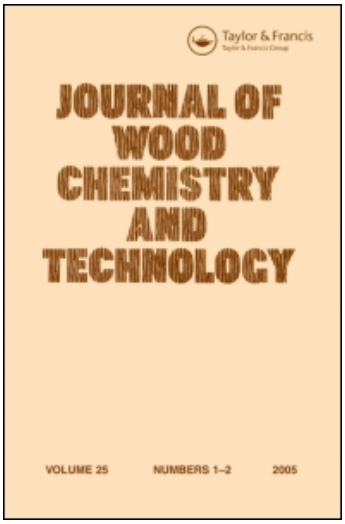

Journal of Wood Chemistry and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597282

Cynara cardunculus as Raw Material for the Production of Binderless Fiberboards: Optimization of Pretreatment and Pressing Conditions

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To cite this Article Mancera, C. , Ferrando, F. and Salvadó, J.(2008) '*Cynara cardunculus* as Raw Material for the Production of Binderless Fiberboards: Optimization of Pretreatment and Pressing Conditions', Journal of Wood Chemistry and Technology, 28: 3, 207 — 226
To link to this Article: DOI: 10.1080/02773810802347024
URL: http://dx.doi.org/10.1080/02773810802347024

Journal of Wood Chemistry and Technology, 28: 207–226, 2008

ISSN: 0277-3813 print / 1532-2319 online
DOI: 10.1080/02773810802347024

Cynara cardunculus as Raw Material for the Production of Binderless Fiberboards: Optimization of Pretreatment and Pressing Conditions

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Abstract: *Cynara cardunculus* was pretreated and used to produce fiberboards without synthetic adhesives. The lignocellulosic material was steam exploded through a thermomechanical vapor process in a batch reactor. After pretreatment the material was dried, ground, and pressed to produce the boards. The effects of pretreatment factors and pressing conditions on the chemical and physico-mechanical properties of the fiberboards were evaluated and the conditions that optimize these properties were found. Response surface methodology based on a central composite design and multiple response optimization were used. The variables studied and their respective variation ranges were: pretreatment temperature, 160–240°C; pretreatment time 2.5–12.5 min; pressing temperature, 190–230°C; initial and final pressing pressures, 4–20 MPa, and initial and final pressing times, 1–9 min. Good properties were obtained at optimum conditions found (modulus of elasticity up to 5970 MPa, modulus of rupture up to 59 MPa, internal bond up to 0.8 MPa, thickness swelling as low as13.5%, and water absorption as low as 18.5%). Some of the boards fully satisfy the standard specifications although they were not produced at the optimum combination of process factors. Optimum operational conditions for producing binderless fiberboards from *Cynara cardunculus* that fully satisfy the European standards were found based on multiple response optimization methodology.

The authors thank Universidad Politecnica de Madrid, Departamento de Produccion Vegetal: Botánica y Protección Vegetal, for supplying the *Cynara cardunculus* stalks. We also express our sincere appreciation to the Rovira i Virgili University for the award of a scholarship, the Spanish Ministry of Science and Technology for its financial support under project number ENE 2004-07624-C03-03, and the autonomous government of Catalonia for finance support to consolidated research groups, SGR 2005-00580.

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Keywords: Binderless fiberboards, cellulose, *Cynara cardunculus*, hemicellulose, lignin, steam explosion

ABBREVIATIONS

MOE: Modulus of elasticity; MOR: Modulus of rupture; IB: Internal bond; TS: thickness swelling; WA: water absorption; T_r: pretreatment temperature; t_r: pretreatment time; T_p: pressing temperature; P_{p_i}: initial press pressure; P_{p_f}: final press pressure ; t_{p_i}: initial pressing time; t_{p_f}: final pressing time.

INTRODUCTION

Depleting natural resources, regulations on synthetic materials, growing environmental awareness, and economic considerations are the main forces behind the drive to use annually renewable resources such as biomass for the production of binderless fiberboards. Using agricultural residues such as *Cynara cardunculus* stalks, which are recyclable and renewable, as raw materials helps to solve problems such as deforestation. Moreover, the current increase in fuel costs and the scarcity of petroleum sources is driving the search for natural substitutes for petroleum by-products such as synthetic adhesives used in the production of fiberboards. Because the binderless board process does not use resins of fossil origin, production costs are lower, no curing periods are needed, and the boards have no formaldehyde emissions as result of adhesive addition. Although the pretreatment process might involve an energy cost, the resulting composite material is friendlier to the environment[1,2] *Cynara* was chosen as raw material because this study is part of a bigger project in which integral valorization of this crop is sought, optimizing final applications for each part of the crop. These final applications are mainly: Biodiesel from *Cynara's* seed oil and solid biofuel, bioethanol, and binderless fiberboards production from its lignocellulosic biomass.

Steam explosion is one of the best ways of pretreating lignocellulosic materials for use in chemical fractionation, bioconversion, and the production of boards and composites because it preserves the fiber structure and separates the lignocellulosic material into its main components (cellulose, hemicelluloses, and lignin).[3,4] It has been claimed[5] that steam explosion plasticizes the lignin and separates the fibers, thus improving the bonding capacity of the material.

This study explores the suitability of steam exploded *Cynara cardunculus* for the production of fiberboards without synthetic binders and attempts to optimize the pretreatment and pressing conditions to achieve this aim.

