 rpm. The extruded materials were finally pelletised using an air-knife.

Testing samples (dog-bone, Type 1A) were moulded from melt-compounded pellets, using an injection moulding machine DEMAG Ergotech 110–430h/310V. The temperature profile was 210–190–170–140–50 °C (from hopper to injection nozzle). The pack pressure and pack time were kept at 400 bar and 15 s, respectively. The cooling time was 40 s and the back pressure was 50 bar. These processing conditions were set for all biocomposites. Finally, specimens were conditioned at a temperature of 23 °C and relative humidity of 50% for at least 16 h before testing.

2.2.2. Thermal properties. Thermal transitions of developed biocomposites were studied by DSC in a DSC Q200 calorimeter for TA Instruments. Samples with an average weight of 8-10 mg were placed in standard aluminium crucibles and subjected to a three-step program that consisted of an initial heating cycle from 0 °C to 250 °C at a rate of 10 °C/min to remove thermal history, followed by a cooling to -20 °C at a rate of 5 °C/min, and a second heating cycle to 350 °C at a rate of 10 °C/min. All the tests were run in nitrogen (N2) atmosphere with a constant flow of N2 of 50 mL/min. The thermal transitions were determined: temperature and enthalpy of fusion after the second heating (Tf and ∆Hf).

The main thermal degradation parameters of biocomposites, degradation initial temperature (Tonset), and temperature for maximum mass loss rate (Tmax) were studied by TGA using a TA Instrument Q500 thermogravimetric analyser. Samples with an average weight comprised between 8 and 10 mg were placed in standard alumina crucibles of 70 µL. In this case, biocomposites were subjected to the following temperature program: from 30 to 600 °C under an N2 atmosphere at a rate of 10 °C/min, and from 600 to 1000 °C under an oxygen (O2) atmosphere at a rate of 10 °C/min with a purge gas flow of 10 mL/min.

2.2.3. Mechanical properties. Tensile tests were performed using an Instron 6025 universal testing machine with 5 kN power sensors. The tensile tests were performed according to standard ISO 527, starting with a crosshead speed of 1 mm/min, accelerating to 5 mm/min when the strain exceeded the 0.25 mm limit. The extensometer used was MTS 634.11F-54. Recorded values include ultimate tensile strength (UTS), Young’s modulus, and strain at break. A total of 5 specimens from each material were tested using standardised samples 1A (dogbone). Impact tests were performed with a Resil 5.5 impact testing device (CEAST RESILIMPACTOR) with a 1 Joule hammer. Test samples were cut and tested according to standard ISO 179 (Charpy un-notched). A total of 10 specimens from each material were tested.

2.2.4. Scanning Electron Microscopy (SEM). The impact fracture surface obtained after a Charpy impact test were analysed using a Jeol JSM-840 SEM system. Samples were gold-coated before analysis, and the energy of the electron beam was 20 kV.
2.2.5. Disintegrability in Composting Conditions. Disintegration tests in composting conditions were performed, by following the ISO 20200 Standard method. The test specimens were extracted from standardised tensile specimens obtained by injection moulding. The samples were cut into pieces (25×25×4 mm), placed on mesh (mesh size 2 mm) and buried in boxes using a compost composed of sawdust (40 wt%), rabbit feed (30 wt %), mature manure (10 wt%), corn starch (10 wt%), sucrose (5 wt%), corn oil (4 wt%) and urea (1 wt%). The boxes were placed in an oven at 58 °C and a relative humidity (RH) of 55 %. Every seven days, the samples were de-tempered, washed with distilled water to remove traces of compost extracted from the box and dried to determine the degree of disintegration, \( D \), according to the equation (1):

\[
D = \frac{m_i - m_t}{m_i} \times 100
\]

where \( m_i \) is the initial dry mass of the sample and \( m_t \) is the mass of the sample recovered after sieving after being buried for a period of time, \( t \).

3. Results

3.1. Thermal Properties of TPS/ASP Biocomposites samples

Table 2 shows the main thermal parameters obtained through DSC (second heating) and TGA characterisation. After the second heating, the material showed two melting peaks \( (T_f) \), a smaller one at around 161.7 °C, and a larger one at 169.3 °C. Also, slight variations of the baseline slope were also observed, which could be due to secondary transitions related to rearrangements of the starch chains [1].

