Biodegradable plastics for improving soil and fruit quality characteristics

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AGENDA/

AITIIP Centro Tecnológico

Introduction: General overview of the project
Materials and Methods
Results and Discussion
Conclusions
AIITIP is a private technological centre whose goal is to increase the competitiveness of companies in the industry of moulds and plastic parts manufacturing.

AIITIP offers advanced technology services to other companies (62% of the income), performs research, development and innovative actions (37% of the income) and training and e-training (1%).

Key Figures (2017)
- 100% privately owned
- 49 employees
- 12,000 m²
- 7 M€ annual turnover
- 1 M€ annual investment
- 175 clients

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European Projects

Circular Economy
- Agricultural waste valorisation
- Biopolymers
- Green composites

Industry 4.0
- Multi-material additive manufacturing and 3D printing
- Robotization of processes

Main Sectors
- Automotive
- Aeronautic
- Agroindustry & Food Packaging

Basic Research
Applied Research
Product proven in real world
INTRODUCTION:
General overview of the project

MATERIALS AND METHODS

RESULTS AND DISCUSSION

CONCLUSIONS
General overview of the project

They are used mainly in Italy, France, Germany, Benelux and Spain.

Production

Consumption

End of life

They are used mainly in Italy, France, Germany, Benelux and Spain.
The overall objective of the project is to demonstrate that the sustainability and efficiency of agricultural practices can be achieved by introducing an innovative, economically viable and soil biodegradable plastic that eliminates waste completely.
Mulching Innovations

- **Boron** deficiency appears in dry climates (important for plant metabolism)
- **Zinc** is required by many enzymes (hormone auxin, little leaf)
- **Manganese** is necessary for photosynthesis (coloration)

Fruit protection bags innovations

- **Macro perforations** to prevent rotting due to the concentration of water vapour.
- **Pigment Skin Active Radiation** capable of producing colour surface fruit.

General overview of the project
General overview of the project

**Raw materials**
- 100% biodegradable
- Polymers based on natural sources

**Production processes**
- Materials extrusion
- Film blowing

**Validation of plastics in laboratory**
- Mechanical tests of materials
- Tests for certification OK BIODEGRADABLE SOIL

**Validation of plastics products in fields**
- Tomato (Spain and France), pepper and cucumber in Spain and sweet potato in Belgium
- Bags for apple and peach (Spain)

**Validation of quality:**
- Soil
- Crop (Pre-harvest)
- Product (Post-harvest)

**Expected results**
- Reduction of plastic waste
- Less CO₂ emitted during the production of plastics/Non-emissions from disposal
- Improvement of soil quality
- Improvement in crop quality
- Certification OK BIODEGRADABLE SOIL
AGENDA//

AITIIP Centro Tecnológico
Introduction: General overview of the project
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Materials & Methods: Production of bioplastics

Control samples:
Conventional LDPE mulching (Comercial Arnedo, Spain)
Conventional waxed paper bags (Cooperative Calanda DO).

Samples | Additives
--- | ---
MULCHING | 2016 M11 A%-B%
| 2016 M21
| 2016 M31
| 2017 M12 A%-B%
| 2017 M42

FRUIT BAGS | 2016 B11 A%-B%
| 2016 B21
| 2016 B31
| 2017 B12 A%-B%
| 2017 B42

Materials & Methods: Production of bioplastics

Zn/Mn complex or Boron

Biopolymers AAPE, Biopolymers PHA-PLA or Biopolymer PBS

Additives

EXTRUSION-COMPOUNDING MACHINE COPERION ZSK26

Film blowing unit LABTECH LF 400

Moreto X DRY AIR T Minidryers

Mulching

Film for fruit bags

Semi-industrial facilities
Materials & Methods: Bioplastics validation

Laboratory plastics validation

<table>
<thead>
<tr>
<th>BIOPOLYMERS CHARACTERIZATION</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Properties of Thin Plastic Sheeting</td>
<td>ASTM D 882 – 12</td>
</tr>
<tr>
<td>Elasticity</td>
<td>ISO 604</td>
</tr>
<tr>
<td>Biodegradation test</td>
<td>ASTM D 5988-12</td>
</tr>
<tr>
<td>Plastics – Methods of exposure to laboratory light sources</td>
<td>EN:ISO 4892-3</td>
</tr>
<tr>
<td>Heavy metals concentration of the biofilms</td>
<td>EPA 3052 1996</td>
</tr>
<tr>
<td>“OK Biodegradable Soil”</td>
<td>EN13432:2000</td>
</tr>
</tbody>
</table>