EXPERIMENTAL

Raw Material Preparation

Cynara cardunculus stalks were obtained from an experimental plantation in Madrid, Spain[6] The material remained in contact with the surrounding atmosphere for a few months, stored in corrugated boxes, after being harvested and before being cleaned. After cleaning, the stalks were pithed and chipped into splinters of less than 5 cm. The average chemical composition of the initial material is shown in Table 1. The table shows that the sum of the chemical composition is more than 100% (106.4%). This is a common result due to the overlapping of the testing results.[7]

Steam Explosion

Cynara cardunculus chips, 150 g dry base per batch, were fed to the steam explosion reactor. The reactor is a stainless steel, cylindrical batch type reactor with a nominal capacity of 8L, 45 bars of pressure, and 250°C of temperature. The steam explosion reactor was design by the university staff and built by Justinox.

The chips were then treated with saturated steam at the desired conditions of temperature (between 160–240°C) and time (between 2.5–12.5 min). After the set time was reached, the chips were suddenly depressurized into a 100-liter recipient. Pulp obtained from this pretreatment was washed with water for clearing the liquor obtained in the pretreatment; this liquor contains extractives and hemicelluloses that are not convenient for binderless fiberboard processing. Finally, the pulp was air dried for one or two days until in equilibrium with the environment.

Table 1. Average chemical composition of *Cynara cardunculus* stalks

Fraction	%p/p (Dry solid bases)
Ash	5.4
Klason lignin	17.5
Acid soluble lignin	0.8
Cellulose	49.0
Hemicelluloses	24.0
Aqueous extractives	9.2
Organic extractives	0.5

Grinding

The pretreated pulps, with moisture content of between 8 and 10%, were ground to pass through a 4-mm sieve. Previous studies have shown that this procedure increases the bonding area and improves the internal strength.[8]

Pressing

The ground material was homogenized and its weight and relative humidity were measured. The material was then shaped into a forming box (150-mm long $\times$ 50-mm wide), which had previously been heated to the desired temperature, together with the press platens. The test boards were made with an objective thickness of 3 mm. After the material was placed into the mold, it was hot pressed in a three-stage cycle:

1. Pressing at the desired temperature and pressure for a given period of time.
2. A breathing period or pressure relaxation for 1 min.
3. Pressing at the desired temperature and pressure for a given period of time. Some of the pressing factors (pressure and time) in this third stage could be different from those in the first stage.

Physical and Mechanical Characterization

The boards were characterized using European standards. The mechanical properties measured were: modulus of elasticity (MOE) and modulus of rupture (MOR),[9] internal bond (IB).[10] Dimensional stability was characterized by measuring: thickness swelling (TS) and water absorption (WA).[11] Additionally, the density was determined.[12] Boards were conditioned at 20°C and 65% RH before any physical or mechanical test was conducted and the dimensions of test pieces were determined based on EN 325 standard.[13]

European standards for these properties are as follows: Density >800 Kg/m^3, MOR $\geq$ 40 MPa, MOE $\geq$ 3000 MPa, IB $\geq$ 0.7 MPa, WA $\leq$ 30%, and TS $\leq$ 20%.

Chemical Characterization

Original raw material and pretreated pulps were analyzed chemically to evaluate the pretreatment process. Standard ASTM methods were used for this aim, the chemical properties analyzed were: Humidity,[14] ash content,[15] aqueous extractives,[16] organic extractives,[17] and Klason lignin,[18] Carbohydrates from Klason lignin hydrolysis were analyzed by HPLC[19] to determine

Cellulose and Hemicelluloses content. Acid-soluble lignin was also analyzed for the original material by UV absorption.[20]

Experimental Design

Mechanical and Physical Properties

Response surface method was used, based on a central composite design to study the effect of 7 factors over 6 response variables in 48 tests; the design was run in a single block. These factors were: pretreatment temperature and time, pressing temperature, pressure and time for the first and third pressing steps. The responses were the physical and mechanical properties. The responses were analyzed using the software Statgraphics Plus 5.0.

Chemical Properties

To study the chemical properties part of the aforementioned design was used, but it was reduced to a 2^2 central composite design, which was orthogonal and rotatable and made up of 16 runs with 8 center repetitions.

RESULTS AND DISCUSSION

The results of the response surface design for physico-mechanical properties and chemical compositions are shown in Tables 2 and 3, respectively. An extra factor is included in these tables. This is the severity factor (log (ρ)),[21] which groups the vapor pretreatment temperature and time in a single variable, thus giving a weight for the severity of the global pretreatment. For each response variable, a variance analysis was performed at a confidence level of 95%.

Density

The model as fitted presents an R^2 of 0.925 and a standard deviation of the residuals (SDR) of 20.3 kg/m^3. Only four factors (pretreatment temperature, pretreatment time, pressing temperature, and initial pressing pressure) were found to be statistically important at a confidence level of 95%. The modeled response surface (Figure 1) shows that increasing the severity of the pretreatment, either by increasing the temperature or increasing the time, increases the density due to a reduction in the compression resistance of the *C. cardunculus*. The same results have been obtained with other materials.[22–24] Figure 1 also