Table 2. Thermal properties of TPS and TPS/ASP biocomposites with ELO, ESBO and ECO in terms of degradation initial temperature \( (T_{\text{onset}}) \), temperature for maximum mass loss rate \( (T_{\text{max}}) \), temperature \( (T_f) \) and enthalpy \( (\Delta H_f) \).

| Samples     | \( T_{\text{onset}} \) (°C) | \( T_{\text{max1}} \) (°C) | \( T_{\text{max2}} \) (°C) | \( T_f1 \) (°C) | \( T_f2 \) (°C) | \( \Delta H_f \) (J·g\(^{-1}\)) |
|-------------|-----------------------------|-----------------------------|-----------------------------|----------------|----------------|----------------|
| TPS         | 328.2±3.2                   | 355.5±2.7                   | 399.5±1.5                   | 161.7±1.4      | 169.3±0.6      | 28.2±0.8       |
| TPS/ASP     | 303.9±0.4                   | 343.8±1.5                   | 398.6±1.1                   | 161.7±0.5      | 168.9±0.3      | 23.5±0.5       |
| TPS/ASP+5 ELO | 304.8±0.8                  | 353.0±1.2                   | 401.3±0.1                   | 156.5±0.7      | 166.1±0.5      | 21.9±1.1       |
| TPS/ASP+10 ELO | 309.4±1.9                  | 350.3±2.6                   | 404.8±1.2                   | 156.3±0.4      | 166.4±1.6      | 20.8±0.1       |
| TPS/ASP+20 ELO | 308.8±0.8                  | 353.1±0.5                   | 410.0±0.8                   | 155.9±0.2      | 166.4±0.2      | 19.3±1.0       |
| TPS/ASP+5 ESBO | 304.7±1.2                  | 351.2±1.3                   | 400.3±0.7                   | 160.3±0.4      | 167.6±1.1      | 21.7±1.6       |
| TPS/ASP+10 ESBO | 311.2±1.0                  | 352.3±1.2                   | 399.2±1.2                   | 160.6±0.2      | 168.9±0.3      | 19.9±0.8       |
| TPS/ASP+20 ESBO | 311.21±2.1                 | 347.9±0.6                   | 404.0±0.7                   | 158.4±0.1      | 167.3±0.1      | 20.4±0.9       |
| TPS/ASP+5 ECO  | 306.0±0.5                   | 345.1±0.3                   | 397.6±1.2                   | 159.0±1.1      | 167.8±0.2      | 22.1±0.1       |
| TPS/ASP+10 ECO  | 302.6±0.7                   | 345.9±1.2                   | 398.9±1.0                   | 157.0±0.5      | 167.8±0.7      | 21.6±0.2       |
| TPS/ASP+20 ECO  | 304.6±0.5                   | 346.2±0.8                   | 403.9±0.6                   | 162.1±0.1      | 169.2±0.2      | 18.8±0.1       |

The addition of 20 wt% ASP hardly altered the reduction of the melting point temperature, remaining between 168-169 °C. In addition, there was a slight decrease in the crystallinity of the material, as demonstrated by a reduction of the enthalpy of fusion \( (\Delta H_f) \) from 28.1 to 22.8 J·g^{-1}.

The addition of EVOs provided a higher mobility of the polymer chains, which contributed to a slight decrease of the melting temperature, \( T_f1 \) and \( T_f2 \), to 156 °C and 166 °C, respectively. In addition, the addition of EVOs decreased the enthalpy of fusion \( (\Delta H_f) \) with respect to the TPS/ASP biocomposite, the crystallisation of the molecular chains decreased.

The as-received starch-based polymer degrades in a single step, and its \( T_{\text{onset}} \) was close to 325.98 °C, thus indicating moderate thermal stability. The addition of almond shell particles reduced the thermal stability of the biocomposites because almond shells start degradation earlier than polymer matrix. \( T_{\text{onset}} \) and \( T_{\text{max1}} \) moved towards lower temperature with the addition of almond shell. \( T_{\text{max2}} \) remained
practically unchanged. The addition of EVOs had a positive effect on the thermal stability of the TPS/Almond shell biocomposite. In particular, the addition of ELO or ESBO increased $T_{\text{onset}}$ from 303 °C to 309 °C and 311 °C, respectively. This effect was observed when the content of the oils is 10-20 phr. In the case of the addition of 5 phr of ELO or ESBO, the $T_{\text{onset}}$ remained practically unchanged. The same occurred when ECO is incorporated. On the other hand, the temperature $T_{\text{max1}}$ improved significantly compared to the TPS/ASP. In particular, $T_{\text{max1}}$ increased from 343 °C to 350-353 °C for TPS/ASP with ELO and between 348-352 °C, for ESBO.

