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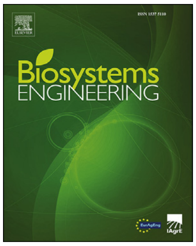

Research Paper

Mechanical characterization of blends containing recycled paper pulp and other lignocellulosic materials to develop hydromulches for weed control

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Spreading of hydromulch as a crop management technique might show important advantages over plastic films for weed control, because it can be applied anywhere in a field, even in orchard and vineyard rows. In the present work, 24 blends were prepared by mixing paper pulp, from recovered paper and cardboard coming from paper mills, with different additives: (a) wheat straw, rice hulls, and substrate used for mushroom cultivation on the one hand as fillers, and (b) rice bran, white glue, sodium silicate, and powered gypsum on the other hand as agglomerating agents. The blends were tested with a texture analyser to evaluate their mechanical properties, testing the puncture resistance (24 blends) and the tensile strength (15 blends). Scanning electron photomicrographs of some blends were obtained in order to explore the relationship between their components and the mechanical properties. The results indicate that a blend prepared with paper pulp, wheat straw sieved at 2.5 mm and gypsum attained the highest stress resistance and tensile strength. An environmentally controlled experiment was performed on this and another hydromulch in which rice husk substituted wheat straw to evaluate their efficiency for reducing weed seedling emergence, using propagules of four common summer weeds. Compared with the control treatment performed, the hydromulches reduced seedling emergence from 64.6% to 95.9%. In general, the percentage of dead seedlings underneath was greater than that which passed through the barrier, making the hydromulches promising tools for preventing seedling emergence and for managing the weed seed bank in field conditions.

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1. Introduction

Crop protection, including weed management, is a critical issue in agriculture (FAO, 2018). Mulching, which consists of covering the soil surface with organic or inorganic materials, is used to control soil moisture, soil temperature, nutrient loss, salinity, erosion, soil structure, etc. (Lalljee, 2013). Its benefits for the growth and yield of annual and perennial crops have long been recognised (Kasirajan & Ngouajio, 2012). Although it is an age-old practice for managing weeds, mulching has largely dwindled since the widespread use of herbicides; however, now it is gaining importance in the context of sustainable agriculture (Lalljee, 2013). Different mulching materials have been used in different crops in different climatic environments for weed management, and results vary according to the chosen approach, growing practices, conditions and species (e.g., Haapala, Palonen, Korpela, & Aokas, 2014; Merwin, Rosenberger, Engle, Rist, & Fargione, 1995; Teasdale & Mohler, 2000; Warnick et al., 2006). These materials include crop residues, non-degradable plastic films, and also biodegradable films. The lifetime of an agricultural mulch varies considerably (i.e., months to years) depending on the nature of the material, its application, its thickness, and the environmental conditions (Kyrikou & Briassoulis, 2007; O'Brien et al., 2018).

Plastic mulching has become widely used, but plastic mulches have the potential to alter soil quality by shifting the edaphic biocoenosis, accelerate C/N metabolism, eventually depleting soil organic matter stocks, increase soil water repellency, and favour the release of greenhouse gases (Steinmetz et al., 2016). Also, non-degradable films must be removed from the fields, and the recovery is labour-intensive and has economic costs (Chiellini & Salaro, 2002; Kyrikou & Briassoulis, 2007). Moreover, adverse effects may arise from plastic additives, with enhanced pesticide runoff and plastic residues likely to fragment into remaining microplastics (Steinmetz et al., 2016). Active research has been done worldwide in recent years to find biodegradable alternatives, such as biodegradable films or paper mulches (e.g., Liu & Gao, 2018; Sartore, Vox, & Schettini, 2013; Treinyte et al., 2018).

Paper mills are the most common end users of recovered paper, which is employed as a feedstock to manufacture recycled paper and other paper products. One possible application for recovered paper and cardboard is to produce, mixed with other lignocellulosic products, biodegradable materials that are applied as slurries, which maintain their integrity throughout the growing season and provide broadleaf and grass weed control (Warnick et al., 2006). Hydromulch can be defined as a mix of water with some sort of lignocellulosic material or polymers, plus other additives suited for the particular purpose, that is applied not as a film but as liquid (Warnick et al., 2006). Hydromulch has been used in many land rehabilitation services, for instance to mitigate post-fire runoff and erosion (Warnick et al., 2006), and has been used experimentally for fruit orchards (Cline, Neilsen, Hogue, Kuchta, & Neilsen, 2011).

Hydromulch can be an effective weed control method with much scope particularly for perennial crops, because it can be applied around already planted trees, where mechanical and chemical weeding is difficult. It has the potential to last for a long time on the soil, although its lifetime is influenced by several environmental factors, among them the frequency and intensity of rain and the amount of solar radiation (Immirzi, Santagata, Vox, & Schettini, 2009). It is vital nowadays to make agricultural production sustainable, and in this respect, one of the new objectives of many governments is the transition to a more circular economic model with products, processes and business models that are designed to maximise the value or utility of resources while at the same time reducing adverse health and environmental impacts (European Union, 2017). In this scenario, the use of low-cost and available organic agricultural residues has been proposed to compound biodegradable mulch materials (França et al., 2018; Liu & Gao, 2018) as a way to make weed management practices cost-effective, labour-saving, and environmentally sound (Warnick et al., 2006). To begin with, hydromulch would provide an outlet for recovered paper. Furthermore, agricultural waste materials such as rice husk or used mushroom substrate have a potential to be used for hydromulches that could be interesting choices for weed management. Soil temperature and solar radiation reaching the soil is generally modified when using mulches, as reviewed by Kasirajan and Ngouajio (2012). Weed seed germination and rhizome sprouting are affected by changes in both environmental factors. Moreover, if weed seeds germinate and/or their rhizomes sprout, these plants may or may not be able to perforate the mulches depending on the combination of their biological characteristics and the physical properties of the mulches.

In fact, very little is found in the literature concerning the physical mechanisms involved in weed control by mulches, and even less in the particular case of hydromulches, which could be composed of more than one material. The physical mixture of two materials with different properties can lead to the formation of a new material, called a composite, the properties of which can be the result of a synergy between the characteristics of the original materials. Unlike other types of mixtures such as alloys, in composites the original materials keep their initial properties intact (Barbero, 2017). In the field of composites science, the materials involved are divided into those that have the capacity to agglomerate (matrix), those that can reinforce the matrix due to their resistance, and finally those that contribute to increase the final volume of the composite and in this way decrease the economic costs of the product, but without modifying the final properties (filler). From a theoretical point of view, the behaviour of a composite can be predicted using a micro-mechanics approach (Aleksendrić & Carlone, 2015, pp. 1–5), termed the rule of mixtures, as defined by equation (1):

$$P_c = P_f V_f + P_m (1 - V_f) \tag{1}$$

where P_c is the property to predict from the properties of the materials (P_m and P_f) and the unitary V_f and (1 - V_f) volumes of each one. In the case of predicting the density, the result obtained by the equation is exact. However, when the purpose is to predict mechanical properties (any type of resistance or modulus of elasticity) a whole series of conditions must be fulfilled, such as the elasticity of the materials, the uniformity of their distribution or the adherence between them. These conditions are not always met, and therefore the formula is only an approximation to the final result. Several authors have proposed modifications to the equation in order to adapt to particular materials that do not comply with all the limitations mentioned previously. Summerscales, Hall, and Virk (2011) and Virk, Hall, and Summerscales (2012) incorporate some adaptations for the reinforcement of fibres of plant origin. In any event, the abovementioned equation indicates that any given property of the composite is located at a point between the properties of the matrix and the reinforcement materials, and is proportional to the volume of each of them.