Table 2. Physico-mechanical properties

	Process factors								Response variables					
Run	T_r [°C]	$t_{\_r}$ [min.]	Log (ρ)	T_p [°C]	P_{p_i} [MPa]	P_{p_f} [MPa]	t_{p_i} [min.]	t_{p_f} [min.]	Density [kg/m^3]	MOR [MPa]	MOE [MPa]	IB [MPa]	WA [%]	TS [%]
1	200	7.5	3.82	210	12	12	5	5	1337	41	5400	0.6	21.4	17.7
2	220	10	4.53	200	16	8	3	7	1342	41	5200	0.6	20.0	14.5
3	180	5	3.05	200	16	8	7	7	1289	27	4029	0.1	37.2	22.6
4	200	7.5	3.82	210	12	12	5	5	1344	39	4688	0.8	24.2	16.5
5	220	5	4.23	200	8	8	7	3	1324	39	5165	0.8	19.8	12.5
6	220	10	4.53	200	8	16	7	7	1349	34	7123	1.3	17.0	13.0
7	180	10	3.36	220	8	8	7	7	1291	25	4315	1.0	27.1	20.2
8	180	5	3.05	200	16	16	7	7	1276	36	5016	0.2	46.4	38.8
9	200	7.5	3.82	210	12	12	5	5	1357	40	4484	0.6	23.2	16.3
10	180	10	3.36	220	16	8	3	7	1253	33	4491	0.4	28.2	14.0
11	200	7.5	3.82	210	12	12	5	5	1333	45	5393	0.7	19.0	22.1
12	180	10	3.36	200	8	16	7	3	1330	20	2527	0.4	36.5	26.5
13	220	5	4.23	200	8	8	3	7	1326	55	4915	0.8	24.5	14.2
14	220	5	4.23	220	16	8	3	3	1344	38	4762	0.4	17.3	15.0
15	200	7.5	3.82	210	12	12	5	5	1346	41	5861	1.0	23.8	16.6
16	180	5	3.05	220	8	8	7	3	1272	30	2871	0.5	33.0	28.6
17	200	7.5	3.82	210	12	12	5	5	1350	49	5280	0.5	20.8	15.4
18	180	10	3.36	200	8	16	3	3	1334	48	5340	0.0	53.4	28.4
19	180	5	3.05	200	8	8	3	3	1237	25	2821	0.0	105.8	54.6
20	200	7.5	3.82	210	12	12	5	5	1348	40	4811	0.7	20.6	18.2
21	180	10	3.36	200	16	16	3	3	1336	34	4482	0.2	67.7	49.2

22	180	5	3.05	220	16	16	3	3	1336	36	4882	0.4	62.6	50.4
23	180	10	3.36	220	8	8	3	7	1346	26	2751	0.8	29.9	23.6
24	220	10	4.53	220	16	8	7	3	1362	27	5444	0.5	13.1	9.5
25	200	7.5	3.82	210	12	12	5	5	1330	45	4274	0.7	21.0	15.7
26	220	5	4.23	200	16	16	3	7	1343	42	3992	0.4	22.5	22.3
27	220	10	4.53	200	16	8	7	3	1369	43	6097	0.6	16.1	12.4
28	180	5	3.05	220	16	16	7	7	1275	52	4923	0.3	32.3	25.8
29	220	10	4.53	220	8	16	3	7	1414	35	6234	0.6	13.0	10.6
30	220	5	4.23	220	8	16	3	3	1363	42	6164	1.3	15.8	15.1
31	220	10	4.53	220	16	16	7	3	1349	28	5400	0.5	13.6	10.9
32	200	7.5	3.82	210	12	12	5	5	1353	35	4946	0.6	23.9	16.7
33	220	5	4.23	220	8	16	7	7	1370	36	5405	0.3	15.1	13.4
34	200	7.5	3.82	210	12	12	5	5	1337	36	4516	0.5	20.4	21.1
35	200	7.5	3.82	210	12	12	1	5	1338	45	4749	0.1	27.9	31.3
36	200	7.5	3.82	210	12	12	5	9	1359	43	5949	0.5	19.7	16.4
37	160	7.5	2.64	210	12	12	5	5	1292	28	4619	0.2	70.3	56.4
38	200	7.5	3.82	210	12	12	5	1	1371	45	5812	0.3	24.8	20.6
39	200	7.5	3.82	210	4	12	5	5	1281	38	3627	0.8	29.1	23.9
40	200	2.5	3.34	210	12	12	5	5	1297	21	2859	0.3	37.2	37.9
41	200	7.5	3.82	190	12	12	5	5	1376	40	5017	0.6	36.7	26.1
42	200	12.5	4.04	210	12	12	5	5	1389	50	6601	0.6	22.2	16.6
43	200	7.5	3.82	210	12	20	5	5	1326	44	5683	0.7	19.3	21.1
44	200	7.5	3.82	210	12	12	9	5	1311	43	6607	0.7	21.7	14.3
45	200	7.5	3.82	210	20	12	5	5	1392	49	5384	0.3	21.4	20.5
46	240	7.5	5.00	210	12	12	5	5	1371	15	4822	1.0	8.2	4.3
47	200	7.5	3.82	230	12	12	5	5	1277	30	5811	1.1	15.8	12.2
48	200	7.5	3.82	210	12	4	5	5	1355	43	5737	0.6	26.4	18.3

Table 3. Chemical compositions of *Cynara cardunculus* with different pretreatment conditions