The incorporation of ECO led to an increase in $T_{\text{max1}}$ of approximately 2 °C, compared to the unadditivated biocomposite. This increase in thermal stability could be related to the fact that these epoxidised vegetable oils provide a physical barrier that hinders the removal of volatile products produced during decomposition [10].

### 3.2. Mechanical Properties of TPS/ASP Biocomposites samples

Table 3 shows the results of the mechanical properties for the injection-moulded samples of as-received TPS and the TPS/ASP biocomposites processed with ELO, ESBO or ECO. The addition of 20 wt% of almond shells increased the stiffness of the material (higher value of Young’s modulus than as-received Mater-Bi DI01A) and the strength and elongation decreased. The addition of EVOs had a plasticising effect on the formulation as it decreased the stiffness of the material, obtaining values of Young's modulus, even below that as-received Mater-Bi DI01A (2096 MPa). In addition, it was observed that when increasing the ESBO and ECO content, the Young's modulus decreased progressively from 1130 MPa and 1290 MPa (oil content: 5 phr) to 1050 MPa and 845 MPa (oil content: 20 phr), respectively.

| Samples         | E  (MPa) | $\sigma_R$ (MPa) | $\varepsilon_R$ (%) | Impact strength (kJ/m²) |
|-----------------|---------|-----------------|---------------------|-------------------------|
| TPS             | 2090±46 | 38.1±2.4        | 2.7±0.1             | 29.88±0.90              |
| TPS/ASP         | 2210±25 | 20.0±1.3        | 1.6±0.1             | 4.39±0.91               |
| TPS/ASP+5 ELO   | 1110±49 | 16.0±2.1        | 1.8±0.3             | 7.17±1.62               |
| TPS/ASP+10 ELO  | 1394±40 | 18.8±0.7        | 1.9±0.1             | 11.99±1.99              |
| TPS/ASP+20 ELO  | 1326±50 | 14.0±0.1        | 1.6±0.1             | 13.51±1.91              |
| TPS/ASP+5 ESBO  | 1130±168| 11.7±2.0        | 1.9±0.7             | 7.02±1.43               |
| TPS/ASP+10 ESBO | 1051±92 | 11.2±0.9        | 1.4±0.1             | 10.99±1.61              |
| TPS/ASP+20 ESBO | 1050±92 | 11.2±0.9        | 1.4±0.1             | 10.98±1.23              |
| TPS/ASP+5 ECO   | 1290±113| 15.9±1.6        | 1.9±0.3             | 7.90±0.17               |
| TPS/ASP+10 ECO  | 1253±67 | 9.3±1.0         | 1.0±0.2             | 5.32±0.79               |
| TPS/ASP+20 ECO  | 1253±67 | 8.7±1.2         | 1.2±0.2             | 4.25±0.96               |

The addition of EVOs reduced the tensile strength of the biocomposite due to the plasticising effect of the oil. Only in the case of ECO, by increasing its amount from 5 to 20 phr, the tensile strength decreased progressively from 15.9 MPa to 8.7 MPa. The variation of the ESBO content did not have a significant effect on the strength of the material, remaining around 11 MPa. Regarding the elongation at break, the incorporation of EVOs practically did not improve the ductility of the material, as can be seen in the obtained values, which remained between 1.2-1.9 % compared to 1.6 % for the TPS/ASP biocomposite without additives. The highest elongations at break, between 1.8 % and 1.9 %, occurred when the concentration of ELO, ESBO and ECO is 5 phr.

The addition of almond shell increased the brittleness of the material. Mater-Bi DI01A had an impact strength of 29.9 kJ/m² which decreased to 4.4 when incorporating 20 % almond shells. The addition of EVOs increased the impact resistance of the biocomposite. The highest values of impact resistance, between 11-13 kJ/m², were presented by the formulations with ELO and ESBO between 10-20 phr.
Regarding the additive concentration in the formulation, it was observed that increasing the concentration of ELO and ESBO from 10 phr to 20 phr did not have an effect on the impact resistance of the biocomposite.

Figure 2(a) shows the SEM images corresponding to TPS/ASP biocomposite. A gap between almond shell particles and the surrounding starch-based polymer matrix can be observed (see arrows). The effect of 20 phr ECO and ESBO on improving particle-polymer interactions is positive. Figure 2(b) and 2(c) show the SEM images corresponding to ELO and ESBO (20 phr) compatibilized biocomposites. In both cases, the gap between the ASP and the surrounding matrix is lower. In the case of the ELO-compatibilized TPS/ASP biocomposite, particles seem fully embedded in the TPS matrix. Regarding ESBO-compatibilized TPS/ASP biocomposite, a plastic deformation (polymer fibrils) can be observed. Both results support the better impact resistance results obtained previously, which showed that these biocomposites presented a more ductile and resistant behaviour.

