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# Production and Tensile Characterization of Thermoplastic Starch Films filled with Iron Scrap Powder Waste and Molded on Different Support Materials

Danilo Battistelli<sup>1</sup>, Carlo Santulli<sup>2\*</sup>

(Telephone number: +39-380-6522232; e-mail: carlo.santulli@unicam.it)

<sup>1,2</sup>Università di Camerino, School of Architecture and Design, viale della Rimembranza 9, 63100 Ascoli Piceno, Italy

## Authors' contributions:

This work was carried out in collaboration among all authors Author DB designed the study and suggested the protocol. Author CS commented on experiment results and contributed to literature search. Both authors wrote the first draft of the paper and read and approved the final manuscript.

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## ABSTRACT

This work concerns the application of waste filler, consisting of scrap iron powder as received from machining in a small-scale workshop, in a self-produced thermoplastic starch (TPS) based on corn starch acidified with acetic acid and plasticized with glycerol. The films obtained had a target thickness of 250 microns. The maximum amount of waste introduced was 0.8% and the material was produced on different supports, consisting either of a glass plate or of a silicone mould.

Tensile testing was performed and the best performance was obtained by the one prepared on glass support, although in general terms it was very far from similar industrial material, not exceeding a 10% maximum strain and being very sensitive to the disposition and geometry of the waste introduced. The value of the work is in the use of waste, which is rarely re-used, and in the possible production of conductive and magnetic biopolymer films

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*Keywords: Iron scrap waste; starch **bio-composites**; DIY bioplastics; tensile characterization*

## 1. INTRODUCTION

Scrap iron is an abundant waste on blacksmith operations, which have found some application to treat some other waste streams containing hazardous materials, for example in the case of the reduction of hexavalent chromium to trivalent one [1]. Large steel works have from a few decades policies leading more recently to “zero waste strategies” [2]. On the other side, the use of scrap iron powder as filler in materials encounters some difficulties in normal working of small blacksmith workshops located e.g., in family enterprises, technical school, etc., where scrap iron powder have to be disposed in special waste collection, which may involve some costs. In particular, reuse as filler may encounter some difficulties for the dimensional scattering of the powder obtained, which can go from around 20 microns to over 500 microns, and also the presence of some impurity, such as dust **and cut-outs**.

A possibility would be to exploit the electrical conductivity and magnetic properties of this waste material to offer them to other materials, which typically have limited conductivity,

\* Tel.: +xx xx 265xxxxx; fax: +xx aa 462xxxxx.  
E-mail address: xyz@abc.com.

39 such as biopolymers. The production of the so-called DIY (Do-It-Yourself) bioplastics,  
40 directly developed in the laboratory at a small scale, based mainly on starch-glycerol  
41 mixtures and therefore identifiable as “thermoplastic starches” (TPS) may offer an  
42 opportunity in this sense, since TPS are adapted to the introduction of fillers in powder form,  
43 such as clay, even with limited control of their dimensions [3]. In a number of cases, these  
44 were able to effectively include waste, mostly from the food-production sector [4-5]. The  
45 considerable cost of conductive polymer structures, obtained normally e.g., through  
46 appropriate doping [6], or else by the introduction of carbon nanofibers [7], does suggest that  
47 the use of waste filler could be an option. In the case of starch-based bioplastics, which have  
48 a, though limited conductivity, yet normally no magnetic properties, this appears particularly  
49 reasonable [8].

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51 In this work, some experiments have been performed by introducing unfiltered iron scrap  
52 powder in the production of a thermoplastic starch, based on corn starch, glycerol and acetic  
53 acid using three slightly different procedures. The materials have been subjected to tensile  
54 tests to compare the three procedures, and to suggest which can be the most suitable and  
55 which improvements can be possibly applied in future studies leading to the development of  
56 a material film for possible application where electrical conductivity is desirable. In particular,  
57 an opportunity envisaged for the prospective material would be its application in the chassis  
58 of cell phones as a conductive yet biodegradable, hence sustainable, material [9]. Other  
59 possibilities would be for example in the production of small magnets as gadgets, etc.  
60 However, so far magnetization of bioplastics has only been proposed with complexes based  
61 on iron, which do not fit the purpose of low cost application, for which a market would be  
62 available though [10]. To conclude, it appears that this material obtained from waste could  
63 be a solution for these low profile uses, although it would need for a start a first  
64 characterization to set-up a proper and effective fabrication method.