	Process factors			Response variables			
Run	T_r [°C]	$t_{\_r}$ [min.]	Log (ρ)	Ash [%]	Lignin [%]	Cellulose [%]	Hemicellulose [%]
Original	—	—	—	5.4	17.5	49	24
4	200	7.5	3.8	0.5	18.7	59.3	15.1
5	220	5	4.2	0.4	13.6	65.5	9.4
9	200	7.5	3.8	0.5	18.7	60.1	15.9
10	180	10	3.4	1.0	19.9	53.9	23.2
11	200	7.5	3.8	0.5	17.8	60.8	14.4
16	180	5	3.1	1.3	17.8	53.8	29.3
17	200	7.5	3.8	0.5	17.9	63.0	13.2
20	200	7.5	3.8	0.6	18.2	64.6	17.8
24	220	10	4.5	0.6	14.9	68.1	0.2
25	200	7.5	3.8	0.5	17.8	62.4	14.3
32	200	7.5	3.8	0.6	17.9	62.7	15.0
34	200	7.5	3.8	0.7	18.3	58.5	16.9
37	160	7.5	2.6	1.9	16.5	50.5	35.2
40	200	2.5	3.3	0.9	17.4	58.0	21.8
42	200	12.5	4.0	0.5	16.7	62.8	13.6
46	240	7.5	5.0	2.8	16.3	53.2	0.2

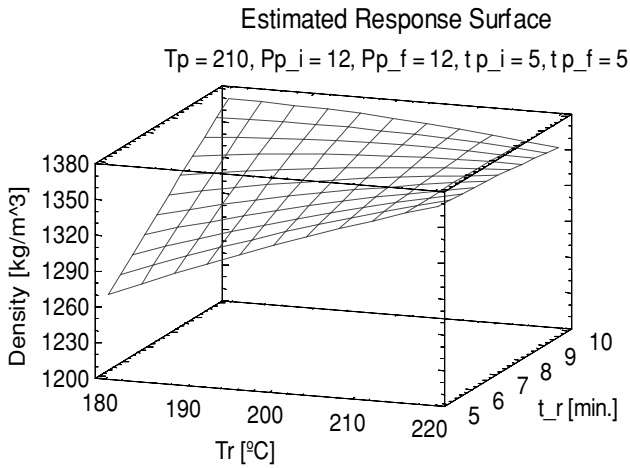

Figure 1. Estimated response surface for density, T_r vs. $t_{\_r}$.

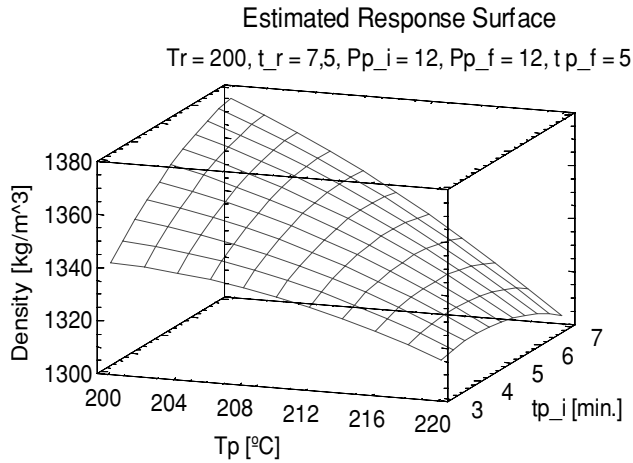

Figure 2. Estimated response surface for density, T_p vs. t_{p_i}.

shows that the pretreatment time has a bigger influence at low temperatures than at high temperatures.

From the response surfaces shown in Figures 2 and 3, it can be seen that low pressing temperatures or high initial press pressures and long pressing times favor an increase in density. To allow a good distribution of lignin between the fibers during the pressing process, it is necessary to apply enough heat and pressure to melt the lignin through the whole board.

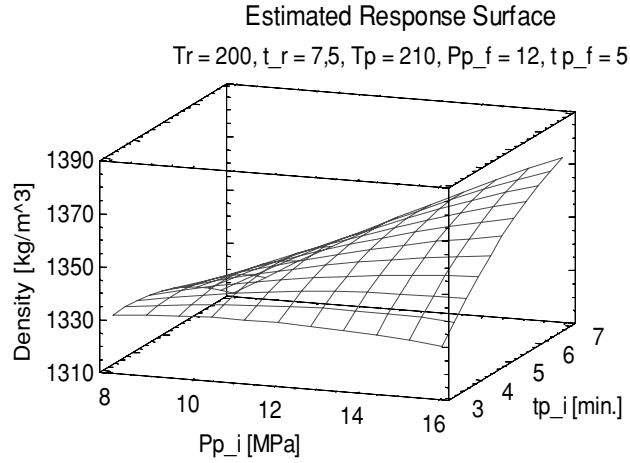

Figure 3. Estimated response surface for density, P_{p_i} vs. t_{p_i}.

Strength and Modulus of Elasticity

The MOR and MOE were analyzed together because they came from the same bending assay. The fitted model for MOR gave an R^2 of 0.941 and an SDR of 4.2 MPa. The fitted model for MOE gave an R^2 of 0.884 and an SDR of 696 MPa. Only one factor (pretreatment time) was statistically significant for MOR, whereas for MOE three factors were statistically significant (pretreatment time, initial pressing pressure, and initial pressing time). The modeled surface in Figure 4 shows that the best MOR values are obtained at low pretreatment temperatures and long pretreatment times. The same is true for MOE. These results also agree with density behavior. Vapor pretreatments at low temperatures preserve the fibrillar structure, but long times are needed to achieve the chemical and physical modifications that enhance the adhesive behavior of the lignin. This is confirmed by the behavior of pretreatment time, which has a bigger influence at low pretreatment temperatures than at high pretreatment temperatures (see Figure 4).

The modeled surface in Figure 5 shows that low pressing temperatures and long pressing times enhance MOR, which agrees with density behavior. However, in Figure 6 it can be seen that the trend for MOE is different: it increases when the pressing time rises at high pressing temperatures while the MOR decreases with the same combination of factors.

