Biodegradable plastics for improving soil and fruit quality characteristics

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Abstract

Current existing intensive farming practices require the use of large quantities of mulching film and fruit protection bags since they prevent the growth of weeds, regulate soil temperature, retain water and nutrients and protect crops from insects. For these farming practices, single-use conventional non-degradable polymers are used, creating a serious problem of waste management since it is time-consuming, expensive to recycle and, more importantly, is non-environmentally friendly contaminating the environment. By using biodegradable plastics, this problem could be solved by preventing the creation of waste. The objectives of our study were to produce an innovative biobased and biodegradable plastic film that entirely biodegrades in natural conditions on the field within a short time after their usage and to know it effects in tomato and peach crops. For this purpose, this film has been used to produce both biomulching to which specific oligo elements has been added to test in tomato crops and biobags to protect the fruits from insects and improve quality at harvest in peaches. Tests carried out on tomato showed that, these new bioplastics improved soil quality by increasing (up to 13%) the concentration of some oligo elements and by decreasing (65%) blossom end rot. In the case of peaches, by using biobags, a uniform colour (without red blush), which is a required characteristic in this type of commodity (Protected Designation of Origin 'Calanda') was obtained, with a decrease in a* colour coordinate (more than 2 points). Moreover, bioplastics only took 6 months to degrade completely within the soil.

Keywords: biobased film, mulching, fruit protection bag, tomato, peach, Life Multibiosol.

INTRODUCTION

For over a half a century farmers have been using plastic materials in agriculture because of their affordability and easiness to apply in the field. The main use of plastics in agriculture is for mulching, and in some Mediterranean areas, for fruit protection bags. The first ones prevent the growth of weeds, regulate soil temperature, and retain water and nutrients thus increasing yields (Kader et al., 2017). The second ones are single use agricultural bag used in tree crops since they can protect the fruit against the Mediterranean fly (*Ceratitis capitata*), the climatic incidences and chiefly the chemicals (Sharma et al., 2014).

The convenience of using this type of plastics has made the consumption of plastics grown rapidly in Europe (Mormile et al., 2007). The global market for agricultural plastic films, 4 million tonnes and approximately 10.6 million USD (2015), is projected to grow 5.6% per year through 2030 (Vitova, 2015). The total consumption in Europe exceeded 500.000 metric tons in 2013 being Spain and Italy the countries with highest consumption, due to their

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intensive horticulture activities. Together, they account for 40% of demand and consuming more than 120.000t per year (Plasteurope.com, 2017).

The main problem of agricultural films is that they have a lifespan of just one cultivation cycle, after which they need to be replaced, which is an intensive, expensive and time-consuming task (Malinconico et al., 2008). The conventional polymers used are non-degradable: LDPE (low density polyethylene) and HDPE (high density polyethylene). The use of this type of plastics create a serious problem of waste management since it is time-consuming, expensive to recycle and, more importantly, is non-environmental friendly. Furthermore, films are increasingly thinner and often end up being damaged during the cultivation process or they fall apart into smaller residues, which complicates recycling and clean up. Only approximately of 24% of mulching film in the European Union is recycled, while about 50% ends up in landfills and the rest is incinerated or abandoned in the fields (European Commission, 2014).

By using biodegradable plastics, these problems could be solved by preventing the creation of waste. Biodegradable plastics are those plastics in which the degradation results from the action of naturally occurring microorganisms such as bacteria, fungi, and algae. International research over the past few years has carried out many tests to compare soil and crop quality as well as harvest yields between biodegradable films and polyethylene film without showing differences (Kasirajan & Ngouajio, 2012, Steinmetz et al., 2016).

Our research focuses on the development of an innovative biobased and biodegradable plastic film that entirely biodegrades in natural conditions on the field within the period of biodegradation required by Regulation (EN 13432) in order to obtain the "OK Biodegradable SOIL" certification, that ensures that the plastic are entirely biodegradable and not phytotoxic for soil and plants. Furthermore, our study has contemplated the innovation of the addition of certain specific oligo elements (Mn, Zn and B) to the mulching films to study the effects in the biodegradability and quality of the crops. Additionally, with the use of the biodegradable bags, the objective is to obtain a homogenous colour and a high quality that increase the sale price of the peaches.

