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3 **“The use of wet-laid techniques to obtain flax nonwovens with different**
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5 **thermoplastic binding fibers for technical insulation applications”**
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9
10 **Abstract**

11 In this work, the wet-laid technique has been used to obtain flax nonwovens
12 thermally-bonded with different contents of polyvinyl alcohol (PVA) and bicomponent
13 polyamide 6/copolyamide (PA6/CoPA) fibers in the 10-30 wt. % range. Morphology of
14 nonwovens has been studied by scanning electron microscopy (SEM) to evaluate
15 formation of interlock points through melted polymer and flax fibers. Main physical
16 properties (thickness and surface mass) have been determined by standardized tests.
17 Tensile strength and elongation at break have been determined with standard test
18 procedures on longitudinal (preferential deposition direction in hydroformer station) and
19 transversal directions to evaluate anisotropic behavior of nonwovens. The sound
20 absorption properties of stacked sheets of flax:PVA and flax:PA6/CoPA nonwovens
21 have been evaluated with an impedance tube by determining the absorption coefficient
22 in terms of the sound frequency. In addition, thermal insulating properties of individual
23 nonwovens have been tested with the heat flow meter method. Mechanical
24 characterization shows slight anisotropy since higher tensile strength values are
25 obtained in the longitudinal (preferential) direction. The absorption coefficient is
26 interesting in the medium frequencies range and relatively low thermal conductivity and
27 thermal resistance values are obtained with these nonwovens. By taking into account
28 these features, these nonwoven substrates could find interesting applications as sound
29 absorbers and/or thermal insulation materials in technical applications.
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53 **Keywords:** fiber, yarn, fabric formation; environmental sustainability; properties;
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5 **1. Introduction.**
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In the last years, a growing interest on the use of biobased materials or materials from renewable resources has been detected. This interest has also arrived to technical sectors such as building, automotive, transportation, etc. industries due to increasing environmental concerns, potential biodegradability and use of overall eco-friendly materials. Some of the requirements of these technical sectors are focused on comfort and this means that base materials must provide good insulation behavior, both thermal and acoustic^{1, 2}. Mineral and synthetic polymer wools are interesting substrates which are able to offer easy handling and good insulation properties due to the internal structure, therefore their use in building industry and other technical sectors has been generalized³⁻⁷; nevertheless, new materials from renewable resources are being demanded since they could offer similar behavior than traditional heat and sound absorbers, so that, important efforts on the development of natural (plant or animal derived) materials are being done.

In the last decade, a growing use of natural fibers such as hemp, kenaf, pineapple, abaca, flax, coir, etc. and other biobased fibrous components in the form of nonwovens or short fibers has been detected and research on this field is increasing⁸⁻¹⁰. These natural fibers could find potential uses as technical textiles due to excellent balanced properties¹¹. The potential of natural fiber nonwovens opens a wide variety of new applications in the textile industry, for example as biodegradable wipes¹² and also in other technical sectors such as automotive^{7, 13, 14}. Nonwoven structures can be used in a flexible form as thermal and acoustic insulation materials in automotive interior parts as lining materials due to particular shape and internal structure of natural fibers¹⁵⁻¹⁸. In addition, hybrid nonwovens with natural fibers and small amounts of binding

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3 fibers can lead to obtaining base materials for composite production by hot-press
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5 molding as rigid interior panels in automotive and transportation industries with
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7 balanced mechanical performance and acoustic and thermal insulation behavior ¹⁹. In
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9 this field, interesting results have been obtained by using bicomponent fibers in which,
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11 the sheath can be melted by temperature to promote a homogeneous matrix thus leading
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13 to composites with interesting applications in technical sectors²⁰⁻²². The use of
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15 nonwovens is also generalized as filtering media and new substrates based on natural
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17 fibers are being developed^{23, 24}.
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21 The wet-laid process, a widely used process in the paper industry, represents an
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23 interesting technology to obtain nonwoven structures based on different base
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25 components both raw and waste materials. Short fibers from microns up to 15-20 mm
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27 can serve as base materials for nonwoven production in a continuous way ²⁵.
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29 Furthermore, hybrid nonwoven structures can be obtained by combining different fibers
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31 so that it is possible to mix a base natural fiber with a binding fiber to provide cohesion
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33 after a thermo-bonding process²⁶⁻²⁸. The wet-laid process, which uses highly diluted
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35 fiber-water dispersions, is an eco-friendly process since, although it consumes high
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37 water amounts, all the water is recirculated since it only acts as the fiber carrier
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39 component so that, almost all water is recovered in the hydroformer station in which
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41 nonwoven formation occurs²⁹.
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46 The main aim of this work is to determine the potential of flax-based nonwovens
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48 obtained by the wet-laid process as candidate materials for thermal and acoustic
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50 insulation applications. To provide cohesion on nonwovens, different thermoplastic
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52 binder fibers have been used: polyvinyl alcohol (PVA) as fully biodegradable
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54 thermoplastic fiber and a typical binder bicomponent polyamide6/copolyamide
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56 (PA6/CoPA) fiber. Optimum processing conditions in the-wet laid process are
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3 determined. The morphology of nonwovens is characterized by scanning electron
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5 microscopy (SEM). The main physical properties such as thickness and surface mass
6
7 are determined by standardized tests. In addition, mechanical performance on
8
9 longitudinal (preferential formation direction in the hydroformer station) and transversal
10
11 directions is evaluated by determining tensile strength and elongation at break in terms
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13 of the binder fiber type and content. The potential of these nonwovens as sound
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15 absorber and/or thermal insulation materials is determined with standardized tests using
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17 an impedance tube to determine the absorption coefficient and a heat flow meter
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19 apparatus to determine the thermal conductivity and thermal resistance.
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27 **2. Experimental.**

28 **2.1. Materials.**

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31 Commercial flax fibers F 513/6 supplied by STW Fibres were used as base
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33 fibres for nonwoven formation with technical flax fibres (50-100 μm thickness) and
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35 elementary fibres with a thickness comprised between 10-20 μm . General properties of
36
37 flax fibres are summarized in Table 1.
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43 **Table 1**