The optimal conditions found were: (i) $T_r = 160^\circ$C, $t_{\_r} = 12.5$ min, $T_p = 222^\circ$C, $P_{p\_i} = 17.7$ MPa, $P_{p\_f} = 12.2$ MPa, $t_{p\_i} = 1$ min, $t_{p\_f} = 1$ min for MOR; and (ii) $T_r = 160\ ^\circ$C, $t_{\_r} = 12.5$ min, $T_p = 230^\circ$C, $P_{p\_i} = 11.9$ MPa, $P_{p\_f} = 7.4$ MPa, $t_{p\_i} = 3.3$ min, $t_{p\_f} = 1$ min for MOE. The low pressing times

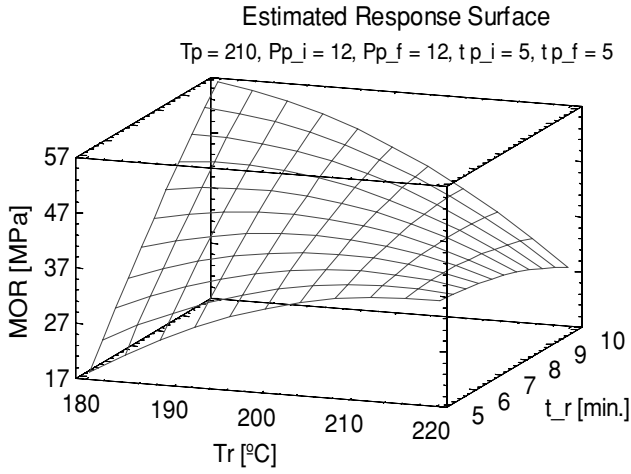

Figure 4. Estimated response surface for MOR, T_r vs. $t_{\_r}$.

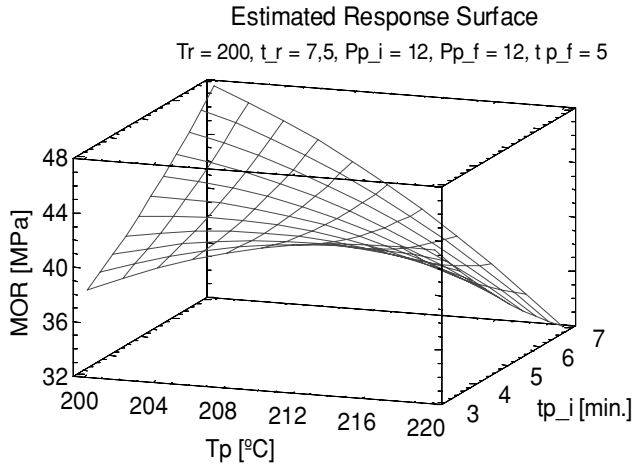

Figure 5. Estimated response surface for MOR, T_p vs. t_{p_i}.

predicted for the model as optimums for both factors were due to the high predicted values for pressing temperatures and pressures.

Internal Bond

The fitted model gave an R^2 of 0.945 and an SDR of 0.14 MPa. Four factors (pre-treatment temperature, pressing temperature, initial pressing time, and initial

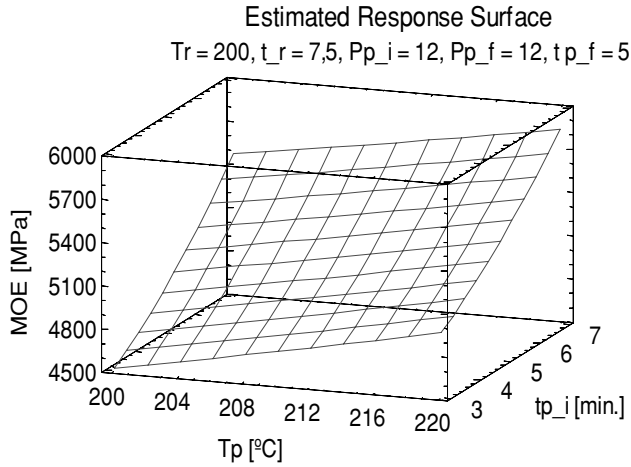

Figure 6. Estimated response surface for MOE, T_p vs. t_{p_i}.

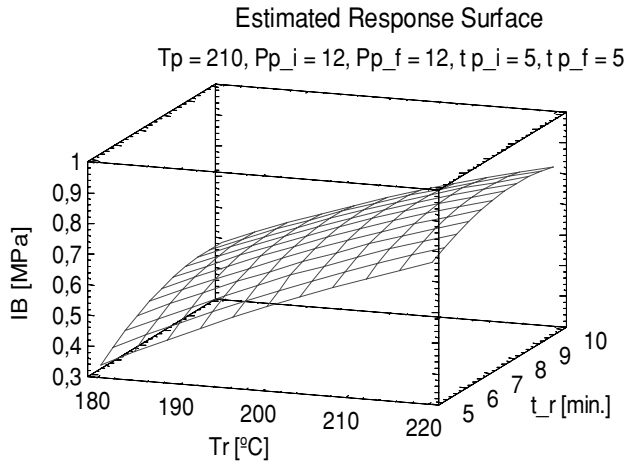

Figure 7. Estimated response surface for IB, T_r vs. $t_{\_r}$.

press pressure) were statistically significant. The modeled surface on Figure 7 shows that the best IB values were obtained at high pretreatment temperatures. This can be explained by the rising quantity of particles that appeared when the pretreatment temperature increased,[25] which increased the area available for bonding. Also, high pretreatment temperatures promote a higher extraction of hemicelluloses and extractives and partially depolymerize the lignin, which helps the bonding action.

