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4 **Production and Tensile Characterization of**
5 **Thermoplastic Starch Films filled with Iron**
6 **Scrap Powder Waste and Molded on Different**
7 **Support Materials**

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11 **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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13 *Keywords: Iron scrap waste; starch biocomposites; DIY bioplastics; tensile characterization*

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15 **1. INTRODUCTION**

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17 Scrap iron is an abundant waste on blacksmith operations, which have found some
18 application to treat some other waste streams containing hazardous materials, for example
19 in the case of the reduction of hexavalent chromium to trivalent one [1]. Large steel works
20 have from a few decades policies leading more recently to “zero waste strategies” [2]. On
21 the other side, the use of scrap iron powder as filler in materials encounters some difficulties
22 in normal working of small blacksmith workshops located e.g., in family enterprises, technical
23 school, etc., where scrap iron powder have to be disposed in special waste collection, which
24 may involve some costs. In particular, reuse as filler may encounter some difficulties for the
25 dimensional scattering of the powder obtained, which can go from around 20 microns to over
26 500 microns, and also the presence of some impurity, such as dust.

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28 A possibility would be to exploit the electrical conductivity and magnetic properties of this
29 waste material to offer them to other materials, which typically have limited conductivity,
30 such as biopolymers. The production of the so-called DIY bioplastics, based mainly on
31 starch-glycerol mixtures and therefore identifiable as “thermoplastic starches” (TPS) may
32 offer an opportunity in this sense, since TPS are adapted to the introduction of fillers in
33 powder form, such as clay, even with limited control of their dimensions [3]. In a number of
34 cases, these were able to effectively include waste, mostly from the food-production sector
35 [4-5]. The considerable cost of conductive polymer structures, obtained normally e.g.,
36 through appropriate doping [6], or else by the introduction of carbon nanofibres [7], does
37 suggest that the use of waste filler could be an option. In the case of starch-based

38 bioplastics, which have a, though limited conductivity, yet normally no magnetic properties,
39 this appears particularly reasonable [8].

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

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56 **2. MATERIAL AND METHODS**

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58 **2.1 Production of the material**

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

63 The ingredients were placed in a container and mixed with care in order to amalgamate the
64 starch component and prevent the formation of lumps. When the compound appears well
65 amalgamated, cook it mixing constantly on a low fire, taking care that gelification
66 temperature is reached and not exceeded. This occurs when the compound starts becoming
67 dense and similar to a gel. After this, the TPS can be uniformly poured on a flat support and
68 flattened using a roller to a 250 microns film in order to include in the thickness even the
69 largest iron scrap particles. The thickness was accurate up to a ± 20 microns.