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47 The thermo-bonding process was carried out with different thermoplastic fibres:
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49 polyvinyl alcohol (PVA) and a bicomponent fibre with a polyamide 6 (PA6) core
50
51 sheathed by a copolyamide (CoPA).
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55 PVA fibres, commercial grade PVA 401/6, were supplied by STW Fibres. The
56
57 degradation temperature is above 240 °C and the softening temperature is close to 120
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3 °C. The highly hydrophilic nature of these fibres enables good dispersion in aqueous
4
5 media. With regard to the bicomponent fibre, a commercial grade Grilon® BA 140
6
7 supplied by EMS-GRILTECH was used. The core (50 wt. %) consists on polyamide 6
8
9 (PA6) polymer with a melt point of 222 °C while the sheath (50 wt. %) is composed of a
10
11 low melt point (135 °C) copolyamide (CoPA).
12

13 14 15 16 **2.2. Formation of non-woven flax-thermoplastic fiber substrates by wet-laid process.**

17 18 *2.2.1. Weighting and fibre separation.*

19
20 In a first stage, the appropriate fibre amounts are weighted in order to obtain
21
22 different flax/thermoplastic binder ratios: 90/10, 80/20 and 70/30.
23

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25 Initially, fibres are separated in water by using a high shear pulper (*PILL*
26
27 *NASSVLIESTECHNIK GmbH WET-LAID NONWOVEN TECHNOLOGY*) with a total
28
29 capacity of 35 L. The fibre concentration in the water dispersion is maintained at 10 g L⁻¹
30
31 and the mixing process has been carried out with strong agitation at 2300 rpm for 10
32
33 min.
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36 37 38 *2.2.2. Maintenance of fibre dispersion in water.*

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40 After the initial separation and subsequent dispersion in water, the water-flax
41
42 dispersion is dropped into a dispersion tank with total capacity 1200 L in which, the flax
43
44 dispersion is diluted to a concentration of 1 g L⁻¹ and it is subjected to less vigorous
45
46 agitation at 170 rpm to maintain good fibre dispersion for a total time of 15 min.
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50 51 52 *2.2.3. Nonwoven formation.*

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54 Once fibres are appropriately dispersed in aqueous solution the water-fibre
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56 mixture is moved to the hydroformer station by using hydraulic pumps. The
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3 hydroformer station has been supplied by PILL NASSVLIESTECHNIK GmbH and it is
4
5 constructed in stainless steel with different polycarbonate windows to observe the
6
7 process evolution. The maximum width is 510 mm, the take-off angle is 20 ° and the
8
9 conveyor speed can vary between 1 and 10 m min⁻¹. Two different tanks are used to
10
11 ensure optimum dispersion conditions; when one of these two tanks is used, the other is
12
13 empty and *vice versa*. The water-fibre dispersion is pumped to the hydroformer station
14
15 as observed in Scheme 1, but before reaching to the forming strip, the water-fibre
16
17 dispersion is diluted once again up to a final concentration of 0.33 g L⁻¹. Then the
18
19 water-fibre dispersion is dropped onto the porous forming strip which acts as a filter
20
21 media in which water is removed by vacuum and fibres are deposited.
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27 **Scheme 1**

31 *2.2.4. Nonwoven consolidation*

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34 Consolidation of flax nonwovens has been carried out by a thermo-bonding
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36 process in two stages; a first drying stage with hot air followed by a second thermo-
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38 bonding process in a hot calender. The drying module (SDT-600) and the calendaring
39
40 equipment (CL-600) have been supplied by TALLERES TACOME S.A. The main
41
42 parameters for the drying and calendaring of nonwoven substrates are summarized in
43
44 Table 2. Finally, nonwovens are rolled in a roller module (EN-600) supplied by
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46 TALLERES TACOME S.A.
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51 **Table 2**

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3 **2.3. Morphology characterization by SEM.**
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5 Surface morphology of PVA and PA6/CoPA thermo-bonded flax nonwovens
6
7 has been evaluated by scanning electron microscopy with a Quanta 200SEM at a
8
9 voltage of 25 kV. Different images at 200x and 400x have been recorded without
10
11 previous sputtering.
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16 **2.4. Determination of thickness and surface mass.**
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18 Thickness of nonwovens has been measured with a Sodemat thickness apparatus
19
20 with a test pressure of 0.5 KPa, following the guidelines of the ISO 9073-2:1997.
21
22 Surface mass was measured on 1 dm² shapes obtained by cutting nonwovens with a
23
24 circular die mod. 1.333 RS supplied by Horvecal S.A. Five different samples have been
25
26 cut and weighed in a precision balance model GR-200-EC by A&D Instruments Ltd.
27
28 Temperature and relative humidity have been maintained at 20±2 °C and 65±4 %
29
30 respectively for both tests.
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36 **2.5. Characterization of mechanical properties.**
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38 Tensile properties (both longitudinal and transversal) of nonwovens have been
39
40 determined by following the guidelines of the ISO 29073-3:1993 with an Instron
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42 dynamometer model 4501 (Instron, Barcelona, Spain). The clamp distance was set to
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44 200 mm and the crosshead rate was 100 mm min⁻¹. At least five samples were tested
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46 and average values for tensile strength and elongation at break were calculated.
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48 Temperature and relative humidity have been maintained at 20±2 °C and 65±4 %
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50 respectively.
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3 **2.6. Thermal and acoustic insulation characterization.**
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5 The thermal insulation properties have been evaluated by determining the
6 thermal conductivity and thermal resistance according to the guidelines of the ISO
7 8301:1991 standard with a heat flow meter apparatus HFM 436 Lambda supplied by
8 NETZSCH. In this method, samples sizing 30x30 cm² are placed between a hot and a
9 cold plate and the heat flow created by the well-defined temperature difference is
10 measured with a heat flux sensor thus allowing the calculation of both thermal
11 conductivity and thermal resistance.
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20 Acoustic insulation properties have been evaluated on nonwovens with a
21 flax:binder fiber ratio of 80:20, by measuring the absorption coefficient of 12 stacked
22 nonwoven substrates by following the guidelines of the ISO 10534-2:2002 standard
23 with an impedance tube kit Type 4206 supplied by Brüel&Haer. A double channel
24 Symphonie analyzer with FFT has been used with 1/2 in and 1/4 in microphones. The
25 experimental frequency has varied in the 50 - 6400 Hz with a step of 2 Hz.
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36 **3. Results and discussion.**
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38 **3.1. Morphology of flax nonwovens thermo-bonded with PVA and COPA fibres.**
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40 SEM images of flax nonwovens thermally bonded with different amounts of
41 PVA fibers do not show clear melted PVA areas (Fig. 1). As we have described
42 previously, the softening point of PVA is close to 120 °C. The thermal-bonding process
43 has been carried out at 195 °C but even the use of this relatively high drying temperature
44 is not enough to fully melt PVA fibers to embed flax fibers. This phenomenon could be
45 related to the fact that PVA fibers absorb water (up to 4-6 wt. %) so that, it is necessary
46 the use of higher temperatures or longer drying times to fully melt PVA polymer. This
47 morphology would play a key role on overall mechanical performance of flax:PVA
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3 nonwovens; but even in this case, good thermal and acoustic insulation properties can
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5 be achieved since softened PVA fibers can interact with some flax fibers.
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8 9 **Figure 1**