MATERIALS AND METHODS

Production of bioplastics

Biodegradable films were obtained by mixing selected natural biopolymers and additives in a conventional extrusion compounding process (Coperion ZSK26 co-rotating twin-screw extruder, semi-industrial) at AITIIP facilities (Zaragoza, Spain). The compounded materials were afterwards dried (Mini dryers Moretto X DRY AIR T) to ensure a low water level content that could negatively affect film properties. Finally, the blown extrusion machinery (LABTECH LBM 125, semi-industrial) equipped with a film module (Type LF-400-COEX) was used to obtain all biodegradable film products.

Mulching and fruit protection bags samples were based on Mater-Bi™ (corn thermoplastic starch, co-polyester, Novamont S.p.A.), Danimer™ (PHA/PLA, Meridian Holding Group) or BioPBS™ (bio-based polybutylene succinate, Japan Pulp & Paper GmbH). All the mulching films were 30-40 µm-thick and carbon black was used as a color additive using the masterbatch techniques. In addition, different percentages of oligoelements were added to the samples: Zn/Mn complex and Boron. Protection fruit bags were 40-50 µm-thick and white pigment (WP) was added as bleaching additive in the masterbatch processing. The different concentrations of ologoelements or bleaching agents have been coded as A for the lowest level and B for the highest level, due to the industrial secret that exists and that prevents to indicate the exact percentages. Conventional LDPE mulching (Comercial Arnedo, Spain) and conventional waxed paper bags (Cooperative Calanda DO) were used as control samples.

Mechanical properties

The modulus of elasticity (E) was determined using ISO 604 "Plastics – Determination of Compressive Properties". Elongation at break (ε) and tensile strength at break (σ) was determined by tensile testing in accordance with ASTM D 882 – 12 "Standard Test Method for Tensile Properties of Thin Plastic Sheeting".

Biodegradation tests

Biodegradation tests were carried out according to ASTM D 5988-12 "Standard Test Method for Determining Aerobic Biodegradation of Plastic Materials in Soil". The biodegradation tests were performed by using commercial soil, added with a compost (as inoculum), both produced by Gardea, in the best experimental conditions (50:50 inoculum/soil and 300 mg of cellulose as reference material).

Heavy metals concentration of the biofilms

The concentration of the heavy metals was quantified using the EPA 3052 1996 "Microwave assisted acid digestion of siliceous and organically based matrices" and EPA 6010C 2007 "Inductively coupled plasma-atomic emission spectrometry". The heavy metals are defined in the Standard EN 13432:2000 "Packaging: requirements for packaging recoverable through composting and biodegradation".

Vegetables and fruit samples

For mulching, tomato (*Solanum lycopersicum* 'Manitu') were manually planted (May 25, 2016; June 2, 2017) with a separation between plants of 50 cm and harvest at random in a commercial orchard located in the Mid-Ebro Valley (Zaragoza, Spain) at the time of optimum commercial harvest (August 25, 2016; August 31, 2017). For fruit protection bags in peaches (*Prunus persica* '58GC'), the bags were placed in a commercial orchard located in the Ebro Valley (Calanda, Spain) at random in the middle of the season (July 14, 2016; July 17, 2017) and fruits were harvest at optimum commercial harvest (September 13, 2016; September 6, 2017). Both crops were grown under drip irrigation and following the agronomic practices of the area. All samples were transferred immediately to the laboratory to carry out the fruit quality analysis.

Experimental design

Two crop seasons (2016 and 2017) were analyzed for both mulching and fruit protection bags. For mulching, in the first season were tested 3 different plastics (M11, M21 and M31), placed in the field with a separation of 1 m between lines, with two different concentration of oligoelements (Mn/Zn (codified as A for the lowest concentration and B for the highest)). In the second season, the sample with lower mechanical performance was discarded and it was added different amounts of oligoelements (two percentages of Mn/Zn and other two percentages of B (codified as A for the lowest concentration and B for the highest). Treatments were randomly distributed in three blocks, with six repetitions each block. For fruit protection bags, in 2016, three different bioplastic were assayed (B11, B21 and B31) with two levels of added white pigment (codified as A for the lowest concentration and B for the highest). 300 bags per batch were tested and randomly distributed in six blocks, with one tree each block. In the second season, the best performance plastic (B12) and a new one (B42) were tested with a reduction in the amount of pigment content (codified as A for the lowest concentration and B for the highest). 500 bags per batch were tested and randomly distributed in six blocks.