70 It needs to be worked for 30 seconds, and then dried for a time of 1-3 days at ambient
71 temperature.

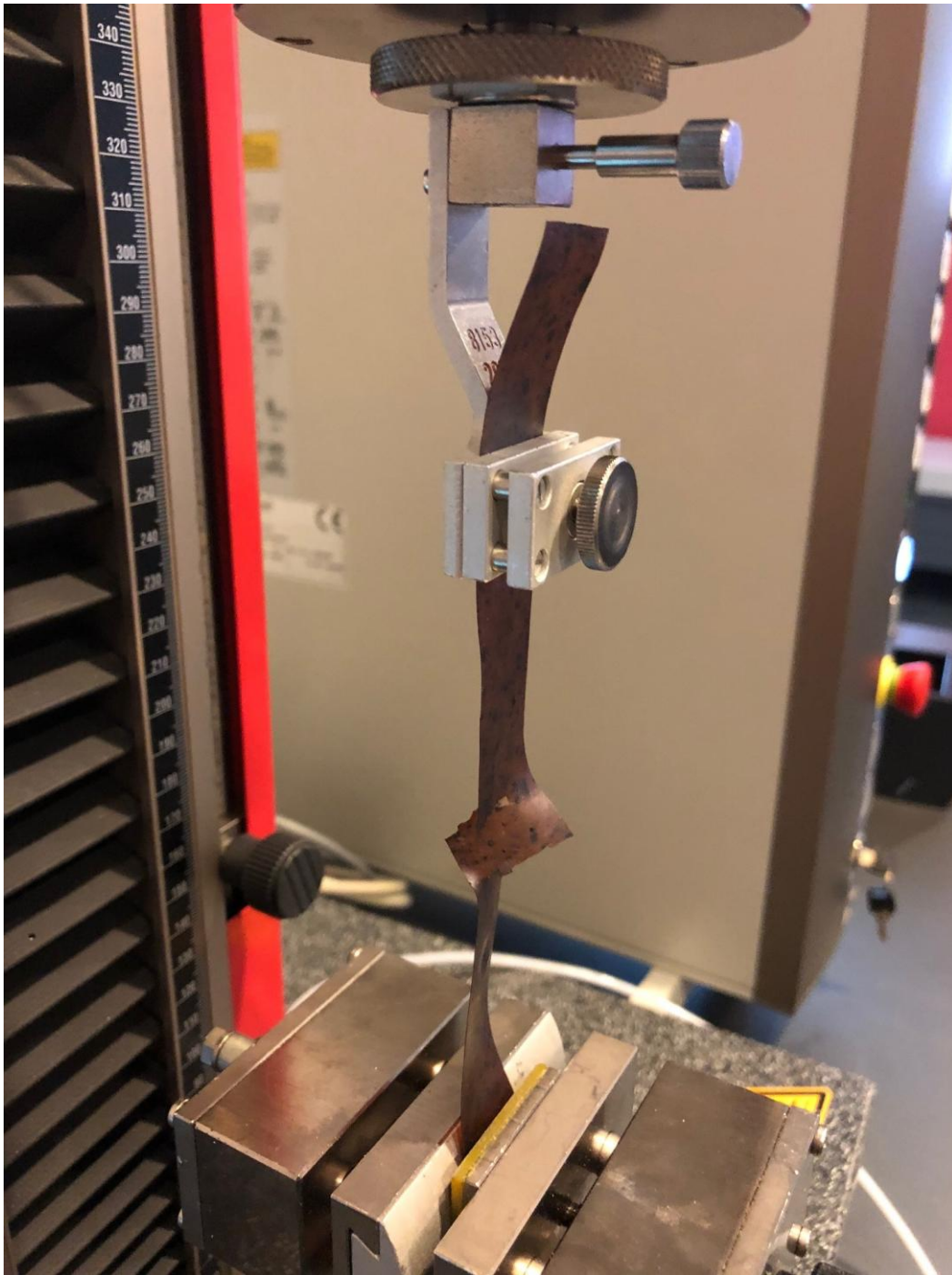
72 Two different supports were selected for production, one of which was a glass plate of 5 mm
73 thickness and the other a silicon mould of 2 mm thickness. On the glass plate a TPS loaded
74 with 0.8% iron scrap waste was produced, hereinafter referred to as "glass", whereas on the
75 silicon mould a TPS loaded with either 0.4% (MAT1) or 0.8% iron scrap waste (MAT2) was
76 produced.

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78 **2.2 Tensile tests**

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



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Fig. 1 Image of the set-up for tensile testing

94 **3. RESULTS AND DISCUSSION**

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96 **3.1 Results**

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98 The three types of samples produced led to different results, in the sense that those, MAT 1
99 and MAT 2, obtained using a silicone support, did not come out mainly flat, yet with
100 considerable lumps and with pronounced aggregation of the scrap iron particles, as it is
101 shown in Figure 2. This led, when a larger amount of waste is introduced, hence in MAT 2,
102 to some kind of detachment in a part of the composite plate obtained, which becomes partly
103 non usable for testing. On the other side though, the detachment of the sample after
104 production is easier with silicone than from the glass support, possibly due to the fact that
105 some moisture from the film tends to spread out on the glass plate.

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107 As regards tensile testing, the image in Figure 3 clarifies how the samples typically break,
108 the fracture passes through the film in a quite fragile mode, only being deviated by the
109 possible presence of larger iron particles. Necking was also tentatively measured to correct
110 the values of Young's modulus, though with some inaccuracy, due to the fact that the
111 samples were not possibly gripped at both ends, but only at one of them. In terms of sample
112 width, necking brought to its reduction in the order of around 10%.