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14 With regard to flax nonwovens with PA6/CoPA bicomponent fibers as binder
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16 (Fig. 2), we can clearly detect a decrease in porosity as the total amount of PA6/CoPA
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18 fiber increases. We can also observe the typical shape and geometry of individual flax
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20 fibers as they show a wide range of variability in diameter between 10 – 500 μm . As we
21
22 have described before, the sheath is based on a low melt point copolyamide (CoPA)
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24 with a melt point close to 135 °C; so that, the use of temperatures close to 195 °C during
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26 drying and subsequent calendering is enough to fully melt the sheath which represents
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28 50 wt. % of total binder fiber. So that, the real binder fiber content (CoPA) is around 5,
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30 10 and 15 wt. %, corresponding to total PA6/CoPA contents of 10, 20 and 30 wt. % on
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32 flax nonwovens. We can clearly observe presence of binding areas characterized by
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34 partially embedded flax fibers. As CoPA melts it moves over individual flax fibers thus
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36 embedding them. These melted areas are responsible for formation of interlock points
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38 which can provide higher mechanical resistance than previously described flax:PVA
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40 nonwovens.
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47 48 **Figure 2**

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52 The binding structure of thermally-bonded nonwovens highly depends on the
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54 heat transfer method as well as the web structure and the nature of the binding fiber. By
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56 using thermal calendering, heat transfer is achieved by conduction phenomena; this
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3 produces the melt of the CoPA sheath on PA6/CoPA fibers. The melted polymer which
4 covers the PA6 core fiber can also partially embed flax fibers due to the high mobility
5 of melted polymer and this embedded fibers form flax-flax and flax-PA6 interlock
6 points which will have a positive effect on mechanical resistance of nonwovens. As a
7 result, flax:PA6/CoPA nonwovens are characterized by a more stable structure since the
8 core remains solid during the drying-calendering process while the sheath melts at
9 135°C. Then, the melted sheath acts as fusible adhesive and it can be distributed very
10 finely throughout the supporting PA6 and flax fibers and the solid PA6 core provides
11 strong support for nonwovens both during and after bonding.
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23 As the real binding component (CoPA) represents only 5, 10 and 15 wt. % it is
24 homogeneously distributed over PA6 supporting fibers but in addition, the melted
25 CoPA polymer can adhere to nearby flax fibers, thus acting as a bridge to form flax-flax
26 and flax-PA6 interlock points. Nonwovens with a PA6/CoPA content of 30 wt. % (15
27 wt. % CoPA binder) are characterized by a homogeneous flax:PA6 nonwoven structure
28 homogeneously embedded in a CoPA matrix (Fig. 2e and 2f).
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39 ***3.2. Physical and mechanical properties of flax nonwovens thermally-bonded with*** 40 ***different thermoplastic fibers.*** 41

42 With regard to flax:PVA nonwovens, the surface mass is close to 400 g m⁻² with
43 relatively low variability (Table 3). Higher values are obtained for flax:PVA with 30 wt.
44 % PVA binder fiber with surface mass values of about 465 g m⁻². This is typical
45 variability of nonwovens due to random deposition of fibers during nonwoven
46 formation. In general terms, dispersion is relatively low between 4-7 %. With regard to
47 nonwovens with bicomponent PA6/CoPA binder fiber, the surface mass is similar for
48 different PA6/CoPA contents as observed in Table 3. Surface mass values are in a
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3 narrow range between 415-427 g m⁻² with low standard deviation which represents a
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5 percentage variation of less than 3.5 %.
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8 9 10 **Table 3**

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14 Thickness of flax:PVA nonwovens is in the 1.5-2.0 mm range as observed in
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16 Table 4. Nevertheless we observe relatively high dispersion values (between 10 – 16 %)
17
18 due to the nature of the deposition process on the hydroformer station. Regarding to
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20 flax:PA6/CoPA nonwovens, the average thickness is close to 2 mm with lower
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22 variability. In both cases, an average thickness of 2 mm can be achieved with the wet-
23
24 laid process.
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28 29 30 **Table 4**

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34 Once surface mass and thickness values have been described, we proceed with
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36 mechanical properties of flax nonwovens thermally bonded with PVA and PA6/CoPA
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38 binding fibers. In the case of flax:PVA nonwovens, the previous SEM study has
39
40 revealed low interaction between flax and melted PVA fibers. Thus, relatively poor
41
42 mechanical resistance can be expected. Fig. 3 shows a comparative plot of the
43
44 longitudinal tensile strength of flax nonwovens with PVA and PA6/CoPA binding
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46 fibers. With regard to flax:PVA, we can see that tensile strength is relatively low with
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48 values below 100 N. Also, as the PVA binder fiber increases, the tensile strength even
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50 decreases thus indicating poor interaction after the thermal-bonding process (91, 72 and
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52 61 N for 10, 20 and 30 wt. % PVA binder fiber respectively). These results are in total
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54 agreement with microstructure studied by SEM which has revealed absence of melted
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3 PVA fibers so that, flax fibers can't be fully embedded by PVA polymer. With regard to
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5 flax nonwovens thermally bonded with PA6/CoPA bicomponent fiber, tensile strength
6
7 is highly dependent on total content of binding fiber. Therefore, the tensile strength
8
9 changes from values around 174 N for 10 wt. % PA6/CoPA fiber (real binder content of
10
11 5 wt. % CoPA) up to values of about 489 N for 30 wt. % PA6/CoPA fiber (real binder
12
13 content of 15 wt. % CoPA polymer). Nevertheless, it is important to remark that the
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15 only binding component is the CoPA sheath with a melting point at 135 °C while the
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17 PA6 core fiber remains solid and contributes to improve mechanical performance of
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19 nonwovens with flax.
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25 **Figure 3**

