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Valorization of Insulation Cellulose Waste as Solid Biomass Fuel

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Abstract: This paper investigates the ability of insulation cellulose fiber powder (CFP) to be pelletized for its valorization as biomass fuel. CFP is a waste originating from insulation cellulose manufacturing that lacks any method of valorization because of its boron salts content. A sugar byproduct and lignosulfonate (LS) were considered as binders for the pellet manufacturing process. Physical tests were carried out to characterize the pellets' performance. Chemical and combustion tests were considered to state the pellets' potential as a green energy source. Raw CFP showed good ability in its pelletization and durability in the range of 15–30% of moisture content. The pellet's density decreased as water content increased. Binders increased the pellet's length before and after the durability test. Binders also increased the CFP pellet's water absorption, demonstrating a potential decrease in durability against environmental factors. Binders also decreased the lower heating value. Ultimate analysis showed a slight Nitrogen increase in both binder combinations that could potentially raise the pollutant NO_x combustion emissions. All the combinations showed adequate combustion characteristics, but binders increased ash production. Additives decreased the CFP volatile matter content and increased the fixed carbon, which could facilitate a more stable combustion. DTA curves showed a mass loss rate decrease in the volatile stage for the binder combinations, which also could be considered as an indicator of a more stable combustion. The ashes' chemical compositions when analyzed by XPS showed boron contents oscillating between 10.03% and 16.42%, demonstrating the possibility of recovering them from the combustion ashes.

Keywords: cellulose fiber powder; biomass fuel; pelletization; sugar; lignosulfonate

1. Introduction

Cellulose fiber (CF) is a popular material for the insulation of the external envelope of buildings. This is due to its favorable thermal and acoustic properties, low manufacturing cost and easy installation by blowing it in closed wall cavities or attics. CF is a natural, renewable and recyclable material with low embodied energy and carbon dioxide footprint. Moreover, it is a non-toxic resource and, thanks to its high hygrothermal conductivity, it can help control the building's indoor air humidity [1–6]. CF is usually manufactured from recycled newspaper which is clawn in a fiberizer machine to separate the fibers. Because of the combustible and organic nature of this material, the addition of substances, usually boron salts known as borax, is required to increase its fire resistance and to avoid cellulose vermin and rotting. During manufacturing, once CF and borax are mixed, a separator removes the shorter fibers which are not adequate for applying as an insulator. As a result, a fine cellulose fiber powder (CFP) is generated, remaining as a useless waste because of its boron salts content. Based on actual circular economy thinking, CFP would not be considered a waste but a valuable resource to take advantage of, saving resources and avoiding its disposal in landfills.

The manufacture of non-conventional fuel alternatives to fossil fuels is an application with sizeable economic and environmental benefits that would contribute to the valorization of this waste [7,8]. Many published studies have demonstrated the ability of the manufacturing of non-conventional fuels for the valorization of different kinds of waste. Among these, different kinds of biomass stand out, like agricultural crop residues [9–12], agroindustrial residues [13–15], forest biomass [16] and wood processing industry residues [17,18]. Other wastes benefit from this advantageous method of valorization, such as sewage sludge [19–21] or waste paper [22]. CFP could become a green energy source based on its close composition to wastepaper. Table 1 shows the heating value ranges of different wastes used for the manufacturing of solid fuels, as well as the EN 14961-2 and EN ISO 17225-6 Standard heating requirements for woody and non-woody solid biofuels, respectively.

Table 1. Heating values of different wastes' constituents of non-conventional fuels and EN 14961-2 and EN ISO 17225-6 Standard heating requirements.