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## 66 2. MATERIAL AND METHODS

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### 68 2.1 Production of the material

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70 To a self-produced thermoplastic starch (TPS) mixture, which included 84% water, 7.7%  
71 corn starch, 7.7% glycerol and 0.6% acetic acid, an amount of untreated iron scrap waste  
72 was added of either 0.4 or 0.8%, putting the TPS as equal to 100%.

73 The ingredients were placed in a container and mixed with care in order to amalgamate the  
74 starch component and prevent the formation of lumps. When the compound appears well  
75 amalgamated, cook it mixing constantly on a low fire, taking care that gelification  
76 temperature is reached and not exceeded. This occurs when the compound starts becoming  
77 dense and similar to a gel. After this, the TPS can be uniformly poured on a flat support and  
78 flattened using a roller to a 250 microns film in order to include in the thickness even the  
79 largest iron scrap particles. The thickness was accurate up to a  $\pm 20$  microns. It needs to be  
80 worked for 30 seconds, and then dried for a time of 1-3 days at ambient temperature.

81 Iron scrap waste was received, as shown in Figure 1, and then some extraneous waste,  
82 such as dust and small elongated off-cuts, which are equally visible in the image, were  
83 manually removed. However, any further treatment (e.g., magnetic) for purification was  
84 considered out of the scope of this preliminary study.

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86 Two different supports were selected for production, one of which was a glass plate of 5 mm  
87 thickness and the other a silicon mould of 2 mm thickness. On the glass plate a TPS loaded  
88 with 0.8% iron scrap waste was produced, hereinafter referred to as “glass”, whereas on the  
89 silicon mould a TPS loaded with either 0.4% (MAT1) or 0.8% iron scrap waste (MAT2) was  
90 produced.

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**FIG. 1 Scrap iron material as received**

## 2.2 Tensile tests

Tensile tests have been carried out on the three materials produced, preparing a minimum number of five samples for each of them, cut using scissors from a material plate, with 250 mm length and 25 mm width. Elongation was measured over 150 mm length using an extensometer. As the result, one of the ends was kept loose, as depicted in **Figure 2**. This was done to avoid fracture at the end of the samples, to ensure that the tests were valid, after some attempts that proved this problem was present. A Zwick Roell Z005 universal screw-driven tensile machine was used with a maximum load of 2.5 kN, fitted with a 5 N load cell and with pneumatic grips. Tests were carried out according to the ISO527 standard. In particular, a pre-load of 0.05 N was applied and the velocity applied for the measurement of the tensile modulus was 1 mm/minute, while the general test speed was equal to 10 mm/minute.



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**FIG. 2 Image of the set-up for tensile testing (one of the “glass samples”)**

### **3. RESULTS AND DISCUSSION**

#### **3.1 Results**

The three types of samples produced led to different results, in the sense that those, MAT 1 and MAT 2, obtained using a silicone support, did not come out mainly flat, yet with considerable lumps and with pronounced aggregation of the scrap iron particles, as it is

117 shown in **Figure 3**. This led, when a larger amount of waste is introduced, hence in MAT 2,  
118 to some kind of detachment in a part of the composite plate obtained, which becomes partly  
119 non usable for testing. On the other side though, the detachment of the sample after  
120 production is easier with silicone than from the glass support, possibly due to the fact that  
121 some moisture from the film tends to spread out on the glass plate.

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123 As regards tensile testing, the image in **Figure 4** clarifies how the samples typically break,  
124 the fracture passes through the film in a quite fragile mode, only being deviated by the  
125 possible presence of larger iron particles. Necking was also tentatively measured to correct  
126 the values of Young's modulus, though with some inaccuracy, due to the fact that the  
127 samples were not possibly gripped at both ends, but only at one of them. In terms of sample  
128 width, necking brought to its reduction in the order of around 10%.

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130 Passing to tensile curves obtained from all films, it can be observed first qualitatively from  
131 **Figure 5** that in all cases, the results yielded by the tests were not much dispersed, the main  
132 issue being given by some variation of the strain, which led to discarding some results from  
133 the following evaluation. Only those that are closer to the general trend were considered in  
134 the evaluation, in particular five per series of samples. This variation of strain was attributed  
135 to the presence of larger iron particles, which were able to impede necking of the sample,  
136 hence producing early fracture.