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30 As the hydroformer station is characterized by a preferential nonwoven
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32 formation direction, the transversal mechanical response has also been evaluated. Fig. 4
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34 shows a comparative plot of the tensile strength (transversal direction) for flax
35
36 nonwovens with PVA and PA6/CoPA binder fibers. In the case of flax:PVA
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38 nonwovens, we observe similar tendency than observed for longitudinal direction but
39
40 even lower values are obtained so that indicating that better mechanical properties are
41
42 obtained in the preferential formation direction. The tensile strength is lower than 72 N
43
44 and an increase in PVA binder content doesn't lead to improvement on mechanical
45
46 response due to the lack of interaction between PVA and flax fibers. Nevertheless, when
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48 using PA6/CoPA bicomponent fiber, we observe a clear increasing tendency on tensile
49
50 strength values as the binder content increases. Nonwovens with 10 wt. % PA6/CoPA
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52 (5 wt. % CoPA binder) show a transversal tensile strength of 145 N and this is increased
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54 up to 341 N for nonwovens with 30 wt. % PA6/CoPA fiber (15 wt. % CoPA binder). If
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3 we compare transversal mechanical response of flax:PA6/CoPA to longitudinal
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5 response, we also corroborate that the longitudinal (preferential) direction provides
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7 better mechanical properties since the intrinsic nature of the hydroformer promotes
8
9 slight higher fiber alignment on longitudinal direction. Once again, these results are in
10
11 total agreement with those predicted by SEM study since flax:PA6/CoPA nonwovens
12
13 are characterized by presence of low melt point CoPA polymer which melts during
14
15 drying and subsequent calendering to act as a bridge between flax fibers and flax-PA6
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17 core fibers and these bridges act as interlock points; so that, as the total content on
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19 CoPA binder increases, mechanical performace is improved.
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25 **Figure 4**

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29 With regard to elongation, Fig. 5 and Fig. 6 show a plot comparison between
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31 elongation at break values for flax nonwovens with different binder fibers for
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33 longitudinal (Fig. 5) and transversal (Fig. 6) directions. In general terms, elongation at
34
35 break is representative for nonwoven cohesion. We can clearly observe that flax:PVA
36
37 nonwovens show relatively low elongation at break values, around 2% for both
38
39 longitudinal and transversal directions while elongation at break of flax:PA6/CoPA
40
41 nonwovens is located around 5%. This is in accordance with the SEM study. Flax:PVA
42
43 nonwovens are characterized by absence of interlock points since PVA doesn't melt
44
45 appropriately during the drying-calendering stage, so that when subjected to tensile
46
47 stress, the overall cohesion can be easily lost with relatively low elongation at break
48
49 values. On other hand the SEM study has revealed the melt of CoPA sheath thus
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51 allowing flax-flax and flax-PA6 interactions which lead to high cohesion on
52
53 flax:PA6/CoPA nonwovens; therefore, higher elongation at break values are needed in
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3 order to break the highly crosslinked structure. In the case of elongation at break we do
4
5 not observe differences between longitudinal and transversal directions and furthermore,
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7 we don't see differences in terms of the total binder content, so we can conclude that
8
9 elongation at break is not a sensitive property to total binder content.
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14 **Figure 5**

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16 **Figure 6**
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21 ***3.3. Thermal and acoustic insulation properties of flax nonwovens thermally-bonded***
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23 ***with different thermoplastic fibers.***
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25 The increasing use of nonwovens in technical applications requires, in most
26
27 cases, good thermal and acoustic insulation properties for comfort. For this reason, both
28
29 thermal and acoustic insulation behavior of flax:PVA and flax:PA6/CoPA nonwovens
30
31 has been evaluated. With regard to acoustic insulation, as the total thickness of
32
33 nonwovens is relatively low (in the 1.5-2.0 mm range), the acoustic insulation
34
35 properties of twelve stacked substrates has been evaluated in a Kundt's tube. Fig. 7
36
37 shows a plot evolution of the acoustic absorption coefficient in terms of the sound
38
39 frequency for flax:PVA and flax:PA6/CoPA nonwovens. We can see that acoustic
40
41 absorption at low frequencies (below 300 Hz) is very low, with absorption coefficients
42
43 in the 0.05 – 0.2 range; in this frequency range, flax:PA6/CoPA show slightly higher
44
45 acoustic insulation properties than flax:PVA nonwovens, but differences are not
46
47 significant. In the medium frequencies range we can observe some interesting
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49 differences. With regard to flax:PVA nonwovens, the absorption coefficient increases
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51 up to values of about 0.4-0.5 in the frequencies range comprised between 300 Hz - 2
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53 kHz, thus indicating interesting and quite homogenous acoustic insulation properties in
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3 this range. Regarding to flax:PA6/CoPA nonwovens, we observe a remarkable increase
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5 in absorption coefficient up to almost 0.6 at about 500-600 Hz, but it decreases up to
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7 values around 0.2 - 0.3 in the 1-2 kHz range. In the high frequencies range (over 2 kHz),
8
9 once again flax:PVA nonwovens show higher acoustic insulation properties than
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11 flax:PA6/CoPA. In general terms we observe good acoustic insulation properties for
12
13 both flax:PVA and flax:PA6/CoPA nonwovens in the medium frequencies range;
14
15 therefore these nonwoven structures show attracting properties for technical
16
17 applications as acoustic absorbers.
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23 **Figure 7**

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27 In addition to acoustic insulation properties, thermal conductivity has also been
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29 determined by the heat flow meter method. Table 5 shows summarized results of
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31 thermal conductivity and thermal resistance for different contents on PVA and
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33 PA6/CoPA binder fibers. Thermal conductivity of flax:PVA nonwovens is relatively
34
35 low, as expected, due to the high thermal insulation properties of flax fibers.
36
37 Specifically, thermal conductivity is lower than $0.024 \text{ W (m K)}^{-1}$ for all flax:PVA
38
39 compositions considered in this work. This fact is interesting since a thermal insulating
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41 material is characterized by thermal conductivity values lower than $0.060 \text{ W (m K)}^{-1}$;
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43 therefore, flax:PVA fulfill these requirements. Despite this, a thermal insulating
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45 material is also characterized by a thermal resistance higher than $0.25 \text{ m}^2 \text{ K W}^{-1}$. So
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47 that, as thermal resistance values change in the $0.060 - 0.075 \text{ m}^2 \text{ K W}^{-1}$, this means that
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49 a minimum of 3-4 stacked flax:PVA nonwovens would be necessary to fully consider
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51 this material as a thermal insulating component. With regard to flax:PA6/CoPA
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53 nonwovens, the thermal conductivity is higher with values in the $0.090 - 0.109 \text{ W (m}$
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3 K)⁻¹. Regarding to thermal resistance, values ranging from 0.012 to 0.018 m² K W⁻¹ are
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5 obtained and this means that a minimum of 15-20 stacked nonwoven substrates must be
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7 used to obtain fully thermal insulating behavior. So that, we can conclude that thermal
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9 insulating properties of flax:PVA nonwovens are better than flax:PA6/CoPA materials.
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14 **Table 5**