Heavy Metals concentration

\[ > \text{Limits (EN 13432)} = \text{Not accepted} \]

Volatile solids concentration

\[ < 50\% = \text{Not accepted} \]

Biodegradation test on soil (ASTM D5988)

\[ < 90\% \text{ within 2 years} \]

Final compost/ SOIL QUALITY

Chemical-physical parameters and heavy metal NOT comply with National or European limits for Regulation on fertilizers

Eco toxicological test on final composted residues

\[ < 90\% \text{ compared with blank sample} \]
Materials & Methods: Vegetables and fruit samples

Tomato *Solanum lycopersicum* “Manitu”
- Planting dates:
  - 24/May/2016
  - 02/June/2017
- Collection dates:
  - 25/August/2016
  - 31/August/2017

Area Mid-Ebro Valley (Zaragoza, Spain):
Year 2016: 0.2 Ha Year 2017: 0.5 Ha

Area Ebro Valley (Calanda, Spain):
1 ha, 5×4 m

Peach *Prunus persica* “58GC”
- Bagging dates (middle of season):
  - 14/July/2016
  - 17/July/2017
- Collection dates:
  - 13/September/2016
  - 06/September/2017

300 bags/batch were tested (2016) and 500 (2017) and randomly distributed in six blocks, with one tree each block.

Treatments were randomly distributed in three blocks, with six repetitions each block.
Materials & Methods: Soil collection and analysis

- Samples were collected randomly in each repetition of each block of plastics. Each sample was composed of 8 sub-samples taken throughout the entire line.
- Soil electrical conductivity, pH, total carbon, total N and total macro- and micronutrient was determined.
- Soil samples, only for mulching, were collected from the upper layer (20 cm).

Incorporation plastics (Y1): 28/03/17
2 months before first soil sampling
At the beginning (Y2): 24/05/2017
At the end (Y2): 03/10/2017
Incorporation plastics into the soil: 07/11/17
Materials & Methods: Quality parameters

150 fruits per experimental unit were analysed

- CIELab colour space with the aid of a spectrophotometer (Konica Minolta mod. CMS 700; Tokyo, Japan).
- Firmness was measured with non-destructive Acoustic Firmness Sensor (AWETA; Netherlands) for peaches and Durofel (Agrosta; Forges Les Eaux, France) for tomatoes.
- Destructive Magness-Taylor using a digital penetrometer (Agrosta) with a tip diameter of 8 mm for peaches and of 4 mm for tomatoes.
- Soluble solid content (SSC) as Brix degrees was determined by crushing the flesh and transferring the intact juice of the 10 samples to a digital refractometer (Atago mod. PR-101; Tokyo, Japan).
- Titratable acidity (TA) by an automatic titrator (Mettler Toledo mod. G20 Compact Titrator; New York, NY, USA).
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Introduction: General overview of the project

Materials and Methods

Results and Discussion

Conclusions
• Additives in general decrease modulus of elasticity.
• Moreover, oligo elements made samples bear lower tensile stress ($\sigma$).
• Samples M1 were much more resistant to fracture ($\varepsilon=552-615\%$) than the others, while M31 was more difficult to process and manipulate.
• Sample M12 showed the best mechanical properties of all samples and similar values to the conventional plastics.