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138 The results obtained are summarized in Table 1. It can be noticed that the maximum stress  
139 remains about constant with the three types of samples and the presence of 0.4% (MAT1) or  
140 0.8% of scrap iron waste does not produce any particular difference in the strength of the  
141 films. The samples produced on the silicone support with 0.4% of scrap iron waste have a  
142 higher elongation, confirming the previous assumption that early fracture may be due to the  
143 presence of iron particles along the crack propagation path during the pulling process of the  
144 sample. In contrast, those, **which were produced using the glass support**, appear  
145 considerably more rigid, as from Young's modulus values.

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### 147 **3.2 Discussion**

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149 To compare the results obtained, literature on TPS has to be considered: it can be easily  
150 considered that the tensile strength obtained is largely inferior to what has been obtained  
151 with industrial products, for example in [11] values of maximum tensile stress in the order of  
152 47 to 59 MPa were measured, the latter being obtained by using sorbitol instead of glycerol  
153 as plasticizer. Another important remark is that it would be possible to increase the amount  
154 of filler used, since as such starch-based plastics are able to contain effectively very large  
155 quantities of waste filler, of course depending on its geometry [12]. This is quite routinely  
156 done using nanofillers [13], however with microfillers other considerations need to be done,  
157 which are developed here below.

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159 This has to be considered as a preliminary work. For this reason, some considerations can  
160 be done to continue along the way towards producing a material suitable for application. The  
161 first important question is related to filtering the particles, in a way that only those that are  
162 considerably smaller than the film thickness are retained in the material. Other aspects  
163 would concern the need to analyze which other materials are present in the scrap iron  
164 powder that is specifically used for production. This is scarcely investigated on the small  
165 scale, although studies are present, which concern cast iron waste from machining on the  
166 large scale, which can be of reference [14]. A possible suggestion in this sense can be  
167 applying magnetic selection, to exclude non-metallic materials from the filler. Another  
168 indication, which can be given, is that the scrap iron, used as-received, could include rusty

169 materials: these can be made more suitable for use by removing rust for example by a  
170 process of pickling, which is normally used e.g., to prepare pigments from iron waste [15].  
171  
172 All the above considerations need to be integrated in further studies that will need to involve  
173 further characterization, such as thermal, especially in view of the scarce resistance of  
174 bioplastics to temperature and **micro-structural** analysis, to investigate the interface between  
175 filler particles and the host material. In addition, of course the measurement of electrical  
176 conductivity and magnetic properties would need to be carried out.  
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GLASS