18 **4. Conclusions.**

20 The use of the wet-laid technique to obtain flax-based nonwovens with different
21 binder thermoplastic fibers has been validated. Thermo-bonding process with PVA
22 binder fibers at 195 °C leads to partially bonded flax:PVA nonwovens as observed by
23 SEM study, since this temperature is not enough to provide full melting but the use of
24 higher temperatures could lead to PVA degradation. This fact is responsible for
25 mechanical performance of these nonwovens characterized by almost constant tensile
26 strength values independently of the PVA binder fiber content (in the 10 – 30 wt. %
27 range). The use of bicomponent PA6/CoPA binder fibers with PA6 core with a melt
28 point of 222 °C and a CoPA sheath with a low melting point located at 135 °C, allows
29 full sheath melting and therefore, flax fibers can be embedded by melted CoPA
30 polymer. This allows flax-flax and flax-PA6 interactions which are responsible for
31 mechanical performance enhancement. In this case, a remarkable increase in tensile
32 strength with the total content of PA6/CoPA binder fiber has been observed. On other
33 hand, as the hydroformer station is characterized by a preferential deposition direction,
34 some anisotropy on nonwovens has been detected since tensile strength values are
35 higher in the longitudinal (preferential) direction than transversal direction.
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3 Although flax:PVA nonwovens show lower mechanical performance than
4 flax:PA6/CoPA substrates, the acoustical insulation properties (measured through the
5 absorption coefficient) are better and more homogeneous for flax:PVA nonwovens in
6 the middle frequencies range. With regard to thermal insulating properties, both
7 flax:PVA and flax:PA6/CoPA nonwovens offer interesting properties but once again,
8 flax:PVA nonwovens possess lower thermal conductivity and good thermal resistance.
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16 In general terms we can conclude that the wet-laid technique is useful to obtain
17 nonwovens from natural flax fibres with different binder thermoplastic fibers. Although
18 some anisotropy is detected, due to intrinsic preferential deposition direction,
19 mechanical performance is enough to ensure good handling. In addition to this,
20 interesting acoustic and thermal insulation properties can be obtained by stacking
21 different sheets thus allowing the use of these materials as technical substrates for sound
22 absorption or thermal insulation applications.
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34 **Acknowledgements**

35
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Implementación de la tecnología wet-laid en la investigación y desarrollo de paneles para aplicaciones técnicas a partir de residuos procedentes de la industria textil.” with expedient number IMDEEA/2011/167 (total aid of 255000 euro) funded by IMPIVA and cofunded (80%) by the European Union through FEDER funds, Valencian Community Operational 2007-2012.