Soil collection and chemical analyses

Soil samples, only for mulching, were collected from the upper layer (20 cm) of the areas where the plastics were placed in to dates: before their colocation, and after 4 months of harvest and incorporation of the plastic into the soil using a tractor. One 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 as Hernández et al. (2016).

Quality parameters

To evaluate quality, destructive and non-destructive methods were used. 150 fruits per experimental unit were analyzed. Colour coordinates were determined using the 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 and by destructive Magness-Taylor using a digital penetrometer (Agrosta) with a tip diameter of 8 mm for peaches and of 4 mm for tomatoes and expressing the results as kg. 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). Ten grams of juice from 10 fruits were brought to 60 mL of distilled H_2O and titrated with 0.1 mol L^{-1} NaOH solution up to pH 8.1, expressing the results as g malic acid per kg.

Statistical analysis

All samples were analysed at least in triplicate per year. Statistical analyses were performed using a one-way ANOVA test and the significance of the difference between means was determined by Duncan's multiple range test (p<0.05). Statistical analysis was performed using the Statistical Package for the Social Science (SPSS) software version 23.0.

RESULTS AND DISCUSSION

The mechanical properties of bioplastics for mulching are showed in the Table 1. In some cases oligoelements made more difficult the plastic processing, and consequently, was necessary to increase the thickness. Sample M11 is much more elastic (ε=552-615%) than the other ones tested, meanwhile mulching sample M31 was difficult to process due their low value of ε, ranging from 62% with oligoelements to 154% without. Moreover, σ is higher in biobased samples than in control sample and decrease with oligoelements in all cases. In the second year, M12 was the best bioplastic. For all of this, in general, M11 and M12 without oligoelements showed the best mechanical properties due to the high values of E, σ and ε.

For fruit protection bags the values obtained were also appropriated for their agriculture use (data not showed). It is remarkable that σ and ε was higher for the bags than for the mulching films, meaning loading the biodegradable matrix with carbon black and oligoelements made samples have lower elongation and tensile strength at break values than those with WP. In addition, thickness was more variable, possibly due to processing problems in adjusting parameters for film blowing.

Table 1. Quality parameters of mulching plastic films[1]

Year	BATCH Material	BATCH Oligoelement Level	Thickness (µm)	E (Mpa)	σ (Mpa)	ε (%)
2016	M11	M11A	20 (0)	183 (69)	24.3 (2)	552 (194)
		M11B	38 (4.1)	55 (10)	8.3 (2)	615 (117)
	M21	M21A	20 (0)	166 (35)	6 (3)	235 (118)
		M21B	30 (0)	108 (29)	5.4 (1)	214 (73)
	M31	M31A	30 (0)	245 (35)	17.3 (3)	154 (10)
		M31B	30 (0)	127 (25)	12 (4)	62 (54)
	Control (LDPE)	-	42 (0.8)	300 (14)	4.5 (1)	600 (20)
2017	M12	M12A	31 (1.5)	190 (55)	25.5 (1.8)	430 (90)
		M12B	33 (1.2)	160 (63)	22.1 (2.2)	583 (129)
	M42	M42A	51 (4.9)	137 (60)	6.4 (2.9)	247 (88)
		M42B	40 (3.3)	122 (55)	4.9 (3.3)	226 (61)
	Control (LDPE)	-	12 (2.6)	187 (20)	26 (3.8)	280 (39)