Results and Discussion

<table>
<thead>
<tr>
<th>Year</th>
<th>Material</th>
<th>Zn/Mn Level</th>
<th>Thickness (µm)</th>
<th>E (Mpa)</th>
<th>$\sigma$ (Mpa)</th>
<th>$\varepsilon$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2016</td>
<td>M11</td>
<td>M11A</td>
<td>20 (0)</td>
<td>183 (69)</td>
<td>24.3 (2)</td>
<td>552 (194)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>M11B</td>
<td>38 (4.1)</td>
<td>55 (10)</td>
<td>8.3 (2)</td>
<td>615 (117)</td>
</tr>
<tr>
<td></td>
<td>M21</td>
<td>M21A</td>
<td>20 (0)</td>
<td>166 (35)</td>
<td>6 (3)</td>
<td>235 (118)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>M21B</td>
<td>30 (0)</td>
<td>108 (29)</td>
<td>5.4 (1)</td>
<td>214 (73)</td>
</tr>
<tr>
<td></td>
<td>M31</td>
<td>M31A</td>
<td>30 (0)</td>
<td>245 (35)</td>
<td>17.3 (3)</td>
<td>154 (10)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>M31B</td>
<td>30 (0)</td>
<td>127 (25)</td>
<td>12 (4)</td>
<td>62 (54)</td>
</tr>
<tr>
<td></td>
<td>Control (LDPE)</td>
<td>-</td>
<td>42 (8)</td>
<td>300 (14)</td>
<td>4.5 (1)</td>
<td>600 (20)</td>
</tr>
<tr>
<td>2017</td>
<td>M12</td>
<td>M12A</td>
<td>31 (1.5)</td>
<td>190 (55)</td>
<td>25.5 (1.8)</td>
<td>430 (90)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>M12B</td>
<td>33 (1.2)</td>
<td>160 (63)</td>
<td>22.1 (2.2)</td>
<td>583 (129)</td>
</tr>
<tr>
<td></td>
<td>M42</td>
<td>M42A</td>
<td>51 (4.9)</td>
<td>137 (60)</td>
<td>6.4 (2.9)</td>
<td>247 (88)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>M42B</td>
<td>40 (3.3)</td>
<td>122 (55)</td>
<td>4.9 (3.3)</td>
<td>226 (61)</td>
</tr>
<tr>
<td></td>
<td>Control (LDPE)</td>
<td>-</td>
<td>12 (2.6)</td>
<td>187 (20)</td>
<td>26 (3.8)</td>
<td>280 (39)</td>
</tr>
</tbody>
</table>
Results and Discussion

• The amount of oligoelements added to the mulching was calculated for fertilization.
• The process was efficient preserving the amounts of oligoelements in final plastics.
• For label “OK biodegradable SOIL” the percentage of Zn has to be lower than the regulation limit of 150 mg kg\(^{-1}\).
• For fruit protection bags, all samples are below the allowed limit (data not showed).

<table>
<thead>
<tr>
<th>Metal</th>
<th>Control</th>
<th>M11A</th>
<th>M11B</th>
<th>M21 A</th>
<th>M21B</th>
<th>M31A</th>
<th>M31B</th>
<th>DL (mg kg(^{-1}) dm)</th>
<th>EN 13432 (mg kg(^{-1}) dm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>2.5</td>
<td>5</td>
</tr>
<tr>
<td>Cadmium</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>0.19</td>
<td>0.5</td>
</tr>
<tr>
<td>Chromium</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>0.70</td>
<td>1.33</td>
<td>1.5</td>
<td>&lt;DL</td>
<td>0.77</td>
<td>0.5</td>
<td>50</td>
</tr>
<tr>
<td>Mercury</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>0.3</td>
<td>0.5</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>0.5</td>
<td>1</td>
</tr>
<tr>
<td>Nickel</td>
<td>1.15</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>1</td>
<td>25</td>
</tr>
<tr>
<td>Lead</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>2</td>
<td>50</td>
</tr>
<tr>
<td>Copper</td>
<td>6.63</td>
<td>&lt;DL</td>
<td>1.61</td>
<td>&lt;DL</td>
<td>1.70</td>
<td>&lt;DL</td>
<td>2.39</td>
<td>1</td>
<td>50</td>
</tr>
<tr>
<td>Selenium</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>&lt;DL</td>
<td>0.5</td>
<td>0.75</td>
</tr>
<tr>
<td>Zinc</td>
<td>&lt;DL</td>
<td>5.88</td>
<td>1360</td>
<td>7.18</td>
<td>1700</td>
<td>10.5</td>
<td>2010</td>
<td>5</td>
<td>150</td>
</tr>
</tbody>
</table>

Standard EN 13432 for compostable packaging

Heavy Metals (mg kg\(^{-1}\) dm) in mulching samples (2016)
## Results and Discussion

### Biodegradation (mean & sd)

<table>
<thead>
<tr>
<th></th>
<th>After 143 days</th>
<th>After 176 days</th>
</tr>
</thead>
<tbody>
<tr>
<td>M11A</td>
<td>98,6 0,28</td>
<td>99,9 0,00</td>
</tr>
<tr>
<td>M21A</td>
<td>79,6 3,54</td>
<td>99,2 0,85</td>
</tr>
<tr>
<td>M31A</td>
<td>76,4 4,10</td>
<td>97,9 1,84</td>
</tr>
<tr>
<td>B31B</td>
<td>80,55 8,27</td>
<td>98,5 1,27</td>
</tr>
</tbody>
</table>