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5 **Figure legends**
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7 **Scheme 1.-** General scheme of the wet-laid process with a water-fiber dispersión and
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9 water recovery system.
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11 **Figure 1.-** SEM images of flax nonwovens thermally bonded with PVA in different
12 flax:PVA wt. % ratio and different magnification. a) 90:10, x200; b) 90:10, x400; c)
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14 80:20, x200; d) 80:20, x400; e) 70:30, x200; f) 70:30, x400.
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18 **Figure 2.-** SEM images of flax nonwovens thermally bonded with PA6/CoPA in
19 different flax:PA6/CoPA wt. % ratio and different magnification. a) 90:10, x200; b)
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21 90:10, x400; c) 80:20, x200; d) 80:20, x400; e) 70:30, x200; f) 70:30, x400.
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25 **Figure 3.-** Longitudinal tensile strength of flax nonwovens thermally-bonded with PVA
26 and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
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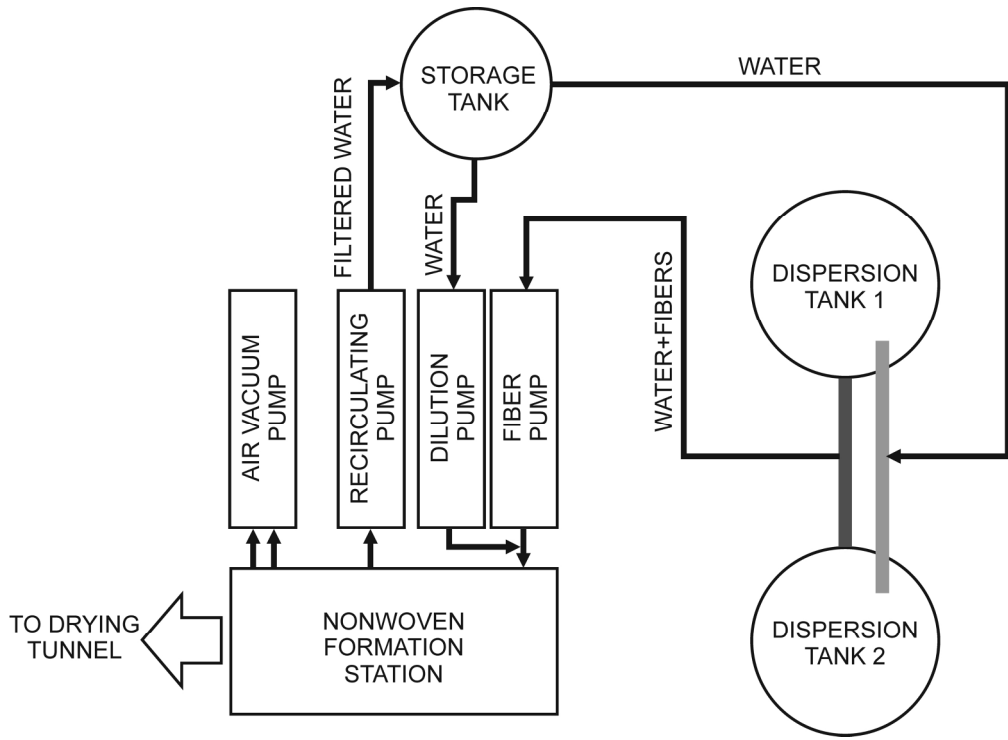
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30 **Figure 4.-** Transversal tensile strength of flax nonwovens thermally-bonded with PVA
31 and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
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35 **Figure 5.-** Longitudinal elongation at break of flax nonwovens thermally-bonded with
36 PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
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40 **Figure 6.-** Transversal elongation at break of flax nonwovens thermally-bonded with
41 PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
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44 **Figure 7.-** Plot evolution of the absorption coefficient of flax nonwovens thermally-
45 bonded with PVA and PA6/CoPA fibers (flax:binder wt. ratio = 80:20) in terms of the
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47 frequency.
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Scheme 1.- General scheme of the wet-laid process with a water-fiber dispersion and water recovery system.
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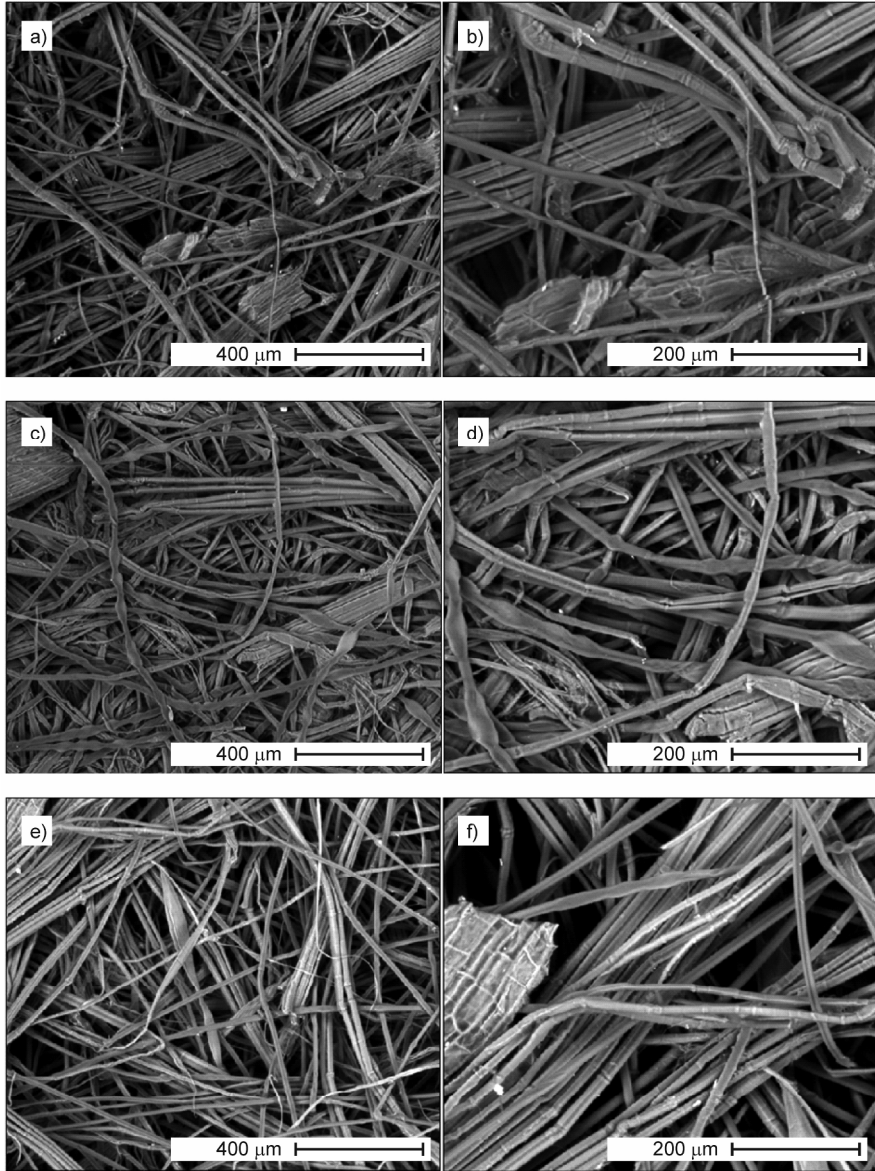


Figure 1.- SEM images of flax nonwovens thermally bonded with PVA in different flax:PVA wt. % ratio and different magnification. a) 90:10, x200; b) 90:10, x400; c) 80:20, x200; d) 80:20, x400; e) 70:30, x200; f) 70:30, x400.
260x348mm (300 x 300 DPI)

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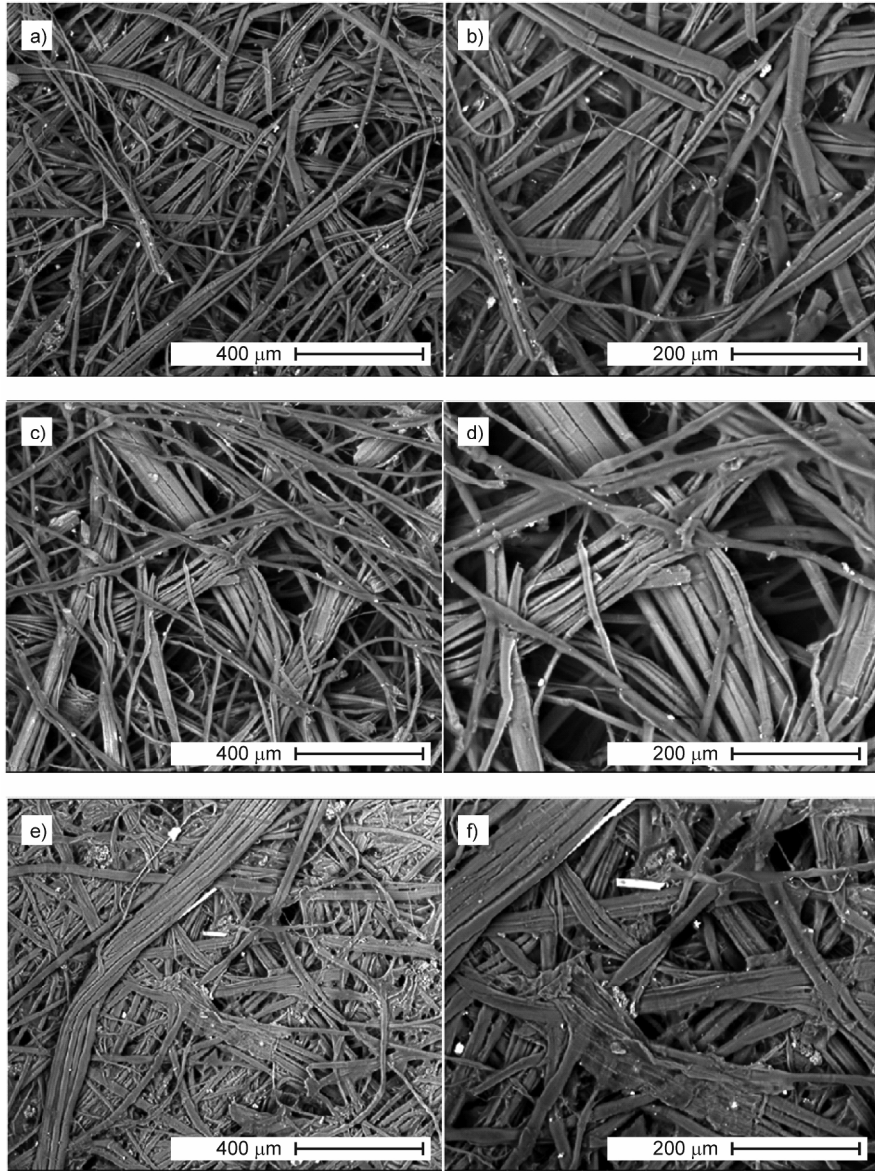