113

114 Passing to tensile curves obtained from all films, it can be observed first qualitatively from
115 Figure 4 that in all cases, the results yielded by the tests were not much dispersed, the main
116 issue being given by some variation of the strain, which led to discarding some results from
117 the following evaluation. Only those that are closer to the general trend were considered in
118 the evaluation, in particular five per series of samples. This variation of strain was attributed
119 to the presence of larger iron particles, which were able to impede necking of the sample,
120 hence producing early fracture.

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122 The results obtained are summarized in Table 1. It can be noticed that the maximum stress
123 remains about constant with the three types of samples and the presence of 0.4% (MAT1) or
124 0.8% of scrap iron waste does not produce any particular difference in the strength of the
125 films. The samples produced on the silicone support with 0.4% of scrap iron waste have a
126 higher elongation, confirming the previous assumption that early fracture may be due to the
127 presence of iron particles along the crack propagation path during the pulling process of the
128 sample. In contrast, those produced using the glass support appear considerably more rigid,
129 as from Young's modulus values.

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131 **3.2 Discussion**

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133 To compare the results obtained, literature on TPS has to be considered: it can be easily
134 considered that the tensile strength obtained is largely inferior to what has been obtained
135 with industrial products, for example in [11] values of maximum tensile stress in the order of
136 47 to 59 MPa were measured, the latter being obtained by using sorbitol instead of glycerol
137 as plasticizer. Another important remark is that it would be possible to increase the amount
138 of filler used, since as such starch-based plastics are able to contain effectively very large
139 quantities of waste filler, of course depending on its geometry [12]. This is quite routinely
140 done using nanofillers [13], however with microfillers other considerations need to be done,
141 which are developed here below.

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143 This has to be considered as a preliminary work. For this reason, some considerations can
144 be done to continue along the way towards producing a material suitable for application. The
145 first important question is related to filtering the particles, in a way that only those that are
146 considerably smaller than the film thickness are retained in the material. Other aspects

147 would concern the need to analyze which other materials are present in the scrap iron
148 powder that is specifically used for production. This is scarcely investigated on the small
149 scale, although studies are present, which concern cast iron waste from machining on the
150 large scale, which can be of reference [14]. A possible suggestion in this sense can be
151 applying magnetic selection, to exclude non-metallic materials from the filler. Another
152 indication, which can be given, is that the scrap iron, used as-received, could include rusty
153 materials: these can be made more suitable for use by removing rust for example by a
154 process of pickling, which is normally used e.g., to prepare pigments from iron waste [15].

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156 All the above considerations need to be integrated in further studies that will need to involve
157 further characterization, such as thermal, especially in view of the scarce resistance of
158 bioplastics to temperature and microstructural analysis, to investigate the interface between
159 filler particles and the host material. In addition, of course the measurement of electrical
160 conductivity and magnetic properties would need to be carried out.