### Biodegradation on soil - ASTM D5988-03

- **Heavy Metals concentration**: > Limits (EN 13432) = Not accepted
- **Volatile solids concentration**: < 50% = Not accepted
- **Biodegradation test on soil (ASTM D5988)**
  - < 90% within 2 years
- **Final compost/ SOIL QUALITY**
  - Chemical-physical parameters and heavy metal NOT comply with National or European limits for Regulation on fertilizers
- **Eco toxicological test on final composted residues**
  - < 90% compared with blank sample

### Biodegradation %

- Not accepted
Results and Discussion

- An increase in the concentration of Mn and Zn was observed using the bioplastics (2016 results). This result shows that the **oligoelements are released into the soil** after plastic degradation.
- For the macronutrients, **N was not affected**, meanwhile the concentration of **P and K was higher** using our bioplastics than control one.

<table>
<thead>
<tr>
<th>Time</th>
<th>Material</th>
<th>Oligoelement Level</th>
<th>N (g 100g⁻¹)</th>
<th>P (g 100g⁻¹)</th>
<th>K (g 100g⁻¹)</th>
<th>Mn (mg kg⁻¹)</th>
<th>Zn (mg kg⁻¹)</th>
<th>C/N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beginning of 2016 season</td>
<td>-</td>
<td>-</td>
<td>0.15</td>
<td>0.10 a</td>
<td>1.18 a</td>
<td>369.4 bc</td>
<td>71.2 b</td>
<td>34.68 a</td>
</tr>
<tr>
<td>Beginning of 2017 season and after 4 months of incorporation of the bioplastics into the soil</td>
<td>M11</td>
<td>M11A</td>
<td>0.16</td>
<td>0.10 a</td>
<td>1.33 bc</td>
<td>345.66 a</td>
<td>68.73 a</td>
<td>28.25 bc</td>
</tr>
<tr>
<td></td>
<td>M11B</td>
<td>0.17</td>
<td>0.15 b</td>
<td>1.31 bc</td>
<td>363.96 b</td>
<td>72.16 bc</td>
<td>23.01 c</td>
<td></td>
</tr>
<tr>
<td></td>
<td>M21</td>
<td>M21A</td>
<td>0.16</td>
<td>0.09 a</td>
<td>1.21 b</td>
<td>347.83 a</td>
<td>67.24 a</td>
<td>32.44 ab</td>
</tr>
<tr>
<td></td>
<td>M21B</td>
<td>0.16</td>
<td>0.11 ab</td>
<td>1.32 bc</td>
<td>378.14 bc</td>
<td>73.24 bc</td>
<td>28.16 bc</td>
<td></td>
</tr>
<tr>
<td></td>
<td>M31</td>
<td>M31A</td>
<td>0.17</td>
<td>0.10 a</td>
<td>1.28 bc</td>
<td>354.12 ba</td>
<td>71.92 ab</td>
<td>25.98 c</td>
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<td></td>
<td>M31B</td>
<td>0.15</td>
<td>0.11 ab</td>
<td>1.47 c</td>
<td>383.94 c</td>
<td>75.49 c</td>
<td>30.96 b</td>
<td></td>
</tr>
<tr>
<td>Control</td>
<td>-</td>
<td>0.16</td>
<td>0.09 a</td>
<td>1.23 b</td>
<td>342.34 a</td>
<td>67.03 a</td>
<td>31.11 b</td>
<td></td>
</tr>
</tbody>
</table>

Different letters in the same column indicate significant differences (p≤0.05) between treatments.
Results and Discussion

• Quality tests carried out on tomatoes have also shown that fruit growth and quality, especially concerning total dry weight, soluble solids, colour and shape gave very similar results between using biodegradable plastic and control mulch.