Figure 2.- SEM images of flax nonwovens thermally bonded with PA6/CoPA in different flax:PA6/CoPA wt. % ratio and different magnification. a) 90:10, x200; b) 90:10, x400; c) 80:20, x200; d) 80:20, x400; e) 70:30, x200; f) 70:30, x400.
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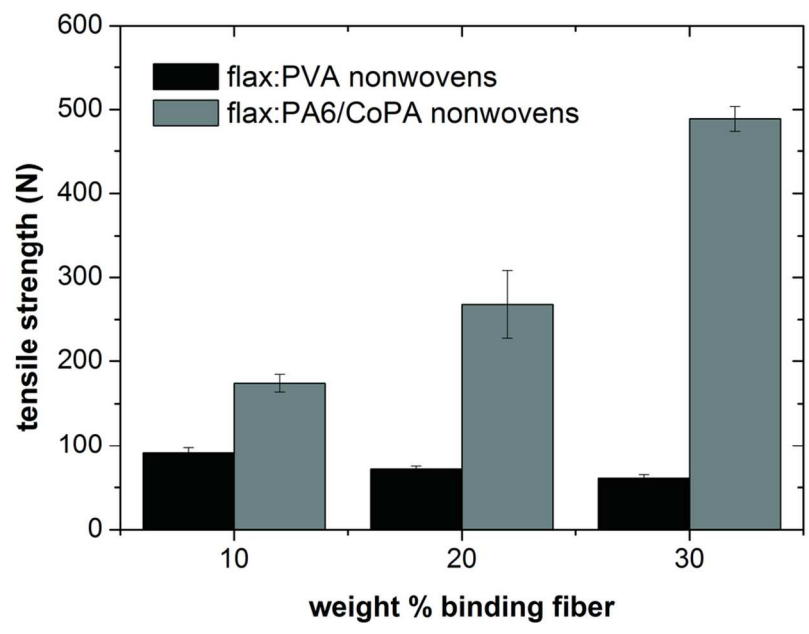


Figure 3.- Longitudinal tensile strength of flax nonwovens thermally-bonded with PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber. 53x37mm (600 x 600 DPI)

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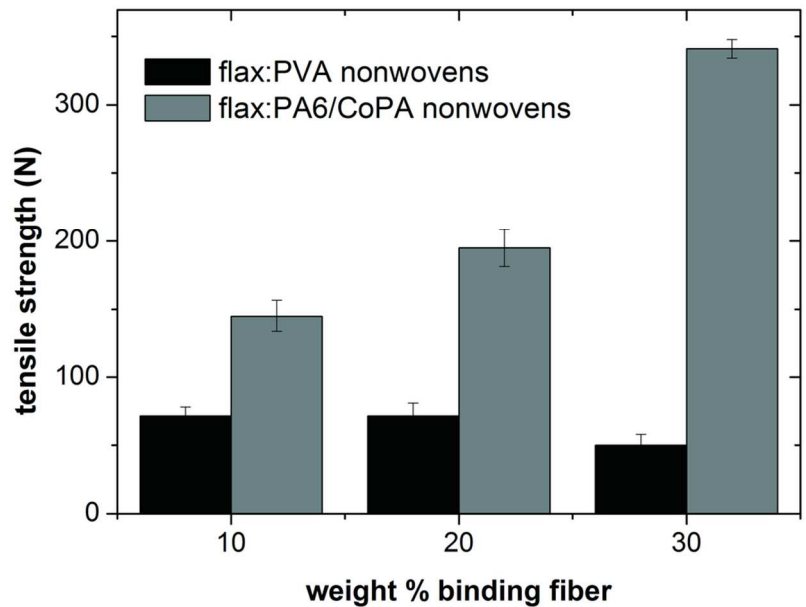


Figure 4.- Transversal tensile strength of flax nonwovens thermally-bonded with PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber. 53x37mm (600 x 600 DPI)

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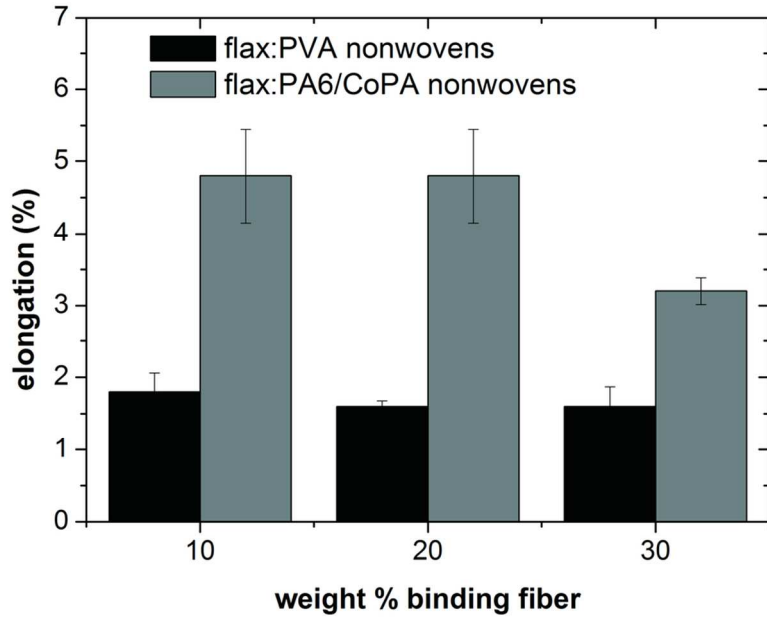