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GLASS

100 μm



MAT 1

100 μm



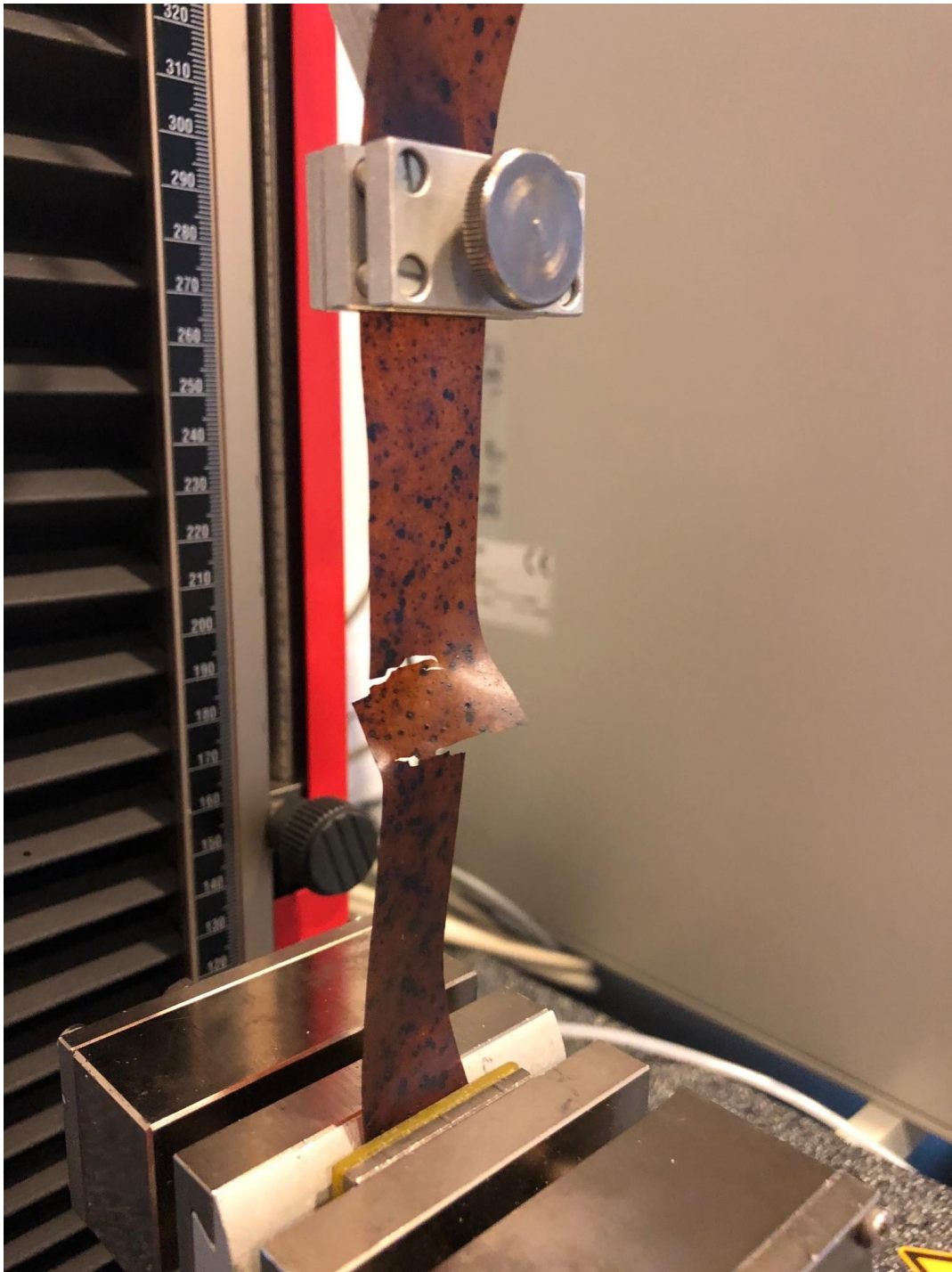
MAT 2

100 μm

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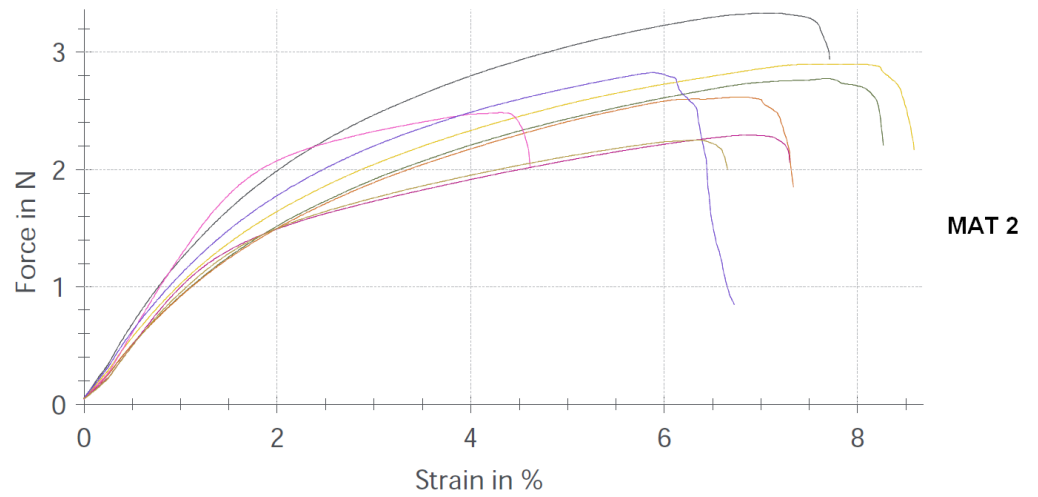
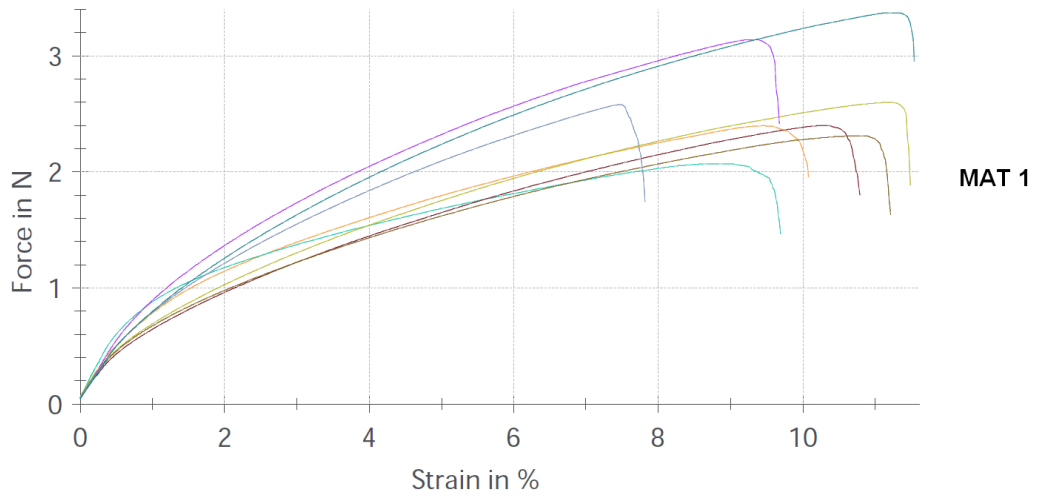
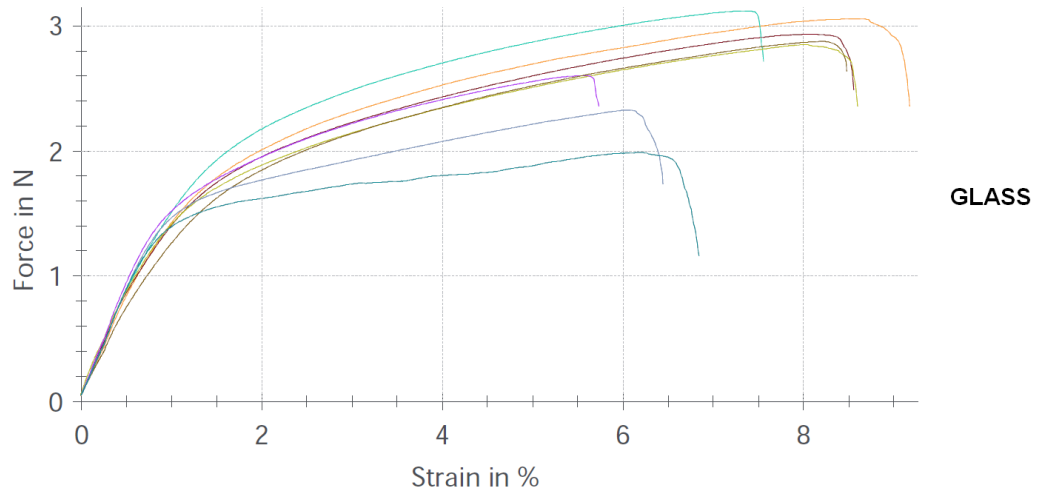
Fig. 2 Images of the three types of samples produced

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Fig. 3 Image of a tensile sample during breakage



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Fig. 4 Tensile tests curves for the materials produced with the three supports

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Table 1 Tensile tests results

Samples	Max. stress (MPa)	Max. strain (%)	Young's modulus (MPa)
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. CONCLUSION

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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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