This paper presents an evaluation test of several blends developed as prototypes to be used as biodegradable hydromulches, whose common and main component is paper pulp from a paper mill. In order to improve the structure and consistency of the pulp several additives were tested, but without losing its characteristic as a liquid mixture suitable for spraying onto the soil surface of the field. Thus, the aims of the study were: a) to characterise the typology of paper pulp fibres); b) to develop several blends by mixing the pulp with other components as fillers and as agglomerating agents; c) to evaluate the resistance to punching and to traction of the mulch layers once the blends have been dried; and d) to test the performance of two hydromulches in reducing seedling emergence of four common summer weed species under controlled environmental conditions.

2. Materials and methods

2.1. *Preparation, composition and mechanical characterization of blends*

Paper pulp was used as a base component to develop the hydromulch samples. The pulp was supplied by Saica, a paper mill in Zaragoza (Spain) that manufactures recovered waste paper and cardboard. The raw material was finely shredded, debugged and refined until a paper pulp was obtained. According to data provided by Saica, obtained by analysing more than 20,000 fibres with a Kajaani FS300 device, the morphological characteristics of the fibres are length 0.48 mm, width 18.64 μm, curl index 15.3%, and fines 35.91%. The microscopic characteristics of the fibres present in the paper pulp can be observed in Fig. 1.

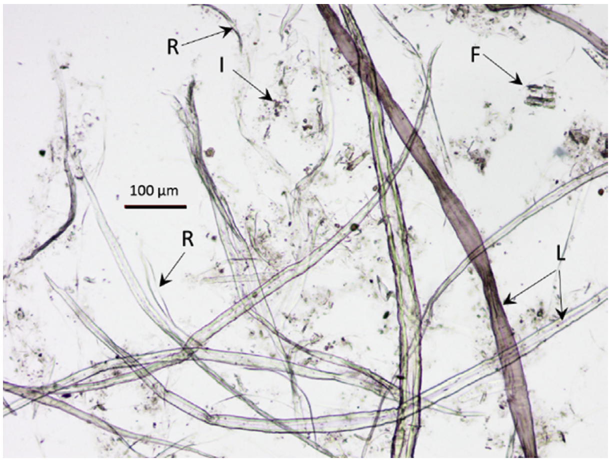

Fig. 1 – Micrograph of the paper pulp from Saica, obtained by processing recovered paper and cardboard. (L) fibres, (F) fines, (I) impurities, (R) fibres that show fibrillation from the outer fibre wall. The pulp was zinc iodine chloride stained.

To prepare the blends representing several hydromulch samples, in addition to paper pulp, the following crop products were used: (1) wheat straw cut in a mill, sieved at 2.5 and at 5 mm, from CITA (Centro de Investigación y Tecnología Agroalimentaria) crop fields, (2) used mushroom substrate, the residual compost waste generated by the mushroom (*Agaricus bisporus*) production industry (provided by Sustratos de la Rioja SL), (3) rice husk and bran provided by Arrocera del Pirineo, Alcolea de Cinca. Figure 2 shows the appearance of these products. Moreover, some non-crop products were also used. They were (i) powdered gypsum type B1, with a calcium sulphate hemihydrates content of <50%, a setting time of <20 min and a characteristic strength of 2 MPa, manufactured according to UNE-EN13279-1:2009, (ii) sodium silicate with a concentration of 34.2%, and a pH of 12 at a concentration of 1%; (iii) commercial white glue (polyvinyl acetate).

In Table 1 we summarise the different blends tested to develop the hydromulches, indicating the dosage of each material used. The component proportions were based on preliminary results reported in Galvis (2015). First the paper pulp was poured into a container. Then the crop products (wheat straw, used mushroom substrate, rice husk and rice bran) were added and all the components were well mixed. At the end and just before pouring the contents of the container into a tray, the components powdered gypsum, white glue and sodium silicate were included and the mixtures were stirred. The trays on which the samples were placed measured 36 × 50 cm and were previously lined with peat moss that was watered and flattened. The sheets obtained were dried in a greenhouse with forced-air equipment. The recorded greenhouse mean temperature was 18.6 °C, and ranged from 6.9 °C to 38.6 °C. Prior to the laboratory assays, the humidity of the dry mulch sheets was checked and was found to range from 7.1% to 14.2%. The thickness of the sheets ranged from 0.75 mm to 8.99 mm.

Tension and punching tests were performed to determine the mechanical characteristics of the blends. We presume two main manners in which recently germinated weed seedlings can overcome the hydromulch layer: penetrating directly or lifting after breaking it (Fig. 3). So, punching tests could inform about the resistance of hydromulch to being penetrated by the weed seedlings and tension tests could provide information about the tensile strength of the hydromulches, and consequently their potential capacity to control the emergence of weed seedlings through a lifting mode.

The equipment used to test the mechanical properties of the samples was a Stable Micro Systems XT-plus Texture Analyser, with specific probes or tensile grips for each type of assay. Figure 4-A displays the texture analyser used in the tests, with details of the equipment prepared to perform the penetration test and the probe used (7.86 mm in diameter), and the tensile grips for conducting the tension test. The load cell of the analyser has a maximum capacity of 500N and the cross head speed in the assays was 1 mm min^{-1} (tension tests)

Table 1 – Components and dosage of materials of the twenty-four 36 × 50 cm blends prepared to develop hydromulches showing weight or volume needed per litre of paper pulp. Acronyms are given in parentheses.