Waste Kind	Higher Heating Value (MJ/kg)	Source	Lower Heating Value (MJ/kg)	Source
Agricultural crop residues	14.64–18.38	[19]		
Forest biomass	13.06–20.82	[23,24]	16.06–18.01	[11,16]
Wood processing industry residues	15.86	[22]		
Other wastes	15.59–19.67	[22,25]		
EN 14961-2			16.50–19.00	
EN ISO 17225-6			>14.50	

Cellulose, the main constituent of CFP, has a lower heating value (LHV) of 14.570 MJ/kg, which suggests this waste's potential for valorization as a solid, non-woody biomass fuel for green energy production [26]. Before use, CFP requires a densification process because of its low density and powdery nature. Once densified, CFP could become a commodity able to be managed by the nowadays common solid biomass fuel handling, transportation, storage, feeding and combustion systems. Pelletization is the most usual biomass densification technology, Ref [27–30]. During this process, solid particles, with or without binders, are bonded to each other by attracting forces, diffusion of molecules or ingredient crystallization. In this way, a hardened commodity with higher density, improved durability and improved combustion properties is produced [11,19,22,23,31–36]. Although there are many works that demonstrate the suitability of different lignocellulosic materials for solid biomass fuels manufacturing [12,37–41], there is a lack of knowledge about the potential of CFP with boron salts as an energy source. Therefore, the aim of the present study is to investigate the suitability of CFP for the manufacturing of a solid biomass fuel by means of the characterization of the key points of solid fuels: (1) physical properties and durability, (2) heating value and combustion characteristics and (3) ashes' production and potential emissions. The following parameters were analyzed: the ability of the CFP to be pelletized, the effect of binders on the pellet's physical characteristics and durability, the energy recovered from its combustion, its combustion properties and the ash production. The ash was chemically characterized to determine its boron content. This way, the energetic valorization of the CFP would allow not only for the production of green energy but also for the recovery of boron.

2. Materials and Methods

For the laboratory investigation, a sample of 250 kg of CFP was collected in the AISLANAT cellulose fiber manufacturing factory in Pamplona (Spain). The sample was homogenized in the laboratory before its physical characterization. The CFP sample's bulk density was 0.091 g/cm^3 and its moisture content reached 1%. For the pellet manufacturing, two binders were considered: lignosulphonate and sugar dust. Lignosulphonate is a commercial additive extracted from carob pop which contains calcium sulphate. It is

usually added in the pelletization process of materials with low or no content of natural lignin, for example, in animal feed production. Sugar dust is a byproduct obtained in the sugar manufacturing process by accumulation of saccharose particles on factory surfaces.

Different combinations of moisture and additive contents were considered for the pellets' production. Sample manufacturing was carried out as follows: for each combination, 2 kg of CFP were prepared. When the combination contained any additives, constituents were carefully dry mixed in a laboratory mixer for 20 min. After that, the water required to reach the considered moisture content was slowly sprayed and mixed for 5 additional minutes. To guarantee the homogeneity of the moisture in the samples, they were maintained in closed containers for 24 h after mixing. Pelleting was carried out in a 4 kW electric pellet mill (PLT100, ECOFRICALIA, Las Pedroñeras, Spain) equipped with two rotating rollers and a steel die ring of 22 mm thickness with 4 mm diameter holes. Prior to manufacturing, the pellets the machine was kept working for 10 min to guarantee the die ring had reached the working temperature. Each mixture was carefully introduced in the machine by its upper hopper. When the material gets inside, pelletizer rotating rollers push it down and compress it in the die ring through the orifices. The resulting pellets come out below the die ring; breaking by themselves, they are collected in an inside chamber and removed from a lower orifice. The first 500 g of pellets of each combination were rejected to avoid any possible contamination by the remains of previous combinations. After manufacturing, pellets were kept in laboratory conditions for 12 h for cooling and drying before testing. Table 2 shows the combinations of CFP, additives and moisture contents considered for the laboratory investigation. Figure 1 shows CFP pellets manufactured without binders with 10% and 40% moisture content.

Table 2. Laboratory investigation combinations.