<table>
<thead>
<tr>
<th>Year</th>
<th>BATCH</th>
<th>Material</th>
<th>Oligoelement Level</th>
<th>Firmness (kg)</th>
<th>Durofel</th>
<th>Weight (g)</th>
<th>SSC (ºBrix)</th>
<th>a* (D65)</th>
<th>Blossom end rot (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2016</td>
<td>M11</td>
<td>M11A</td>
<td>0.32 a</td>
<td>65.04 a</td>
<td>102.11ab</td>
<td>6.73 c</td>
<td>32.76 ab</td>
<td>&lt;1</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>M11B</td>
<td>0.39 d</td>
<td>68.18 b</td>
<td>107.84abc</td>
<td>6.27 ab</td>
<td>34.17 c</td>
<td>&lt;1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>M21</td>
<td>M21A</td>
<td>0.38 cd</td>
<td>70.26 b</td>
<td>97.97a</td>
<td>6.60 bc</td>
<td>31.69 a</td>
<td>&lt;1</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>M21B</td>
<td>0.38 bcd</td>
<td>68.90 b</td>
<td>105.5abc</td>
<td>6.23 ab</td>
<td>33.12 bc</td>
<td>&lt;1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>M31</td>
<td>M31A</td>
<td>0.39 cd</td>
<td>63.62 a</td>
<td>113.42c</td>
<td>5.93 a</td>
<td>31.99 ab</td>
<td>&lt;1</td>
<td></td>
</tr>
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<td></td>
<td></td>
<td>M31B</td>
<td>0.34 abc</td>
<td>68.76 b</td>
<td>102.54ab</td>
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</tr>
<tr>
<td></td>
<td>Control</td>
<td>-</td>
<td>0.33 ab</td>
<td>69.02 b</td>
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<td>6.73 c</td>
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<tr>
<td>2017</td>
<td>M12</td>
<td>M12A</td>
<td>0.44 bc</td>
<td>68.06</td>
<td>143.75</td>
<td>6.47</td>
<td>32.24 7 a</td>
<td>a</td>
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<td></td>
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<td>M12B</td>
<td>0.48 c</td>
<td>70.72</td>
<td>140.33</td>
<td>6.3</td>
<td>32.51 7 a</td>
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<td></td>
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<td>M42A</td>
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<td>69.17</td>
<td>146.99</td>
<td>6.5</td>
<td>32.48 8 a</td>
<td>a</td>
<td></td>
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<td></td>
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<td>M42B</td>
<td>0.37 a</td>
<td>69.21</td>
<td>128.58</td>
<td>6.53</td>
<td>31.23 8 a</td>
<td>a</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Control</td>
<td>-</td>
<td>0.41 ab</td>
<td>70.88</td>
<td>141.48</td>
<td>6.33</td>
<td>32.04 18 b</td>
<td>a</td>
<td></td>
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</tbody>
</table>

1 different letters in the same column indicate significant differences (p≤0.05) between treatments for the same year.
In 2016 season, although significant differences were observed, there was no clear pattern in the use of different plastics for tomatoes. In 2017 season, no differences were observed, showing that the plastics did not have effect in these quality parameters.

Only remarkable that in 2017 the incidence of blossom end rot, a water-soaked spot located at the blossom end of tomato fruits, was higher in the control (18%) than in bioplastics M12 (7%) and M42 (8%). This result could be related with a different temperature in the soil of each plastic, different reflected sunlight, or even the Zn/Mn concentration but it is necessary more assays to confirm this hypothesis.
Firmness, weight, acidity and soluble solids of the peaches were not affected by using biobags. Differences observed may be due to intrinsic differences in crops more than effect of the bioplastics.

A lower red coloration in the fruits (lower coordinate a* value) and a more homogeneous yellowish colour. These values were also lower than the control ones.

<table>
<thead>
<tr>
<th>Year</th>
<th>BATCH Material</th>
<th>WP%</th>
<th>Firmness (kg)</th>
<th>Aweta</th>
<th>Weight (g)</th>
<th>T.A. (g.malic L(^{-1}))</th>
<th>SSC (ºBrix)</th>
<th>a* (D65)</th>
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<tr>
<td>2016</td>
<td>B11</td>
<td></td>
<td>3.19</td>
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<td>14.03 c</td>
<td>14.88 bc</td>
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</tr>
</tbody>
</table>

Different letters in the same column indicate significant differences (p≤0.05) between treatments for the same year.
Results and Discussion

Control

Lower WP %

Higher WP %
AGENDA

AITIIP Centro Tecnológico

Introduction: General overview of the project

Materials and Methods

Results and Discussion

Conclusions
The following conclusions can be drawn from the study:

- Additives (oligoelements and/or colourants) made more difficult plastic processing. In general, all selected bioplastics showed appropriate mechanical properties.
- Adequate biodegradation in the field and in the laboratory was observed with selected bioplastics.
- The addition of Zn is not proper to obtain the “OK biodegradable SOIL” certification, but fertilization effect was reached: higher concentration of Mn and Zn than in control samples was founded in the soil.
- Biomulching in tomatoes decreased the incidence of blossom end rot, improving the production, and not affecting to the rest of quality parameters.
- For peaches, the colour obtained with biobags was more uniform and yellowish than with conventional bags without modifying other quality parameters, improving their sell value for the producers.

http://multibiosol.eu/
Biodegradable plastics for improving soil and fruit quality characteristics

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