100  $\mu\text{m}$



MAT 1

100  $\mu\text{m}$



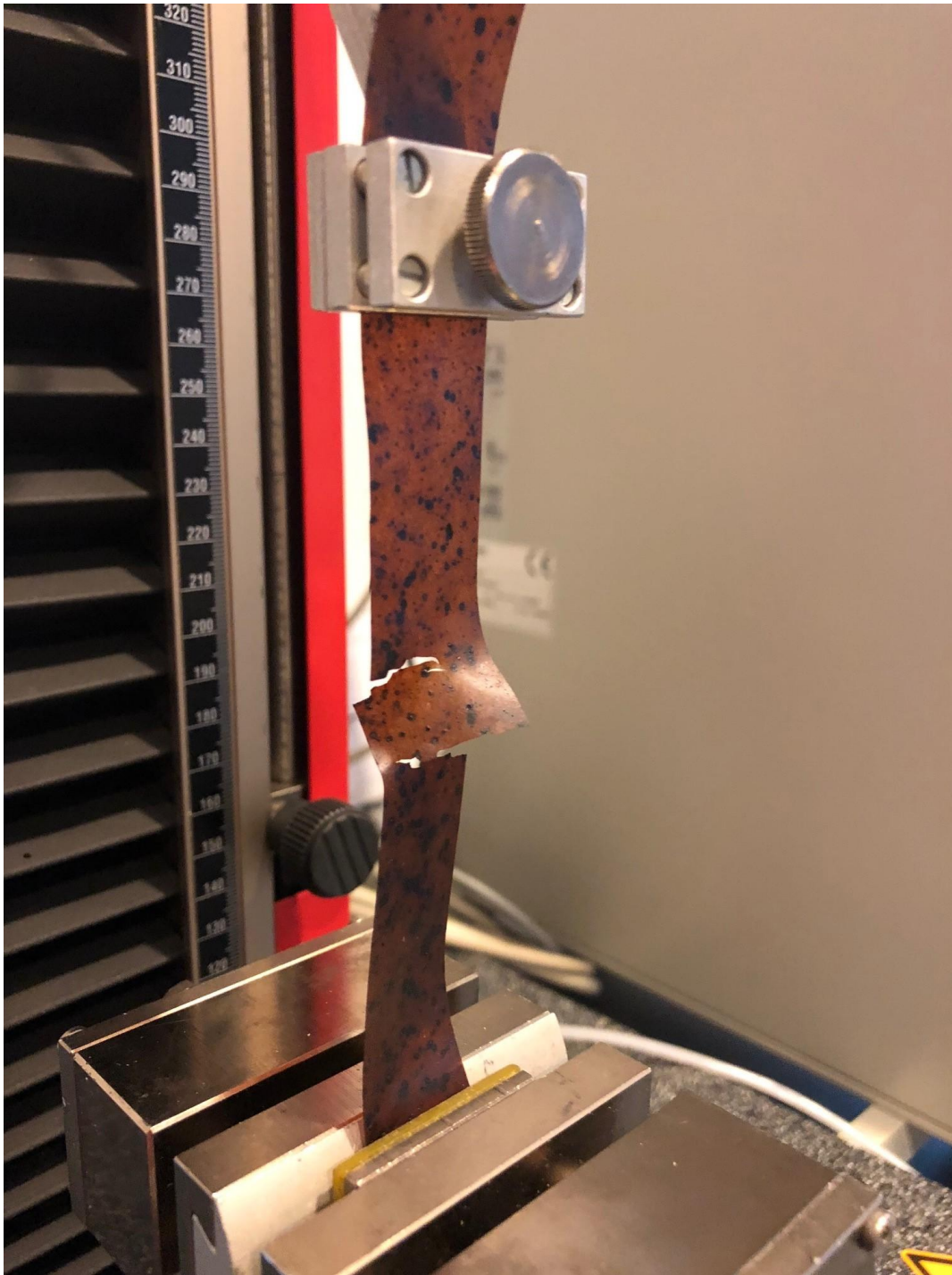
MAT 2

100  $\mu\text{m}$

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**FIG. 3 Images of the three types of samples produced**