Number	Code	Additive	Additive Percentage (%)	Water Added (%)
1	C-0-5W	-	-	5
2	C-0-10W	-	-	10
3	C-0-15W	-	-	15
4	C-0-20W	-	-	20
5	C-0-30W	-	-	30
6	C-0-40W	-	-	40
7	C-1S-5W	Sugar	1	5
8	C-1S-10W	Sugar	1	10
9	C-1S-15W	Sugar	1	15
10	C-1S-20W	Sugar	1	20
11	C-1S-30W	Sugar	1	30
12	C-1S-40W	Sugar	1	40
13	C-2S-5W	Sugar	2	5
14	C-2S-10W	Sugar	2	10
15	C-2S-15W	Sugar	2	15
16	C-2S-20W	Sugar	2	20
17	C-2S-30W	Sugar	2	30
18	C-2S-40W	Sugar	2	40
19	C-1L-5W	Lignosulphonate	1	5
20	C-1L-10W	Lignosulphonate	1	10

Table 2. *Cont.*

| Number	Code	Additive	Additive Percentage (%)	Water Added (%)
21	C-1L-15W	Lignosulphonate	1	15
22	C-1L-20W	Lignosulphonate	1	20
23	C-1L-30W	Lignosulphonate	1	30
24	C-1L-40W	Lignosulphonate	1	40
25	C-2L-5W	Lignosulphonate	2	5
26	C-2L-10W	Lignosulphonate	2	10
27	C-2L-15W	Lignosulphonate	2	15
28	C-2L-20W	Lignosulphonate	2	20
29	C-2L-30W	Lignosulphonate	2	30
30	C-2L-40W	Lignosulphonate	2	40

(**a**)   (**b**)
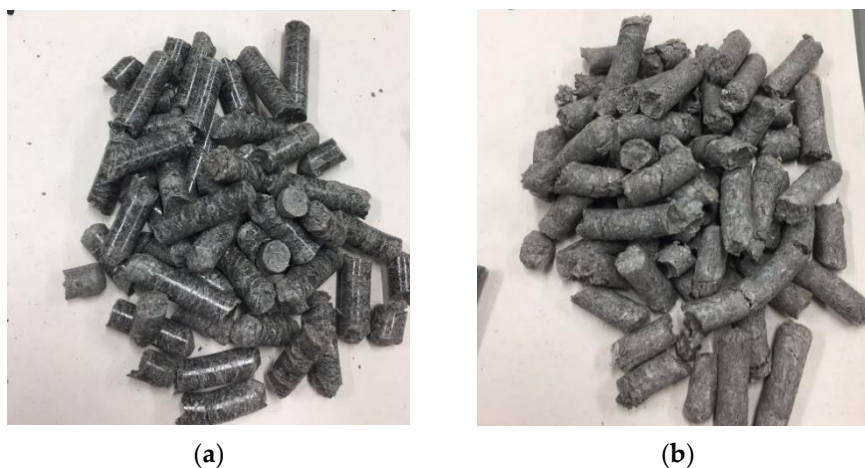
Figure 1. Pellets manufactured, (**a**) moisture content 10% and (**b**) moisture content 40%.

The convenience of pellets as solid biomass fuel was defined by two main characteristics: the first was the ability of the CFP to produce a densified material suitable as a commodity product. The second consisted of the pellets' chemical analysis, combustion properties evaluation and ash characterization. Manufacturing performance was defined as the material ability to produce pellets with a low content of small particles. It was defined as the percentage of mass of the processed sample that was retained by the 4 mm sieve. A pellet's bulk density was considered representative of the energy per volume unit, in accordance with the European Standard EN ISO 17828. Mechanical durability was characterized by means of an abrasion and knocking test adapted from the European Standard EN ISO 17831-1. This test consisted of maintaining 500 g of sample under rotation in a 600 mm diameter drum, 30 degrees tilted for 20 min. After that, mechanical durability was defined by means of two parameters: the first one was the pellets' loss of mass by sieving the sample with the 4 mm sieve. The second mechanical durability parameter was the reduction of a pellet's length. To analyze this parameter, 100 pellets of each combination were randomly chosen, measuring their length before and after testing with an 0.01 mm electronic caliper. Water absorption was considered an estimator of the durability against the environmental conditions. This test was carried out in a wet chamber at 20 °C and 100% of relative humidity over 4 h. A sample of 500 g of each combination was weighted at different testing times in a 0.01 g laboratory balance to measure the weight increases, expressed as a percentage of the initial sample mass. From a chemical point of view, ultimate and proximate analyses were considered. The element compositions of the raw materials were analyzed by means of a Thermo Finnigan FlashEA 1112 analyzer. Tests were conducted