Blends	Matrix			Reinforcement/Filler					
	Gypsum (G)	White glue (WG)	Sodium silicate 34.2% (SS)	Paper pulp (PP)	Wheat straw 5 mm length (WS5)	Wheat straw 2.5 mm length (WS2.5)	Used mushroom substrate (MS)	Rice husk (RH)	Rice bran (RB)
A[a]				2 L					
B[a]				2.5 L					
C[a]				2.5 L	20 g L^{-1}				
D[a]				2 L	25 g L^{-1}				
E[a]				2 L		25 g L^{-1}			
F[a]				2 L		40 g L^{-1}			
G	20 g L^{-1}			2 L	40 g L^{-1}				
H	20 g L^{-1}			2 L		40 g L^{-1}			
I[a]	40 g L^{-1}			2 L		40 g L^{-1}			
J[a]	25 g L^{-1}			2 L		50 g L^{-1}			
K[a]	50 g L^{-1}			2 L		50 g L^{-1}			
L[a]		20 ml L^{-1}		2 L		50 g L^{-1}			
M[a]		2.5 ml L^{-1}		2 L		50 g L^{-1}			
N[a]			1.1 ml L^{-1}	2 L		50 g L^{-1}			
O[a]			2.2 ml L^{-1}	2 L		50 g L^{-1}			
P[a]	20 g L^{-1}			2 L			100 g L^{-1}		
Q	40 g L^{-1}			2 L			100 g L^{-1}		
R		5 ml L^{-1}		2 L			100 g L^{-1}		
U	20 g L^{-1}			2 L				62.5 g L^{-1}	
V	40 g L^{-1}			2 L			125 g L^{-1}		
W				2 L			125 g L^{-1}		
X	20 g L^{-1}			2 L			125 g L^{-1}		
Y				2 L				125 g L^{-1}	30 g L^{-1}
Z[a]	40 g L^{-1}			2 L				125 g L^{-1}	

[a] Blends were tested using tensile tests, in addition to punching tests.

Fig. 2 – **Appearance of some reinforcement components used in the blends. From left to right, wheat straw sieved at 5 mm (WS5), wheat straw sieved at 2.5 mm (WS2.5), used mushroom substrate (MS), rice bran (RB) and rice husks (RH).**

and 4 mm min^{-1} (punching tests). The specimens used in the assays (Fig. 4-B) were circular for the puncture resistance tests and dogbone shaped or rectangular (depending on the characteristics of the sample) for the tensile tests. They were obtained by cutting the sheets.

The area used to calculate tensile strength corresponds to the thickness and width of the specimen at the point of rupture. For punching tests, the surface used to calculate the punching shear corresponds to the lateral surface of a cylinder of 7.86 mm diameter using a mean of three thickness values measured in the penetration section (Fig. 5). For the punching tests 20 specimens belonging to each of the 24 blends (see Table 1) were tested. In the case of tension tests, the number of blends was reduced to 15, and the number of specimens ranged from 5 to 21 depending on the blend. This was due to the fragility of some samples, which could not be tested because they could not be cut to obtain the specimens without breaking the sheet. Likewise, in some tests several specimens crumbled in the manipulation process or they broke in areas closed to the grips and were discarded.

In each type of test, a force–displacement curve was obtained, representing the behaviour of each specimen. The parameters that permitted the characterization of the blends were: a) the maximum breaking strength or modulus of rupture, named Stress, which is obtained by dividing the maximum force registered by the area of the section (transversal in traction test, tangential in punching test), and b) the toughness or specific energy of the assay, that is, the ability of a material to absorb energy and plastically deform without fracturing, named Energy, which is calculated by integrating the area under the force–displacement curve from the start of the assay to the point of maximum force (Fig. 5).

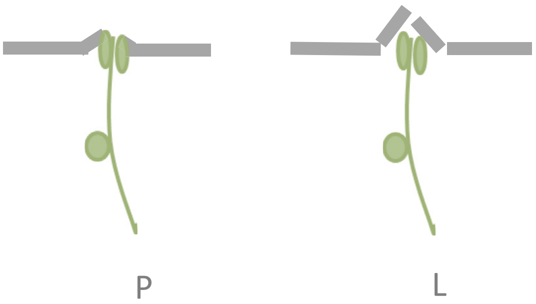

Fig. 3 – **Two main hypothetical modes of seedling emergence through the hydromulch layer: (P) penetration; (L) lifting. Redrawn from Aubertot, Dürr, Richard, Souty, and Duval (2002).**

The variables Stress and associated Energy were statistically analysed by means of analyses of variance and mean comparisons in three ways: i) considering the type of blend as the source of variation, with 24 levels for punching tests and 15 levels for traction tests (Table 1); ii) with the blends that contain wheat straw sieved at 2.5 mm (J, K, L, M, N, and O), the matrix was considered as the source of variation with three levels: gypsum, sodium silicate and white glue, and iii) with some blends containing powdered gypsum (K, P, and Z) the source of variation considered was the filler, with three levels: wheat straw, rice husk, and used mushroom substrate. The analyses of variance (Type III sum of squares) and the separation of means (Tukey HSD test) were performed using the GLM procedure (SAS, 2013); residual analyses were performed to ensure that variances were homogeneous. The chosen level of significance for the statistical tests was $P < 0.05$.

In order to know the role of the different components the most promising blends were examined with the scanning electron microscope (SEM).

2.2. *Effects of hydromulches on weed seedling emergence and evaluation of their suitability for control*

An experiment to know the effect of two different hydromulch coatings on weed seed behaviour, i.e., on their germination and the ability of the seedlings to punch the mulch coat, was performed under controlled environmental conditions. Propagules of four cosmopolitan summer weed species were collected and dry-stored at room temperature in the laboratory at least a year before use. One-seeded spikelets of *Digitaria sanguinalis* L. (Scop.) (large crabgrass), seeds of *Amaranthus retroflexus* L. (redroot pigweed) and one-seeded fruits of *Lactuca serriola* L. (prickly lettuce) were collected at Caldes de Montbui, while one-seeded fruits of *Sonchus oleraceus* L. (sowthistle) were collected at Castelldefels, both localities in Barcelona province.

Propagules were rinsed for 10 min in 5% diluted sodium hypochlorite for surface sterilization (ISTA, 1985) before the beginning of the experiment, conducted in a growth chamber at 25 °C, 12 h darkness/12 h light. Illumination was provided by white fluorescent tubes (4 × 18 W).