[1]The values between parentheses are the standard deviation

The concentration of the heavy metals has been quantified to verify the compliance with the limits defined in the Standard EN 13432 for compostable packaging. The regulation limits in mg kg^{-1} (dry mass) the quantity of certain heavy metals (Arsenic, Cadmium, Chromium, Mercury, Molybdenum, Nickel, Lead, Copper, Selenium and Zinc). The results showed that all metals were below the detection limits of the technique with the exception of the Zinc element (Table 2). In 2016, those mulching samples that were additivated with the high concentration of Zn, presented a value above the regulation limit of 150 mg kg^{-1}. The amount of oligoelement added to the mulching was calculated for fertilization, but in case the plastics were to be labelled as "OK biodegradable SOIL" then the percentage has to be lower. Therefore, in 2017 the concentration of this element in the plastic was reduced (data not showed), but another time, the values were over 150 mg kg^{-1} even with the low amount. For fruit protection bags, all samples are below the allowed limit (data not showed).

Table 2. Heavy metals (mg kg^{-1} dm) in mulching samples (2016)

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

Regarding biodegradation behaviour, with the soil selected for the tests, high percentages of biodegradability in soil were achieved. In 2016 (Table 3), 98-100% of biodegradation was observed after 176 days. These values are very appropriated taking into account that the "OK biodegradable SOIL" certification forces to obtain a degradation of more than 90% in two years.

Table 3. Percentage of biodegradation in plastic matrices in 2016[1]

Time	N04 (0%)	M21 (0%)	P91 (0%)	N05 (5%)
After 143 days	98.8 (0.28)	79.6 (3.5)	76.4 (4.1)	80.6 (8.3)
After 176 days	99.9 (0.0)	99.2 (0.85)	97.9 (1.8)	98.5 (1.3)

[1]The values between parentheses are the standard deviation

For the soil analysis, only the results of the 2016 season after the first incorporation of the bioplastics into the soil are available at this moment (Table 4). An increase in the concentration of Mn and Zn was observed using the bioplastics with the highest concentration of this elements. This result shows that the oligolements are present in 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, being more interesting using bioplastics added with oligoelements.

Table 4. Concentrations of nutrients and oligoelements in the soil[1]

Time	BATCH		N	P	K	Mn	Zn	C/N
	Material	Oligoelement Level	(g $100g^{-1}$)	(g $100g^{-1}$)	(g $100g^{-1}$)	(mg kg^{-1})	(mg kg^{-1})	
Beginning of 2016 season	-	-	0.15	0.10 a	1.18 a	369.4 bc	71.2 b	34.68 a
Beginning of 2017 season and after 4 months of incorporation of the bioplastics into the soil	M11	M11A	0.16	0,10 a	1,33 bc	345,66 a	68,73 a	28,25 bc
		M11B	0.17	0,15 b	1,31 bc	363,96 b	72,16 bc	23,01 c
	M21	M21A	0.16	0,09 a	1,21 b	347,83 a	67,24 a	32,44 ab
		M21B	0.16	0,11 ab	1,32 bc	378,14 bc	73,24 bc	28,16 bc
	M31	M31A	0.17	0,10 a	1,28 bc	354,12ba	71,92 ab	25,98 c
		M31B	0.15	0,11 ab	1,47 c	383,94 c	75,49 c	30,96 b
	Control	-	0.16	0,09 a	1,23 b	342,34 a	67,03 a	31,11 b

[1]different letters in the same column indicate significant differences ($p \leq 0.05$) between treatments

Focusing now on the quality parameters of the crops, in 2016 season, although significant differences were observed, there was no clear pattern in the use of different plastics for tomatoes (Table 5). Therefore, the differences were due more to the intrinsic variability of the sample than to the effect of the plastics on the crop. In 2017 season, no differences were observed, showing that the plastics did not have effect in these quality parameters. Only 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 or the different reflected sunlight, but it is necessary more assays to confirm this hypothesis. Other tests carried out on tomatoes and peppers 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 (Martín-Closas et al. 2008; Cowan, 2013).