in a helium atmosphere at 900 °C with oxygen injection and chromatographic columns gases separation Proximate analyses were carried out in a METTLER-TOLEDO TG-DSC2 system. Tests were conducted with 10 mg of sample under an air flux of 100 mL/min and a heating rate of 10 °C/min. A N_2 atmosphere was used, raising the room temperature to 600 °C and a N_2:O_2 (4:1) atmosphere from 600 °C to 900 °C. Combustion tests were carried out using the same equipment and the same conditions, but with the room temperature at 900 °C with a N_2:O_2 (4:1) oxidizing atmosphere. Lower heating values (LHV) were determined using an IKA C5003 calorimeter. Finally, samples of the different combinations considered were calcined in a laboratory oven at 550 °C, according to EN ISO 18122, and ashes were analyzed by X-ray photoelectron spectroscopy (XPS) in a K-alpha Thermo Scientific instrument to identify and quantify their chemical composition.

3. Results and Discussion

3.1. Manufacturing Performance

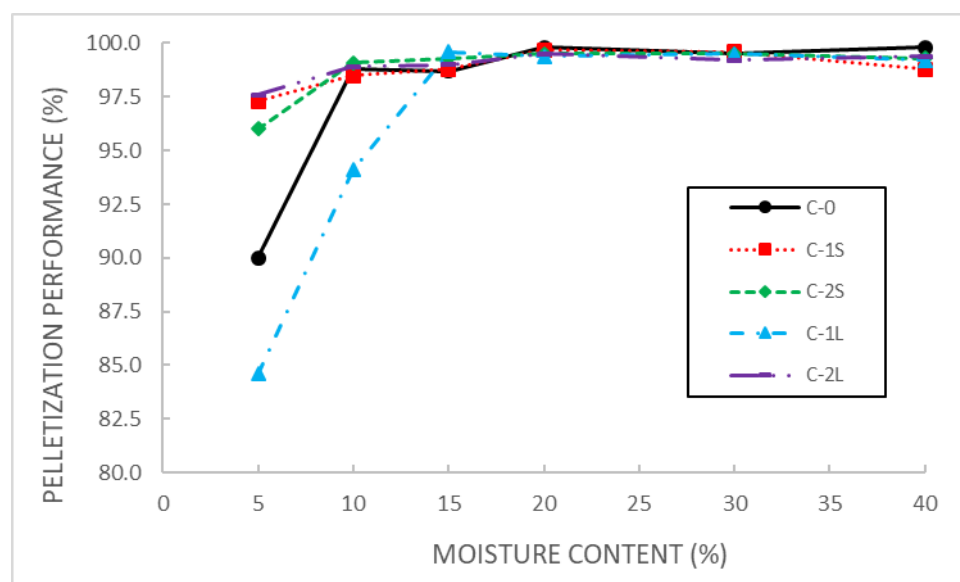Figure 2 shows the pellet manufacturing performance of the tested combinations.

Figure 2. Pelletization performance.

All the combinations reached their lower manufacturing performance results at 5% of moisture content. Thus, C-0-5W, C-1S-5W, C-2S-5W, C-1L-5W and C-2L-5W combinations obtained a performance of 90.0%, 97.3%, 96.0%, 84.6% and 97.6%, respectively. For this water content, sugar improved the raw CFP results for both dosages. On the other hand, LS showed an opposite effect for the same water content, depending on its dosage. At 1%, LS decreased the value achieved by CFP to 84.6%; meanwhile, at 2% of the dosage, performance increased to 97.6%. For all the mixes, the manufacturing performance increased as the water content did, reaching their maximum values in the range of 15–20% of moisture content. Above these water contents, no differences based on the additive kind or dosage were observed. Thus, C-0-20W, C-1S-20W, C-2S-20W, C-1L-15W and C-2L-20W combinations obtained performances of 99.8%, 99.7%, 99.5%, 99.6% and 99.5%, respectively. For higher water contents, performances remained steady or decreased slightly. This demonstrated that CFP pelleting performance could be increased at the lowest moisture content by means of both sugar dosages as well as 2% of lignosulphonate. For higher moisture contents, the use of binders does not significantly increase the CFP pelletizing performance.