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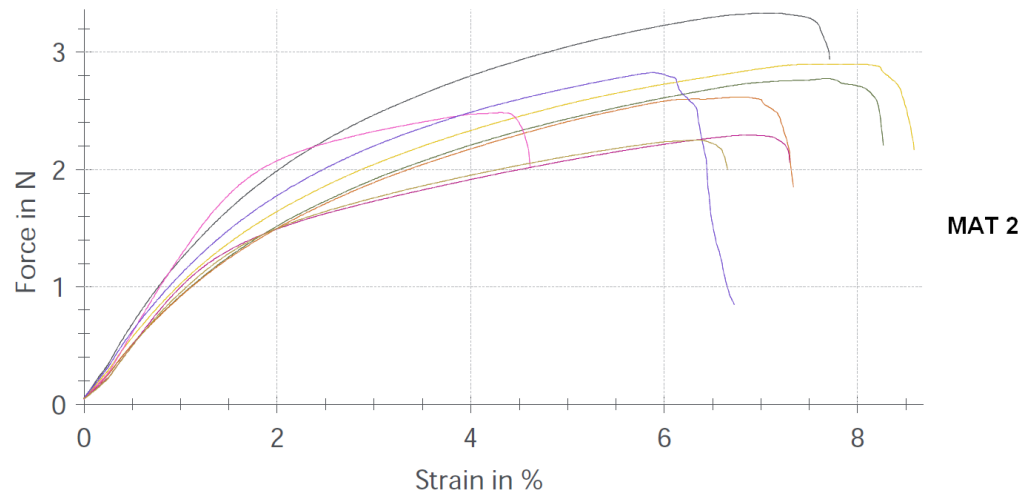
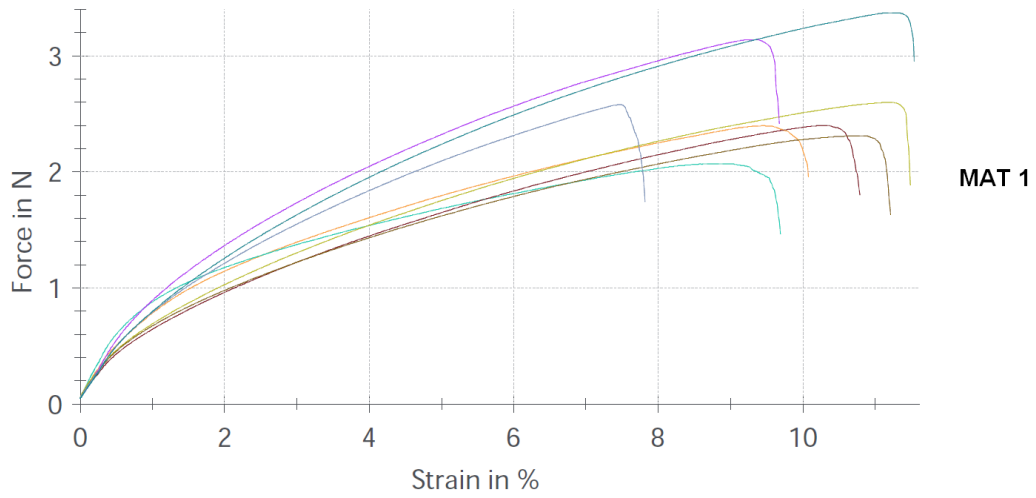
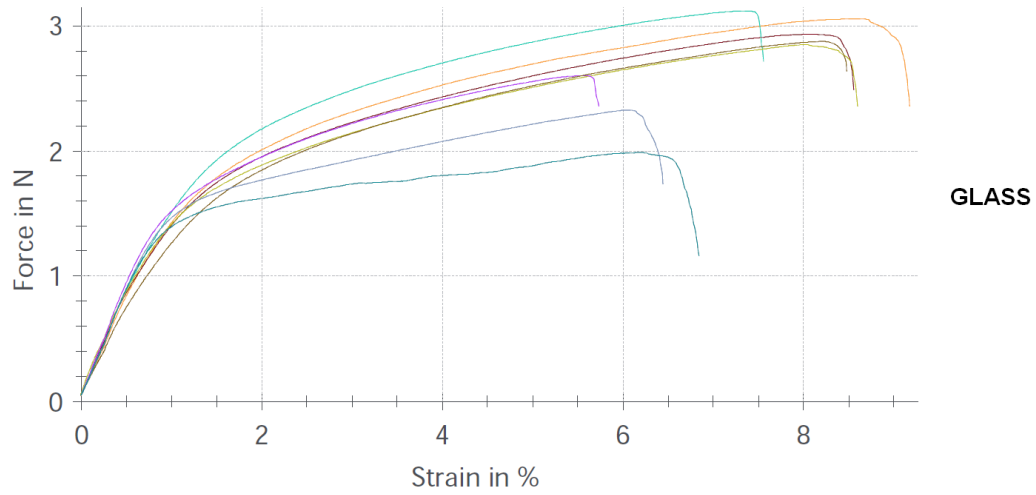


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**FIG. 4 Image of a tensile sample during breakage (one of the “glass samples”)**

\* Tel.: +xx xx 265xxxxx; fax: +xx aa 462xxxxx.  
E-mail address: xyz@abc.com.





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**FIG. 5** Tensile tests curves for the materials produced with the three supports

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**Table 1 Tensile tests results**

<b>Samples</b>	<b>Max. stress (MPa)</b>	<b>Max. strain (%)</b>	<b>Young's modulus (MPa)</b>
GLASS	0.544 ± 0.078	7.2 ± 1.2	32.2 ± 2.6
MAT1	0.522 ± 0.087	9.8 ± 1.3	18.9 ± 2.5
MAT2	0.537 ± 0.071	7.1 ± 1.2	18 ± 3.2

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#### **4. CONCLUSIONS**

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The study concentrated on the possibility to produce biopolymers including scrap iron waste powder as received. Three types of possible production were attempted using either a glass plate with 0.8% waste filler or silicon flat mould with either 0.4 or 0.8% waste filler. The production with glass support proved more reliable, although all of the methods supplied a sufficient number of tensile samples with close properties to validate the tests. Concerns can be raised about the dimensions of the filler particles which need to be filtered and/or ground for better properties and about the possible presence of rust, which further reduces the mechanical performance especially in terms of elongation. However, the tests are considered successful as preliminary experiments in order to lead to further refinement of the production process and subsequent characterization.

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#### **COMPETING INTERESTS**

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Authors have declared that no competing interests exist.

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