Figure 8 shows that low pressing pressures and intermediate pressing times are preferred. As we have seen before, a suitable combination of process factors is the key to obtaining the desired properties. For the IB, due to the upward trend of the pretreatment and pressing temperatures, the pressing pressure should be low to avoid spoiling the fibers and to enable the proper distribution of lignin between them.

The optimum conditions for maximizing the IB are $T_r = 238°C$, $t_{\_r} = 7.4$ min, $T_p = 230°C$, $P_{p\_i} = 4$ MPa, $P_{p\_f} = 4$ MPa, $t_{p\_i} = 6$ min, $t_{p\_f} = 3.9$ min. High severity pretreatments favor the internal bond but this high severity based on temperature rather than time deteriorates the MOR and MOE. With regard to the pressing process, the optimum press temperature is the highest studied and the optimum press pressure is the lowest. Clearly, the optimum values for maximizing the IB are in a different direction to those for maximizing the MOE and MOR.

Water Absorption and Thickness Swelling

The fitted models gave R^2 values of 0.988 for WA and 0.984 for TS and SDRs of 3.8% and 2.9%, respectively. Four factors (pretreatment temperature,

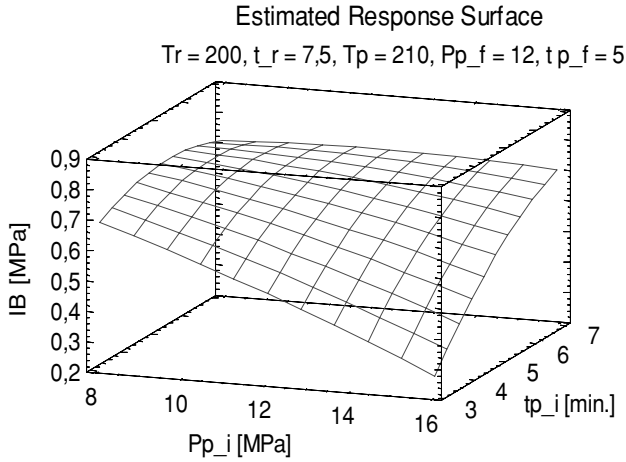

Figure 8. Estimated response surface for IB, P_{p_i} vs. t_{p_i}.

pretreatment time, pressing temperature, and initial pressing time) were significant for both response variables. The modeled surface (Figure 9) shows that the lower values of WA were obtained at high pretreatment temperatures and intermediate-to-long pretreatment times. The same was true for TS. This is because high-severity pretreatments enhance the hydrolysis of the hemicelluloses, which are largely responsible for board instability.[26]

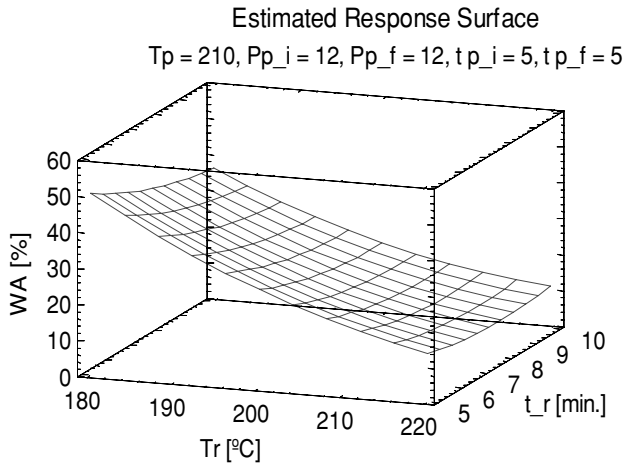

Figure 9. Estimated response surface for WA, T_r vs. $t_{\_r}$.

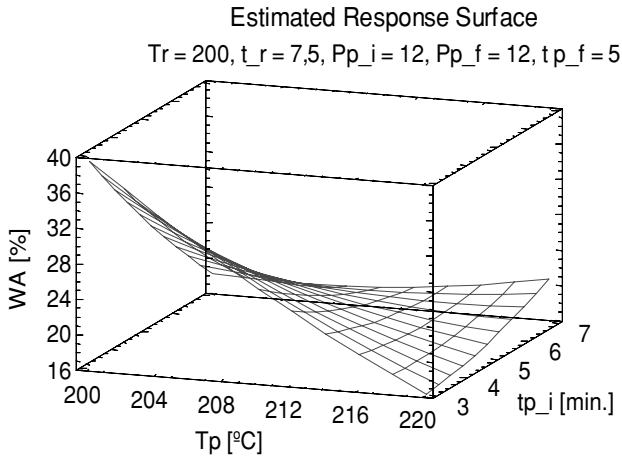

Figure 10. Estimated response surface for WA, T_p vs. t_{p_i}.

The general trend for the pressing process (Figure 10) is to get lower WA and TS at high pressing temperatures and short times, possibly to overcome the heat and mass transfer limitations in the pressing process.