On the one hand, a test was performed to know the potential germination ability of the propagules. Lots of 50 propagules of each species were placed in five 9 cm diameter plastic Petri dishes and then transferred to the incubator. Distilled water (3 ml) was added to each dish at the beginning

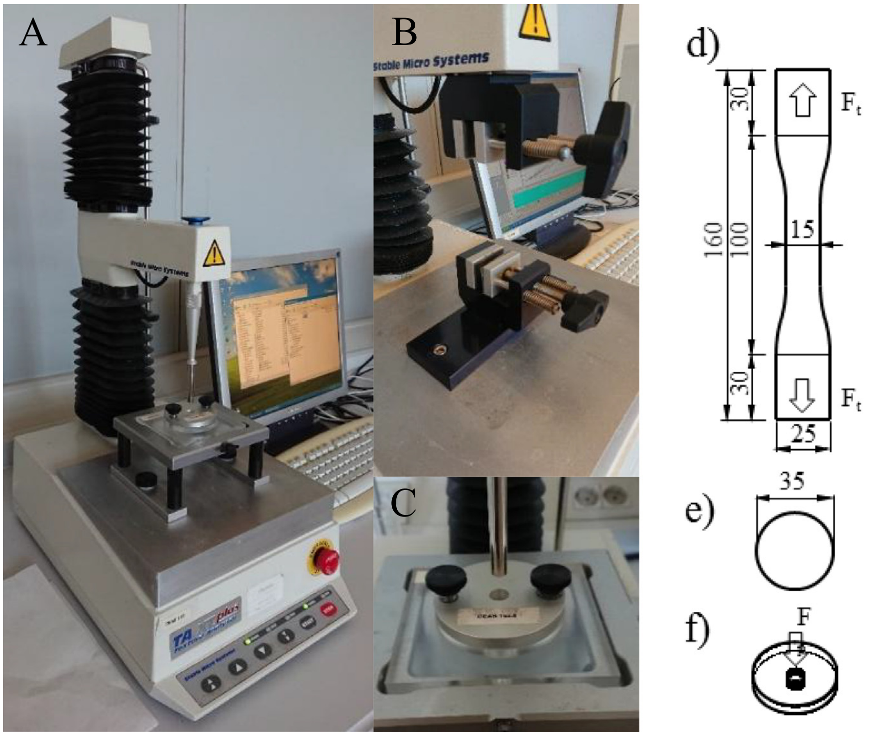

Fig. 4 – A-Texture analyser prepared to perform the punching and the traction tests. B- Probe used for traction tests. C-Probe used for punching tests; the specimen is held between the circular metallic plaque and the PMMA support; the steel cylinder has a diameter of 7.86 mm. At right, specimens used to conduct the traction tests (d-dogbone shape) and punching tests (e,f-circular); values are given in mm.

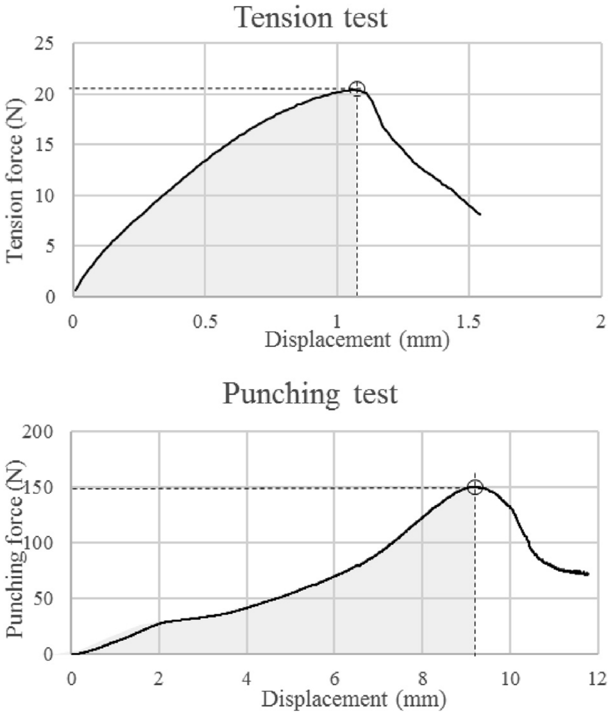

Fig. 5 – Typical force–displacement curves for each type of essay performed.

of the test, and water was added later as required. Seeds were considered to be germinated with the emergence of the radicle. After 30 days, the non-germinated propagules were tested for their viability by means of the tetrazolium test (ISTA, 1985). The percentage of accumulated germination of viable seeds was obtained for each species.

In parallel, the effect of the hydromulch was tested by placing the propagules on a moist substrate with the mulch coating. Specifically, alveoli (4 × 4 × 4 cm) were employed. Each one was filled with moist vermiculite substrate, and topped with a thin quadrat of filter paper finely bored previously to accommodate 50 propagules of each species. The role of this filter paper is to allow the growth of the radicle if any seed germinates, but enable us to recover the non-germinated propagules after the 30 days' incubation period. On top, a 4 × 4 cm mulch coat was placed after its preparation. Every four days, water was provided by capillarity as required. The two types of hydromulches, named wheat straw (WS) and rice husk (RH), were prepared using the following components and proportions: a) 1 L of paper pulp, 50 g of wheat straw of 2.5 mm length, and 30 g of gypsum, and b) 1 L of paper pulp, 75 g of rice husk and 40 g of gypsum. Square sheets with a surface area of 30 × 30 cm, 4 mm thick for WS and 5 mm thick for RH, were obtained after removing excess water with a vacuum device. Each sheet was cut to obtain 4 × 4 cm square coats, which were then placed on top of the prepared alveoli. For each species and type of mulch, three replications in a completely randomised design were utilised. Data related to the number of emerged seedlings (penetrating the mulch layer), the number of non-germinated propagules (observed under the mulch layer once it was removed), and the number of germinated but non-emerged propagules were obtained. After the mulch was removed, the set of non-germinated propagules was subjected to light stimulus for a week; the accumulated seed germination during this week represents the fraction of seeds that, although they did not germinate under the mulch coat, were non-dormant.

Finally, all non-germinated propagules were tested for their viability by means of the tetrazolium test (ISTA, 1985), and so the percentage of non-viable seeds was also obtained.

Three variables were subjected to statistical analysis considering the effect of the type of hydromulch, with two levels (WS, RH): the proportion of seedlings that emerged through the mulch, the proportion of propagules that germinated but were unable to perforate the mulch coat, and the fraction of non-germinated propagules that were non-dormant. The analyses were performed using a generalised linear model with a binomial distribution. Parameters were estimated using a complementary logit link function and Type III analysis options. Likelihood ratio statistics were used to compute the significance of each effect. Finally, least-squares means of the levels of the effects were computed and compared using probability values from the chi-square distribution. The GENMOD procedure (SAS, 2013) was used to perform both generalised linear models and means comparisons.

3. Results

3.1. *Characterization of blends*

Table 2 shows the mean strengths and toughness of the blends obtained in the tests. In spite of the great within-blend variability of the variables measured, the type of blend was a significant effect in the ANOVAs of the four variables. Blend K (paper pulp + wheat straw sieved at 2.5 mm + powdered gypsum at highest dose, Table 1) exhibited the highest means, and particularly in the traction tests showed significant differences from the statistical point of view with respect to all the other blends. To characterise the resistance capacity and plasticity of the blends, the scatter diagrams in Fig. 6 display the values of punching/tension stress related with their observed energy in both types of assays.