Figure 5.- Longitudinal elongation at break of flax nonwovens thermally-bonded with PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
53x37mm (600 x 600 DPI)

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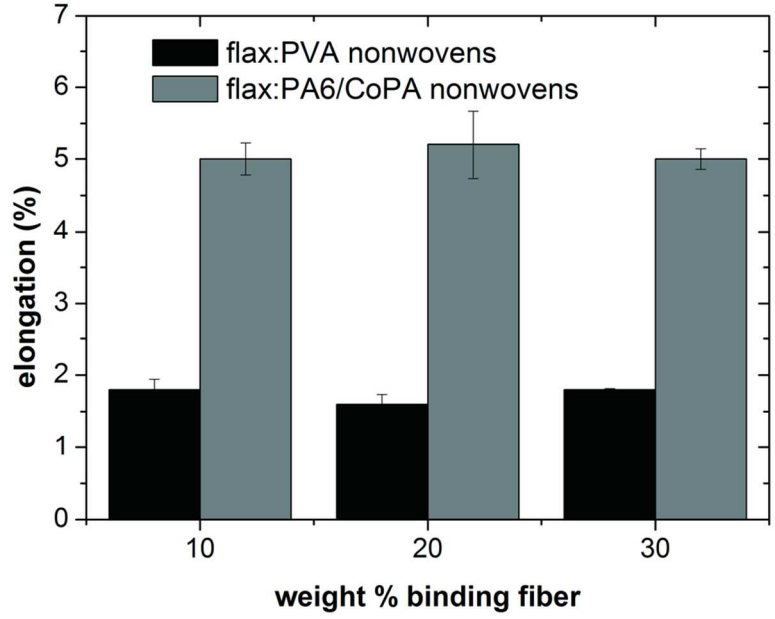


Figure 6.- Transversal elongation at break of flax nonwovens thermally-bonded with PVA and PA6/CoPA fibers in terms of the wt. % of the binding fiber.
53x37mm (600 x 600 DPI)

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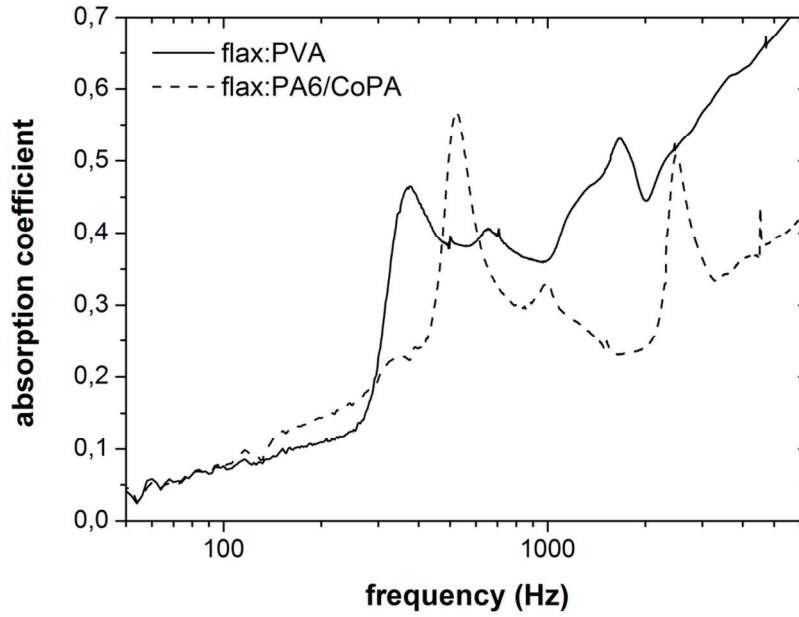


Figure 7.- Plot evolution of the absorption coefficient of flax nonwovens thermally-bonded with PVA and PA6/CoPA fibers (flax:binder wt. ratio = 80:20) in terms of the frequency. 53x37mm (600 x 600 DPI)

Table 1.- General characteristics of flax fibers used as base fibers for nonwoven formation by wet-laid process on a hydroformer station.

Transversal section	Polygonal with oval lumen
Thickness [μm]	10 – 500
Density [g cm^{-3}]	1.4 – 1.5
Resistance to constant heat [$^{\circ}\text{C}$]	75 - 80
Decomposition temperature [$^{\circ}\text{C}$]	300
Moisture content [%]	15
Color	Natural
Fiber length [mm]	6
Light resistance	Moderate
Resistance to acids	Moderate
Resistance to alkalis	Moderate
Water swelling	Moderate

Table 2.- Processing parameters in the drying and calendering stages for flax nonwovens thermally bonded with PVA and PA6/CoPA thermoplastic fibers.

Drying stage	Temperature at the drying module (°C)	195
	Temperature at the boiler (°C)	250
	Drying time (min)	15
Calendering stage	Distance between calendering rollers (mm)	0
	Pressure (bar)	40
	Temperature at the roller surface (°C)	200
	Linear pressure on nonwoven (N mm ⁻¹)	124

Table 3.- Average surface mass values of flax nonwovens thermally bonded with PVA and PA6/CoPA thermoplastic fibers.

Weight % flax:binder	flax:PVA fiber nonwovens		flax: PA6/COPA fiber nonwovens	
	Average	Standard deviation	Average	Standard deviation
90/10	394.0	16.8	415.4	14.4
80/20	404.2	23.8	419.0	8.6
70/30	465.4	35.1	426.8	9.0

Table 4.- Average thickness values of flax nonwovens thermally bonded with different thermoplastic fibers.

Weight %	flax:PVA fiber nonwovens		flax: PA6/COPA fiber nonwovens	
flax:binder	Average	Standard deviation	Average	Standard deviation
90:10	1.52	0.25	2.15	0.23
80:20	1.94	0.09	2.17	0.12
70:30	1.43	0.14	1.96	0.07

Table 5.- Thermal insulation properties of flax:PVA and flax:PA6/CoPA nonwovens in terms of the binder fiber content.

Weight % flax:binder	flax:PVA fiber nonwovens		flax: PA6/COPA fiber nonwovens	
	Thermal conductivity [W (m K)⁻¹]	Thermal resistance (m² K W⁻¹)	Thermal conductivity [W (m K)⁻¹]	Thermal resistance (m² K W⁻¹)
90/10	0.020	0.065	0.093	0.018
80/20	0.024	0.075	0.109	0.012
70/30	0.023	0.060	0.090	0.016