The optimal conditions found were: (i) $T_r = 229°C$, $t_{\_r} = 7.4$ min, $T_p = 215°C$, $P_{p\_i} = 9.7$ MPa, $P_{p\_f} = 13.5$ MPa, $t_{p\_i} = 2.8$ min, $t_{p\_f} = 4.5$ min, for WA; and (ii) $T_r = 232°C$, $t_{\_r} = 11$ min, $T_p = 214°C$, $P_{p\_i} = 11.7$ MPa, $P_{p\_f} = 6.7$ MPa, $t_{p\_i} = 3.7$ min, $t_{p\_f} = 4$ min, for TS. The main differences between the optimums for the mechanical and physical properties were in the pretreatment temperatures and times: very high temperatures improved WA and TS whereas very low temperatures maximized MOR and MOE. These different tendencies suggest that there must be an agreement between the operational factors that result in the production of boards that fully satisfy the European standards.

Ash Content

The fitted model gave an R^2 value of 0.848 and an SDR of 0.31%. For this variable, only pretreatment temperature was statistically important. Ash accounts for mineral salts that are undesirable for the manufacture of fiberboards. Table 3 shows that the original material has a considerable amount of ash that could negatively influence the conformation of the boards. Ash content is greatly reduced by pretreatment; this reduction is due to solubilization of the mineral salts contained in the material, during the pretreatment. The minimum values for this response variable are found at intermediate reaction temperatures and long pretreatment times.

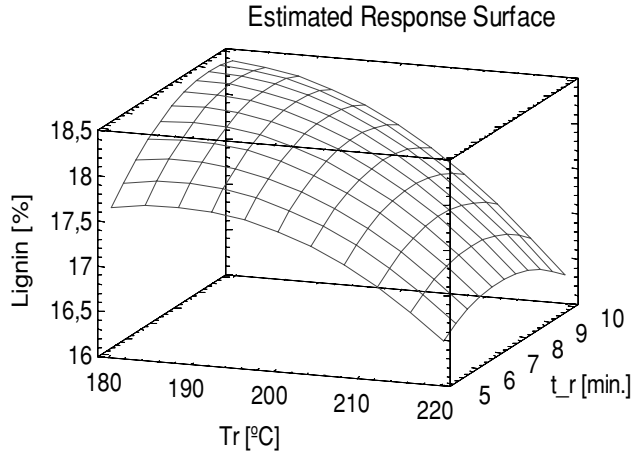

Figure 11. Estimated response surface for Lignin, T_r vs. $t_{\_r}$.

Lignin, Cellulose, and Hemicelluloses

The lignin, cellulose, and hemicelluloses were analyzed together because they came from the same hydrolysis assay. Fitted models gave an R^2 value of 0.651 and an SDR of 3.59% for cellulose, an R^2 value of 0.97 and an SDR of 1.94% for hemicelluloses and an R^2 value of 0.44 and an SDR of 1.40% for Lignin. Both pretreatment temperature and time were statistically significant for hemicellulose content and only pretreatment temperature was statistically significant for cellulose content, but neither of them was significant for lignin content. The response surfaces for the three variables (Figures 11, 12, and 13) show that the cellulose content increased but the hemicellulose content decreased as the severity increased. The quantity of lignin slightly diminished as the severity increased. Similar results for cellulose and hemicellulose behavior have been obtained for other materials[22,23,26] However, the lignin content appears not to be affected very much by pretreatment. This is understandable because lignin is much more stable than cellulose and hemicellulose.

Chemical Composition and Physico-mechanical Properties

It is well known that the dimensional stability of the fiberboards is related to partial hemicellulose hydrolysis because hemicelluloses are very hydrophilic. In Figure 14 we can see that, as expected, WA decreased as the hemicellulose content decreased. The same was true for TS. Some authors[22,23,25,26] have obtained similar results with other materials. The relationship between WA and

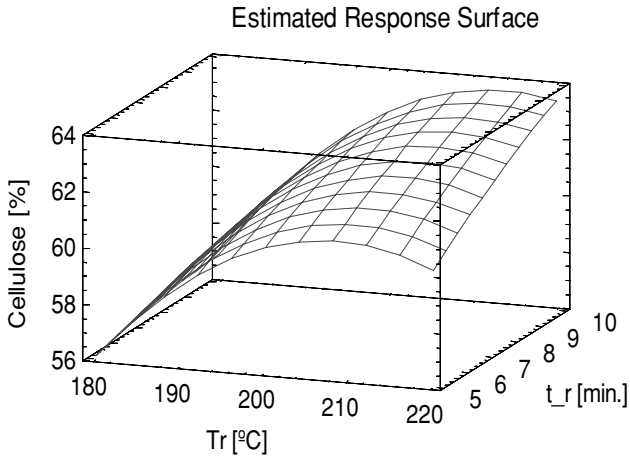

Figure 12. Estimated response surface for Cellulose, T_r vs. t_r.

TS with hemicelluloses is supported by the high R^2 values: 0.758 for WA and 0.652 for TS. The SDR were 7.14% for WA and 7.96% for TS.

Multiple Response Optimization

Multiple response optimization determines the combination of levels for the experimental factors that simultaneously optimize several response variables.

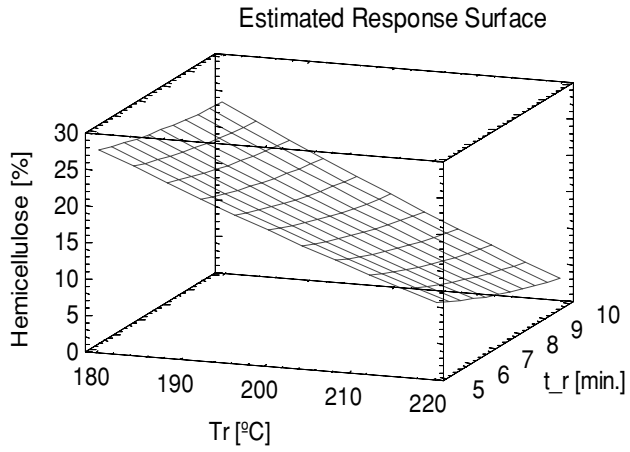

Figure 13. Estimated response surface for Hemicelluloses, T_r vs. t_r.