Large differences in the mean stress and mean energy were observed within the tested blends, both in the punching test and in the traction test (Table 2, Fig. 6). In general, blends located in the upper right-hand corner of the diagrams given in Fig. 6 yielded better mechanical properties. Blends with means located above an imaginary diagonal line running from the zero–zero point to the upper right-hand corner could be considered as composites that optimise resistance, whereas being located below this diagonal represents a tendency towards ductility (Fig. 6). For instance, in relation to the punching tests, blend Z would represent a quite resistant mixture but with medium ductility, while blend D would represent a mixture with a low resistant level but high ductility. In general, blends that behave well in punching tests at the same time display a good performance in traction tests. Considering simultaneously the two types of resistances (tensile stress and punching shear) and the characteristic energies, the performance of the blends allows their mechanical capabilities to be easily established. Moreover, following this criterion the blends can be easily ranked. In Fig. 7 blends located above the same imaginary diagonal line that has been mentioned offered better punching features than traction ones, and the blends located below the diagonal had inverse characteristics. The distribution of the blends in the scatter diagram in the two graphs (Fig. 7) is nearly the same, indicating that forces and energies are relatively proportional.

The components or materials used to prepare the mixtures can explain the latter's different mechanical behaviour. In fact, the mixture should be considered as a composite material formed by a matrix or binding agent and another material or materials acting as reinforcement. In order to compare the role of the reinforcement and binding components of the blends, analyses of variance are performed on some blends (Table 3). In the first instance, gypsum (G), white glue (WG) and sodium silicate (SS) were considered as materials with conglomerate capacity. The performance of the three materials for binding reveals that gypsum is the material that provides the blends with the best compressive and tensile strengths (Table 3). Only in the case of the parameter mean energy, gypsum and white glue did not differ in the punching tests at $P < 0.05$; in fact, the type of binding effect was a non-significant effect for this variable in the ANOVA. Moreover, comparison of the three reinforcement materials (WS, wheat straw; MC, mushroom compost; RH, rice husk) used in the blends together with gypsum as conglomerate showed that wheat straw is the material with the highest means in terms of strengths and characteristic energies of traction (Table 3). Neither of the two variables of the punching tests have significance in the ANOVAs that considered the reinforcement type as an effect.

The SEM photomicrographs on the broken surfaces of some blends (K, I, P, Q, and Z, Table 1) revealed the presence of the different components such as wheat straw, paper pulp fibres, unburned crystals of gypsum, rehydrated gypsum agglomerates and rice husks (Fig. 8). In Fig. 8A (photomicrographs 1 to 4, samples K and I), it can be observed how the gypsum has agglomerated the paper pulp fibres (GA) and yet was not deposited on the wheat straw fragments (WS). On the other hand, in photomicrograph 3 and 4 (Fig. 8A, sample I) we can also see that, due to the smaller amount of gypsum present in the mixture, practically no deposits were formed and the fibres are perfectly distinguishable (F). In photomicrograph 4 (Fig. 8A) there are gypsum deposits but in a discontinuous pattern, and this implies a much lower capacity for resistance. The same situation can be observed in Fig. 8B, where the pulp fibres were coated with gypsum deposits, whereas the rice husk showed some attached fibres but without gypsum deposits. In Fig. 8C the presence of numerous micrometric fungal hyphae can be seen.

3.2. *Hydromulch can efficiently prevent weed seedling emergence and reduce the seed bank*

The accumulated percentages of germination of viable seeds obtained in the Petri dish test were 98.0% for *A. retroflexus*,

Table 2 – Mean punching force and mean associated energy of the 24 blends at left, and mean traction force and mean associated energy of the 15 blends at right. The components and dosage of materials of the blends can be found in Table 1.

Blends	Punching					Traction				
	Stress resistance (MPa)		Energy (J/m^2)			Tensile strength (MPa)		Energy (J/m^2)		
	Mean[a]	SEM[b]	Mean[a]	SEM[b]	n[c]	Mean[a]	SEM	Mean[a]	SEM[b]	n[c]
A	0.68 dc	0.054	1431.9 hg	125.28	20	0.11 b	0.019	57.27 b	23.09	7
B	0.60 dc	0.04	1025.2 h	106.1	20	0.09 cb	0.01	48.52 cb	11.48	14
C	0.62 dc	0.085	1970.9 edhgf	290.8	20	0.02 ed	0.006	26.02 cbd	14.46	9
D	0.48 d	0.055	1514.0 hgf	182.3	20	0.02 ed	0.003	3.86 d	1.14	14
E	0.49 d	0.075	1636.3 ehgf	219.28	20	0.01 ed	0.002	3.23 d	0.68	14
F	0.64 dc	0.075	2215.0 edhgcf	252.51	20	0.01 e	0.001	1.17 d	0.42	5
G	0.94 bdac	0.14	2907.8 ebdacf	431.92	20	–				
H	0.72 dc	0.111	2255.2 ebdhgcf	310.42	20	–				
I	0.94 bdac	0.096	2938.2 ebdacf	260.7	20	0.01 e	0.002	2.48 d	0.95	7
J	1.06 bac	0.141	3209.4 bdac	398.42	20	0.01 e	0.001	2.17 d	0.4	10
K	1.45 a	0.163	4227.0 a	435.08	20	0.18 a	0.023	113.60 a	17.85	12
L	1.00 bdac	0.114	3620.2 bac	401.13	20	0.02 ed	0.002	13.63 cd	2.27	20
M	0.85 dc	0.09	2947.0 ebdacf	303.69	20	0.01 e	0.001	3.82 d	0.56	13
N	0.83 dc	0.105	2717.0 ebdgcf	278.92	20	0.01 e	0.002	3.36 d	1.01	12
O	0.78 dc	0.088	2713.3 ebdgcf	254.67	20	0.01 e	0.001	3.90 d	1.04	15
P	1.11 bac	0.098	3281.1 bdac	276.76	20	0.05 cd	0.006	51.85 cb	7.08	21
Q	0.88 bdc	0.088	2562.0 ebdgcf	315.92	20	–				
R	0.88 bdc	0.098	2757.2 ebdgcf	271.28	20	–				
U	0.94 bdac	0.099	2827.9 ebdagcf	223.3	20	–				
V	0.97 bdac	0.112	2868.1 ebdagcf	263.43	20	–				
W	0.62 dc	0.078	1835.5 edhgf	217.15	20	–				
X	0.89 bdc	0.065	3056.7 ebdac	163.54	20	–				
Y	0.90 bdc	0.092	2564.4 ebdgcf	198.81	20	–				
Z	1.38 ba	0.142	3704.4 ba	321.16	20	0.03 ed	0.005	39.45 cbd	8.31	16

[a] Within columns, means followed by the same letter are not significantly different ($P < 0.05$) according to the Tukey HSD test.
[b] SEM: Standard error of the means.
[c] n: number of specimens tested.