3.2. Pellet Bulk Density

Figure 3 shows the pellets' bulk dry densities reached by the mixes tested related to their water content.
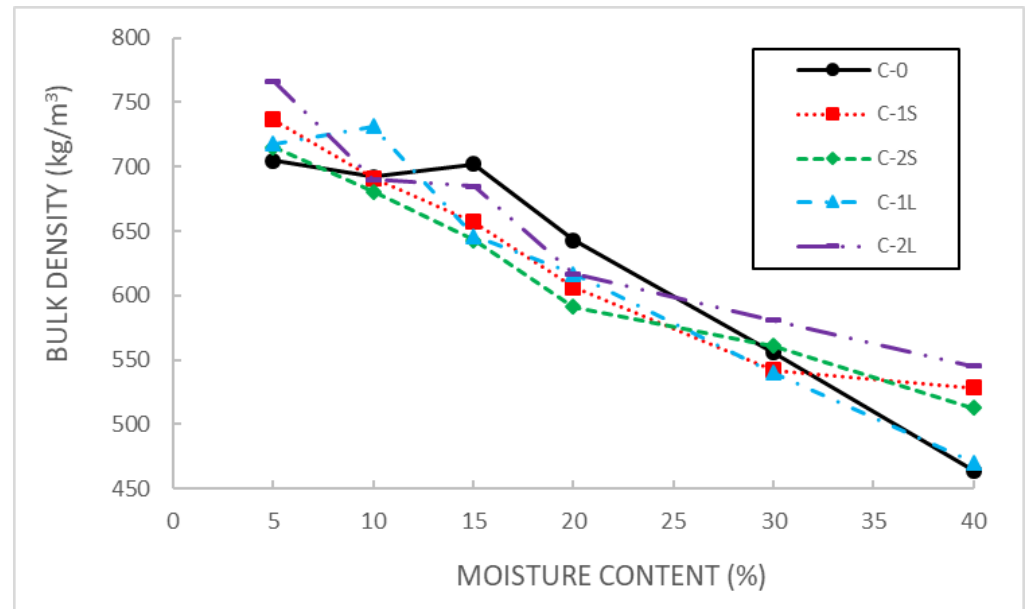

Figure 3. Bulk dry densities.

Moisture content showed an indirect relationship with the pellets' bulk dry densities due to the lack of compressibility of water absorbed by cellulose fibers, which does not allow the CFP proper compression during the pelletizer process or evaporation after manufacturing. The samples' densities oscillated between 766.20 kg/m^3 and 464.20 kg/m^3 for the C-2L-5W and C-0-40W combinations, respectively. No relationship was observed between a pellet's bulk density and the use or absence of additives, their dosage or the additive kind. The ISO 17225-6 Standard states a minimum bulk density for the non-woody pellets of 600 kg/m^3 that is only achieved by samples being manufactured with a moisture content lower than 20%.

3.3. Mechanical Durability

As stated previously, pellets' mechanical durabilities were characterized by the loss of mass by sieving after the abrasion and knocking test and the decrease in their length. Figure 4 shows the pellets' loss of mass after testing as a percentage of the initial samples' masses.