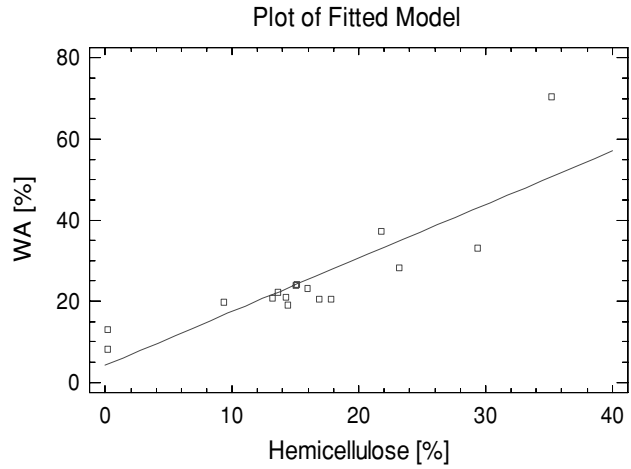

Figure 14. Relationship between WA and Hemicelluloses.

The procedure consists of building a desirability function based on the fitted models of each factor to be optimized. The optimum value of desirability was 0.995 over 1 for the following factor levels: $T_r = 218^{\circ}C$, $t_{\_r} = 5.4$ min, $T_p = 220^{\circ}C$, $P_{p\_i} = 7.2$ MPa, $P_{p\_f} = 19$ MPa, $t_{p\_i} = 2.5$ min, $t_{p\_f} = 2.8$ min. Figure 15 shows that mid-high pretreatment temperatures and short times are the best choice for simultaneously preserving the fiber structure and encouraging hydrolysis of hemicelluloses and lignin release.

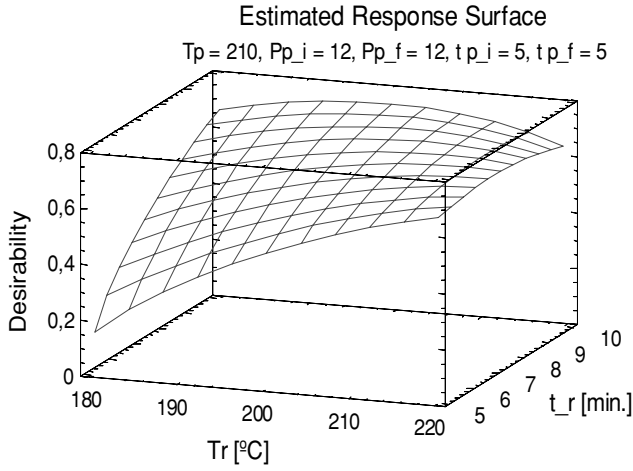

Figure 15. Estimated response surface for desirability, T_r vs. $t_{\_r}$.

A set of fiberboards was prepared using the combination of factors provided by the multiple response optimization. These fiberboards fully satisfy the European standards. The mean values for the board properties were: Density = 1320 kg/m^3, MOR = 52 MPa, MOE = 5970 MPa, IB = 0.8 MPa, WA = 18.5%, and TS = 13.5%.

The multiple response optimization model studied suggests that the best physico-mechanical properties for the boards are found at high pretreatment temperatures and low pretreatment times. The model also suggests using high pressing temperatures in combination with short pressing times and low pressing pressures in the first stage but high pressures in the third pressing stage. This suggests that in the third pressing stage, the internal defects generated in the relaxation stage are corrected and that, in the first stage, the humidity is vaporized and the lignin is redistributed over the fibers where the chemical bonds are developed.

CONCLUSIONS

It was possible to produce binderless fiberboards from *Cynara cardunculus* that meet the European standards for fiberboards of internal use, thus giving an aggregated value to this energetic crop and contributing to its full exploitation. *Cynara cardunculus* is not the best material to produce binderless fiberboards compared with other materials studied previously such as *Miscanthus sinensis*[22] and residual softwood.[3,5] This is probably due to its lower lignin content and its higher ash content, but still it can be used to obtain fiberboards of good quality without adhesives of fossil origin.

Both steam explosion pretreatment and hot pressing had great influence on the final physico-mechanical properties of the fiberboards obtained. First, pressing stage has shown to be statistically significant for almost all the mechanical properties analyzed (Density, MOE, and IB), but the last stage of the pressing was not statistically significant, in the range studied. It can be concluded that the values of the analyzed parameters in the last stage of pressing can be adjusted to lower pressing times and lower pressing pressures than the studied according to the convenience of an industrial application.

Increasing the severity of the pretreatment improves the physical properties (WA and TS) of the boards. Similarly, hemicellulose and ash contents of the pretreated fibers clearly decrease as the severity increases, which lead to lower hygroscopicity and minimize abrasive materials that are undesirable for the fabrication of fiberboards. Pretreated *Cynara* generally has higher cellulose and lignin contents than the original material due to decreased hemicellulose content.

The multiple response optimization model has been useful for finding the best levels of the process factors for producing the best fiberboards, particularly

for the difference found between the optimization trends for physical and mechanical properties.

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