![Figure 2](image-url)

**Figure 2.** Scanning electron microscope (SEM) image of the impact fracture surfaces of the biocomposites (×150 magnification and a scale marker of 100 µm) (a) TPS/ASP. (b) TPS/ASP+20 ELO. (c) TPS/ASP+20 ESBO.

### 3.3. Disintegration Tests

The weight loss of the injection-molded pieces during the disintegration process can be observed in figure 3. In addition, figure 4 shows the visual aspect of the samples during the disintegration process.

![Figure 3](image-url)

**Figure 3.** Disintegrability (%) of TPS and TPS/ASP biocomposites with 10 and 20 phr as a function of degradation time under composting conditions at 58 °C.
The incorporation of almond shell in the formulation accelerates the disintegration rate with respect to the as-received Mater-Bi DI01A. This could be attributed to the hydrophilic character of the lignocellulosic fibres, which allows a higher permeability of water to the polymeric matrix, activating the hydrolytic degradation process. It can be observed that from week 4, the difference in weight loss between two samples is significant. After 12 weeks of the test, the TPS/ASP biocomposite reached a disintegration rate of 76% compared to 70% for the as-received material.

The addition of EVOs had a stabilising effect on the disintegration of the biocomposites. From week 3-4, it could be observed that the sample with 10 phr ESBO showed a greater mass loss than the rest of the formulations, increasing progressively until week 9, from which the sample is stabilised and did not lose more mass. In the case of the biocomposite with 20 phr ESBO, the degradation rate was equal to that of 10 phr from week 6 onwards. In both cases, a degree of disintegration of 60% was achieved at the end of the test. The ECO-added biocomposites showed a mass loss of 70% at the end, similar to Mater-Bi DI01A. Biocomposites containing ELO showed the lowest weight loss percentage of disintegration at the end of the test (32-42%).

|       | Week 1 | Week 2 | Week 3 | Week 4 | Week 5 | Week 6 | Week 7 | Week 8 | Week 9 | Week 10 | Week 11 | Week 12 |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|---------|---------|---------|
| TPS   |        |        |        |        |        |        |        |        |        |         |         |         |
| TPS/ASP |       |        |        |        |        |        |        |        |        |         |         |         |
| 10 phr ELO |     |        |        |        |        |        |        |        |        |         |         |         |
| 20 phr ELO |     |        |        |        |        |        |        |        |        |         |         |         |
| 10 phr ESBO |   |        |        |        |        |        |        |        |        |         |         |         |
| 20 phr ESBO |   |        |        |        |        |        |        |        |        |         |         |         |
| 10 phr ECO  |   |        |        |        |        |        |        |        |        |         |         |         |

**Figure 4.** Visual appearance of Mater-Bi DI01A-based biocomposites with 20 wt% of ASP at different testing weeks at 58 °C.

4. Conclusions

TPS/Almond shell biocomposites were successfully manufactured on conventional extrusion-compounding and injection moulding equipment. The almond shell powder was incorporated at 20 wt% and different concentrations of ELO, ESBO and ECO were added to improve the polymer/particle interfacial adhesion.

Experimental results revealed that the incorporation of almond shells increased the stiffness and the brittleness of the material. The addition of EVOs increased the flexibility of the biocomposite, reaching values of Young's modulus even lower than those of the as-received TPS. In addition, an improvement in impact strength was also observed.

The TPS/almond shell biocomposites with 20 phr ELO or ESBO offered balanced properties. In particular, both formulations showed the highest impact strength values and acceptable Young's modulus. Additionally, it achieved the optimal TPS/ASP interaction, as confirmed by SEM analysis.

Regarding the thermal properties, the addition of EVOs had a positive effect on the thermal stability of the TPS/ASP biocomposite. In particular, the addition of ELO or ESBO between 10-20 phr increased \( T_{\text{onset}} \).

As for the disintegration tests, the incorporation of almond shells accelerates the disintegration rate with respect to as-received TPS. This is attributed to the hydrophilic character of the almond shell, which
allows a higher permeability of water to the polymeric matrix, activating the hydrolytic degradation process. The addition of EVOs had a stabilising effect on the disintegration of the material.

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