96.5% for *D. sanguinalis*, 97.2% for *L. serriola* and 99.5% for *S. oleraceus*. The non-viable seeds were a small fraction of the total seeds employed, ranging on average from the 0.5% of *S. oleraceus* to the 3.5% of *D. sanguinalis*. Considering this information, and the mean percentages of emergence shown in Table 4, the results are conclusive: the barrier effect of the two mulches was quite high. In the case of RH the control of weed emergence expressed as a percentage ranged from 85.9% to 64.6%. The WS mulch exercised even higher control, from 92.9% to 85.7%, depending on the species.

Moreover, within seeds that were able to germinate, except for *A. retroflexus*, the mean percentage of non-emerged seedlings was greater than the mean percentage that passed through the mulch. The values obtained varied depending on the species and the mulch type (Table 4), those of *L. serriola* being particularly interesting; in this species up to 60% of the viable seeds germinated but seedlings died underneath the RH mulch coat. In contrast, only 12% of the seedlings of *A. retroflexus* failed to emerge underneath the RH mulch coat.

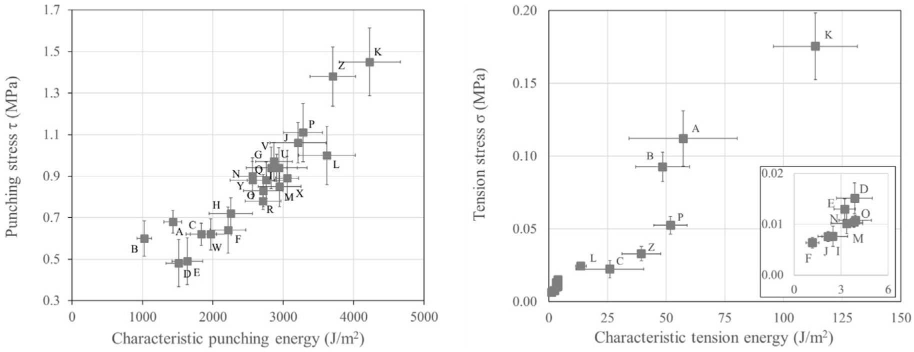

Fig. 6 – Relationship between mean stress and mean characteristic energies obtained in the punching tests, at left, and in the traction tests, at right, conducted in the texture analyser with the specimens of the different blends. In the scatter plot, each point together with its capital letter represents a blend (see Table 1). The bars correspond to the standard error of the mean.

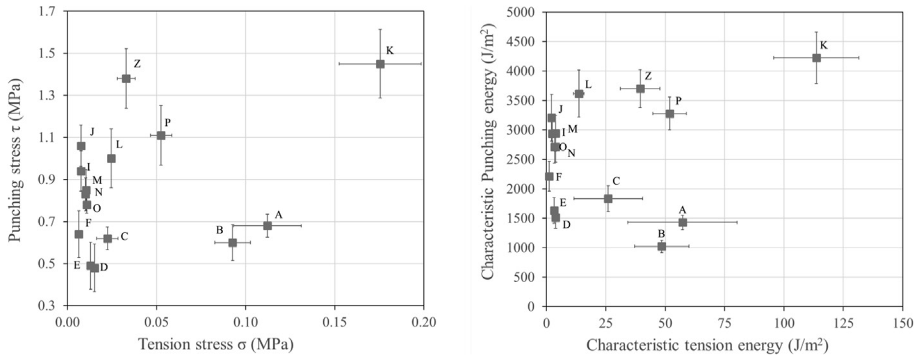

Fig. 7 – **Relationship between mean punching and mean tension stress, at left, and their respective characteristic energies, at right, observed in the assays conducted in the texture analyser with the specimens of the different blends. In the scatter plot, each point together with its capital letter represents a blend (see Table 1). The bars correspond to the standard error of the mean.**

After removal of the mulch coats, the light stimulus promoted the germination of the majority of the remaining seeds (Table 4), with the exception of those of *D. sanguinalis*, which were almost all dormant, and up to 65% of those of *A. retroflexus*.

4. Discussion

The information supplied by Saica about the paper mill indicates that the fibres we used have a similar length and thickness as those belonging to pulps from deciduous species (0.75 < L < 1.5 mm and 10 < T < 20 microns), but the content of fines is higher than in virgin paper pulp (García Hortal, 1988). These characteristics would permit the production of recycled paper or cardboard, but they are insufficient and unsuitable for developing hydromulches with the desired durability properties because the material would be destroyed. This is especially true for durability when in contact with water, due to the fact that the hydrogen bonds between fibres would be destroyed. For this reason, other low-cost materials and agricultural wastes considered as refuse were chosen to add and mix with paper pulp in order to improve its properties.

Table 3 – Mean punching and traction forces and mean associated energies of (*I*) the three reinforcement materials (WS, wheat straw; MC, mushroom compost; RH, rice husk) when the binding material of the blends was powdered gypsum, and (*II*) the three matrices (G, gypsum; WG, white glue; SS, sodium silicate) with conglomerating capacity used to develop the blends.

	Punching tests (n = 20)		Traction tests	
	Stress (MPa)	Energy (J/m²)	Stress (MPa)	Energy (J/m²)
I				
WS	1.446 a	4227.0 a	0.174 a (n = 12)	113.59 a (n = 12)
MC	1.108 a	3281.1 a	0.053 b (n = 21)	51.85 b (n = 21)
RH	1.381 a	3704.4 a	0.033 b (n = 16)	39.45 b (n = 16)
II				
G	1.251 a	3718.2 a	0.099 a (n = 22)	62.94 a (n = 22)
WG	0.928 b	3283.6 ab	0.019 b (n = 33)	9.76 b (n = 33)
SS	0.805 b	2715.1 b	0.010 b (n = 27)	3.66 b (n = 27)

Within columns I and II, means followed by the same letter are not significantly different ($P < 0.05$) according to the Tukey HSD test.
n: numbers of specimens tested are given in parentheses.

From the mechanical point of view, in the assays the materials are subjected to different tension states. The resistance of the blends (as composites) will depend on the traction and compression capability of the matrix and the fibres, as well as the adhesion between fibres and matrix (Barbero, 2017). The ideas of Summerscales et al. (2011) and Virk et al. (2012) regarding composites with fibres of plant origin allow us to look at the mechanical behaviour of the materials tested based on the characteristics of the components employed at different dosages in the blends. The results presented in Table 2 and Figs. 6 and 7 make it possible to analyse the role of the different components used in the blends and their contribution to the resistance of the mixture. Therefore, the different materials used in the blends can be considered as binding agents or as reinforcement ones, in spite of the results indicating that many of them played the role of filler materials. Due to the formation of hydrogen bonds between fibres, recycled paper pulp could be considered to have a certain binding capacity. However, in the present work it has been considered as reinforcement material, as is usually the case with cellulosic materials in the field of composites. Furthermore, the formation of this type of bond is only effective between materials of the same fibrous nature, while it would be ineffective in other cases. Both blends A and B (Table 1) contain only recycled paper pulp. Their mechanical behaviour is clearly defined by their good tensile strength (Table 2). Nevertheless, both display the worst resistance to punching (less than 50% of the best blend). There are no statistical significant differences between the two in either test. So this material, as expected owing to its fibrous nature, has good tensile strength and very bad punching resistance and, therefore, oblique compression. These results justify considering pulp as a reinforcement element, and not as a binder, and the need to improve its resistance to compression in order to obtain a good hydromulch. As far as we know, there is no information about the mechanical behaviour of composites such as those we present in this work. Immirzi et al. (2009) and França et al. (2018) have worked with composite materials to