Table 5. Quality parameters in 'Manitu' tomato at harvest[1]

Year	BATCH		Firmness (kg)	Durofel	Weight (g)	SSC (ºBrix)	a* (D65)	Blossom end rot (%)
	Material	Oligoelement Level						
2016	M11	M11A	0.32 a	65.04 a	102.11ab	6.73 c	32.76 ab	<1
		M11B	0.39 d	68.18 b	107.84abc	6.27 ab	34.17 c	<1
	M21	M21A	0.38 cd	70.26 b	97.97a	6.60 bc	31.69 a	<1
		M21B	0.38 bcd	68.90 b	105.5abc	6.23 ab	33.12 bc	<1
	M31	M31A	0.39 cd	63.62 a	113.42c	5.93 a	31.99 ab	<1
		M31B	0.34 abc	68.76 b	102.54ab	6.70 c	32.75 ab	<1
	Control	-	0.33 ab	69.02 b	110.42bc	6.73 c	33.22 bc	<1
2017	M12	M12A	0.44 bc	68.06	143.75	6.47	32.24	7 a
		M12B	0.48 c	70.72	140.33	6.3	32.51	7 a
	M42	M42A	0.43 bc	69.17	146.99	6.5	32.48	8 a
		M42B	0.37 a	69.21	128.58	6.53	31.23	8 a
	Control	-	0.41 ab	70.88	141.48	6.33	32.04	18 b

[1]different letters in the same column indicate significant differences (*p*≤0.05) between treatments for the same year.

In the fabrication of the fruit protection biobags we are looking for, on the one hand, fruits with a homogeneous colour and, on the other hand, quality parameters not affected by the biobags. For all of this, WP was incorporated to help crops against UV rays and laser micro-perforations were done in the bottom of the bag to allow the necessary elimination of water vapour created during fruit ripening on trees. The use of this type of biobags not affected the quality parameters of the peaches except for the colour (Table 6). The use of biobags with WP generated a lower red coloration in the fruit and resulted in a lower coordinate a* value (from 14.88 to 16.05 without WP and from 12.39 to 14.49 with WP) and a more homogeneous orange colour. These values were also lower than the control ones (16.40 and 15.37 for 2016 and 2017 season, respectively). The differences observed in the rest of parameters may be due to intrinsic differences in crops more than effect of the bioplastics.

Table 6. Quality parameters in '58GC' peach at harvest[1]

Year	BATCH		Firmness (kg)	Aweta	Weight (g)	T.A. (g.malic L^{-1})	SSC (ºBrix)	a* (D65)
	Material	WP						
2016	B11	B11A	3.19	10.03	220.83	5.64 ab	14.03 c	14.88 bc
		B11B	3.23	9.55	235.17	5.85 b	14.58 c	14.49 ab
	B21	B21A	3.24	12.79	232.27	5.14 a	12.93 ab	15.76 bcd
		B21B	3.03	13.23	230.00	5.14 a	12.35 a	13.34 a
	B31	B31A	3.20	8.81	207.53	6.02 b	14.70 c	16.05 cd
		B31B	3.23	12.87	221.30	5.51 ab	13.08 b	13.16 a
	Control	-	3.21	15.09	233.77	5.18 a	12.78 ab	16.40 d
2017	B12	B12A	2.56 b	9.32 a	215.46	6.75 a	13.43	15.45 c
		B12B	2.21 ab	9.64 ab	221.92	6.87 a	13	14.14 b
	B42	B42A	2.06 a	11.63 abc	211.58	7.26 a	12.63	15.62 c
		B42B	3.62 c	13.9 c	233.19	7.77 a	12.6	12.39 a
	Control	-	2.11 ab	11.88 bc	217.69	9.38 b	13.13	15.37 c

[1]different letters in the same column indicate significant differences (*p*≤0.05) between treatments for the same year.

CONCLUSIONS

- In general, the biomulching and biobags films showed proper mechanical properties. The addition of oligoelements or white pigment made more difficult the plastic processing, and consequently, was necessary to increase the thickness.
- A high soil biodegradation was observed in all the bioplastics.
- The addition of Zn is not proper to obtain the "OK biodegradable SOIL" certification in biomulching films.
- The soil biomulching degradation increased the concentration of Mn and Zn.
- The use of biomulching in tomatoes decreased the incidence of blossom end rot and not affected the rest of quality parameters.
- For peaches, the colour was more uniform using biobags than the control ones, which is a good characteristic for the producers.

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