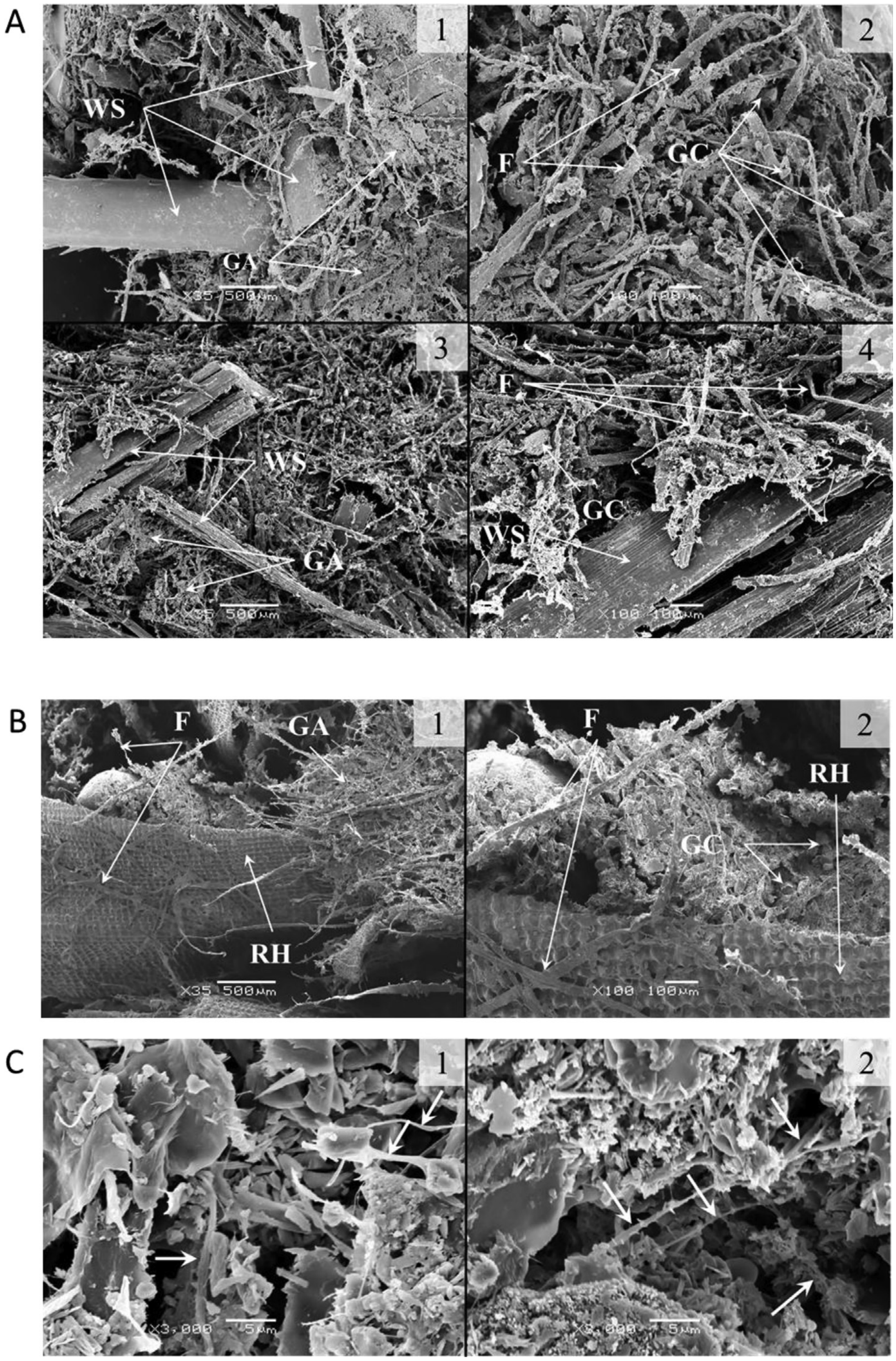

Fig. 8 – SEM photomicrographs on broken surfaces of the samples obtained from the blends: (A): K (1 and 2) and I (3 and 4). Wheat straw (WS), paper pulp fibres (F), gypsum agglomerates (GA), and gypsum crystals (GC). (B) Z (1- Magnification at 35x, and 2- Magnification at 100x). Paper pulp fibres (F), gypsum agglomerates (GA), gypsum crystals (GC), and rice husk (RH). (C) P (1) and Q (2) prepared with mushroom substrate and powdered gypsum (in different quantities). The arrows indicate the presence of fungal hyphae that act as reinforcement material.

develop mulches, but their results are not comparable with those obtained in this work, since the mechanical characterization methods are slightly different, although some films tested by the mentioned authors for agricultural mulch application, either biodegradable polymer films or polyethylene films, give tensile strength values at least two times higher than those obtained with the best blends presented here (França et al., 2018; Hosseinabadi, Zebarjad, & Mazinani, 2011).

There are significant mean differences in stress and traction energy between wheat straw and the other two reinforcement materials (used mushroom substrate and rice

Table 4 – Significance of the hydromulch effect for three variables analysed, related with the behaviour of the weed propagules after 30 days of incubation at 25 °C, 12 h darkness/12 h light regime. At right, least square means of the levels of the effect, rice husk and wheat straw, are given.

Variables	Species	Effect	lsmeans (%)[a]	
		P > chi-square	Rice husk	Wheat straw
Emerged seedlings *versus* viable propagules	*A. retroflexus*	<0.0001	35.17 a	8.16 b
	D. sanguinalis	0.2840	14.13 a	11.07 a
	L. serriola	<0.0001	34.72 a	14.29 b
	S. oleraceus	<0.0001	35.43 a	7.14 b
Germinated but not emerged *versus* viable propagules	*A. retroflexus*	<0.0001	12.07 a	34.29 b
	D. sanguinalis	0.2742	23.42 a	19.56 a
	L. serriola	0.0002	62.50 a	42.86 b
	S. oleraceus	0.8739	35.04 a	35.71 a
Non-dormant *versus* non-germinated propagules	*A. retroflexus*	0.7134	32.03 a	34.04 a
	D. sanguinalis	0.0824	1.19 a	0 a
	L. serriola	0.0266	100 a	53.13 a
	S. oleraceus	0.0688	98.67 a	93.75 a

Within rows, means followed by the same letter are not significantly different ($P < 0.05$) according to the Tukey HSD test.

husk) (Table 3), and so blend K is potentially the best for hydromulch purposes, compared with P or Z (Table 1). Blends composed of fragments of wheat straw sieved at 2.5 mm obtain worse results in the traction tests than those in which this plant component was sieved at 5 mm, except in blend K with a gypsum dosage of 50 g L^{-1} (Table 1). The lack of significant differences among them seems to indicate that their mechanical behaviour to traction does not depend on the agglomerate component present in the blend. In spite of the content in fibres, wheat straw (unlike paper pulp) does not form hydrogen bonds, and for this reason this material does not produce adhesion between components. Moreover, bundles of wheat straw have a very smooth outer surface that hinders mechanical friction and facilitates the formation of fracture planes, which is not desirable. In photomicrographs 3 and 4 in Fig. 8A the wheat straw fragments do not display any adhered fibres or any particle of binding material originating from the hydration of gypsum. In the punching tests, the 5 mm sieved straw fragments could play a rafter role due to the layout of the specimen located between the circular probe and the hole in the support, and so the punching resistance would improve with respect to the specimens containing 2.5 mm sieved straw. Both reinforcement materials obtained quite good results in the punching assays. However, the resistance mechanisms involved in the blends composed of these two materials are different.

The roughness of the rice husk (Fig. 8B) could be responsible for its good results in the punching tests, because the internal friction improves the performance of the mixture. Nevertheless, the low adhesion regarding the pulp fibres-gypsum assembly explains why the tensile resistance was not high. In relation to the blends with mushroom substrate, the occurrence of many fungal hyphae (Fig. 8C) allowed the formation of lumps, but these masses do not have adhesion capacity, and they can easily disperse. Lumps improve the punching resistance, particularly when they appear in the gap between the probe and the hole in the support of the texture analyser. In terms of tensile strength, lumps do not seem to be very effective.

Among the three binders used in the blends, gypsum yielded the best results (Table 3). Except for the characteristic mean energy in the punching tests, its means were significantly different from those of the other binders, which did not differ in mean energies or in mean stress. The images in Fig. 8 show that to a large degree the agglomerates of this powdered gypsum were hydrated and nucleated on the surface of paper pulp fibres (Fig. 8A3 and 8B1), whereas both the surface of wheat straw and rice husk remain practically free of gypsum deposits. This could be because there are hydrophobic substances on the wheat straw and rice husk that prevent the precipitation of gypsum crystals. The areas marked with GA in Fig. 8A are representative of a quite compact frame that permits a suitable punching and traction resistance. The amount of powdered gypsum present in the blends seemed relevant for its mechanical characteristics. If it was low (<40 g L^{-1}), the hydrated material did not generate a continuous deposit and it appeared dispersed on the paper pulp fibres, where it can be identified perfectly. However, if the quantity was 50 g L^{-1}, the particles of gypsum agglomerated on the paper pulp fibres were able to come into contact with each other and form a continuous layer with better mechanical capacity.

The two hydromulches tested for their effect on seedling emergence were prepared with the components that give the highest mean values of stress resistance and associated energy in the punching tests. They both exercised weed control ability in three ways: first, the percentage of seeds that were able to germinate were reduced; second, on average more than 60% of the seedlings were unable to pass through the mulch layer and died; and third, a large fraction of the non-germinated seeds went into secondary dormancy.

In terms of reducing emergence, the best control was achieved by the wheat straw hydromulch for *A. retroflexus* and *S. oleraceus* (Table 4), whose emergence was reduced to a similar degree as by other biodegradable mulches (Cirujeda et al., 2012). Seedlings that failed to go through the mulch layer represent the fraction of the seed bank that diminished as a direct effect of the mulch, and from this point of view we highlight the results attained by rice husk hydromulch on *L.*

serriola, but in general for both types of hydromulch and all four weed species, the efficiencies were far from negligible. In any case, they are comparable to those achieved with solarization techniques (Arora & Yaduraju, 2008), in which land cultivation is incompatible with the weed control treatment. From this point of view, the hydromulches tested were clearly promising for weed management.

With respect to the seeds that remain non-germinated underneath the mulch layer, non-dormant seeds can germinate immediately after mulch removal, but those that acquire dormancy will not be able to germinate until some time has passed, and this is another means of control, because although the seed bank is not reduced, the weed density during the next season could be considerably lower. Here, the behaviour could be diverse, because it is known that the induction or the interruption of dormancy when seeds experience light or dark periods varies among species (Baskin & Baskin, 1998). Two of the species tested, *L. serriola* and *S. oleraceus*, were not controlled in this sense by the hydromulches, but *A. retroflexus* was fairly well controlled and *D. sanguinalis* was excellently controlled (Table 4).

5. Conclusions

The hydromulch blends developed in this work displayed similar behaviour to that of a composite formed by more than one material. In fact, the materials can be classified as matrix or binder, reinforcement, and filler. In relation to the binding materials (or agglomerate), the best was powdered gypsum, although the minimum amount necessary to obtain a continuous matrix is above 50 g per litre of paper pulp. At the mentioned dose, gypsum made, in combination with paper pulp and wheat straw, the significantly best mechanically performing hydromulch. It attained mean values of 1.45 MPa in punching stress and of 0.17 MPa in tension stress, while the associated mean energy values were 4227.0 J/m^2 and 113.6 J/m^2 respectively. At the same time, paper pulp behaved as a reinforcement material since it was able to nucleate on its surface most of the gypsum added to the mixture. The rest of the reinforcing materials used behaved as fillers that contributed very little to the mechanical resistance of the blends. Nevertheless, the mean tension stress of wheat straw was significantly higher than those of the other lignocellulosic waste materials tested, giving mean values of resistance and energy up to 50% higher than those of the blends reinforced with rice husk and used mushroom substrate.

Although the role of the reinforcing materials needs to be improved, two hydromulches, both composed of paper pulp and gypsum, but containing wheat straw or rice husk alternatively, reached promising levels of weed emergence reduction, because many seedlings were unable to pass through the mulch layer, ranging from 62.5% in prickly lettuce to 12% in redroot pigweed, both under hydromulch containing rice husk. Moreover, their great ability to control the weed seed bank in the short and the long term was the main finding of the environmentally controlled experiment, because a large fraction of the seeds that failed to germinate underneath the hydromulches went into secondary dormancy or lost viability. In particular, the most promising results were obtained with large crabgrass underneath hydromulch containing wheat straw: only 11% of the seeds were able to germinate and pass through the mulch layer, 20% of the seeds germinated but were unable to emerge, and 69% went into secondary dormancy.

Declaration of Competing Interest

Authors declare no conflicts of interest.

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