Mixes with 5% of moisture content showed the highest losses of mass in this test. Thus, C-0-5W, C-1S-5W, C-2S-5W, C-1L-5W and C-2L-5W combinations lost 22.32%, 6.31%, 6.30%, 17.66% and 4.40% of the initial mass, respectively. For higher moisture contents the loss of mass decreased, reaching the mixes' lowest losses in the range of 10–20% of moisture content, showing the existence of an optimum moisture content for particle bonding. Thus, C-0-20W, C-1S-20W, C-2S-15W, C-1L-15W and C-2L-10W combinations lost 2.95%, 1.67%, 1.84%, 0.94% and 1.10% of the initial mass, respectively. For values of moisture content above the optimum for each mix, all the combinations showed a slight increase in the loss of mass, probably due to an excess of water that hampered the particles' contact and bonding mechanisms. Combinations between 10% and 30% of moisture content showed a loss of mass under the limits stated in the Standard ISO 17225-6.
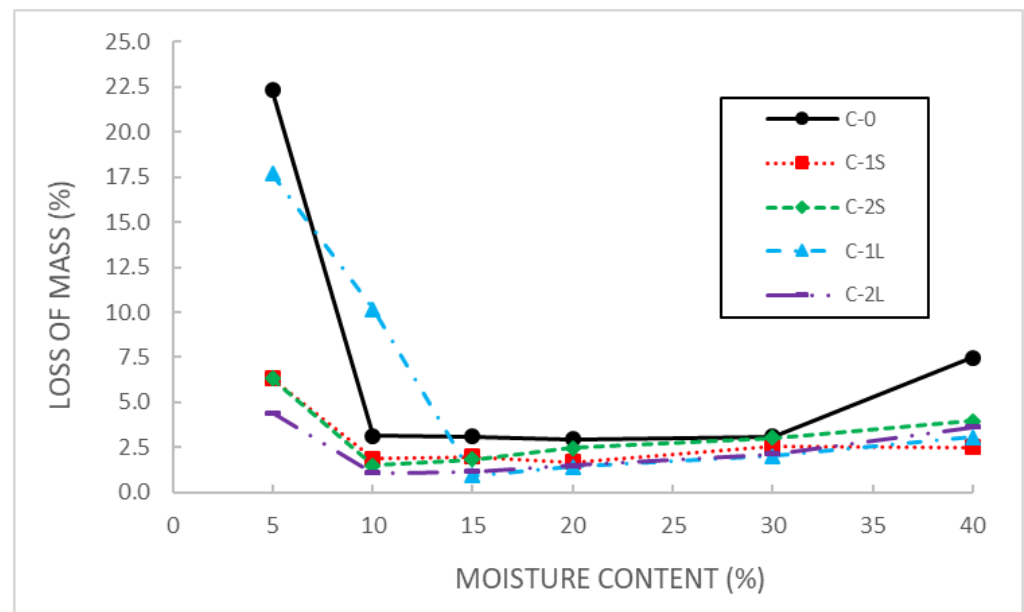
**Figure 4.** Loss of mass of the mechanical durability test.

Figure 5 shows the pellets' lengths before and after the abrasion and knocking test.

CFP produced the longest pellets for all the moisture contents. Thus, for the combination of C-0-5W the pellets' mean lengths reached 18.5 mm. For higher water contents, pellet lengths increased up to mean values close to 30 mm for the 10–40% moisture content range. After the abrasion and knocking test, CFP pellet lengths decreased to about 54–69% of the initial value of each combination. For the sugar-containing combinations, pellets reached mean length values between 17.8 for the combination C-1S-5W and 23.1 mm for the C-1S-15W. After the abrasion and knocking test, pellet lengths decreased, oscillating the final lengths between 70% and 96% of the initial values for the C-2S-10W and C-1S-40W combinations, respectively.

No relationship was observed between the pellet lengths and the sugar dosage or the moisture content. It is remarkable that after the abrasion and knocking test, only sugar pellet lengths clearly overcame the CFP results for 5% of moisture content mixes. On the other hand, 1% of lignosulphonate combinations required a minimum moisture content of 15% to reach these mixes' potential lengths of about 20 mm. The pellet length obtained its higher value of 20.7 mm for a 20% moisture content and slightly decreased for higher moisture contents, showing the existence of a possible adverse effect of the excess water, in accordance with the loss of mass results. The 2% lignosulphonate mixes showed a much better result than the 1% dosage for the 5 and 10% moisture contents. The best pellet length was reached by combination C-2L-15W, at 22.0 mm. For higher moisture contents results slightly decreased, demonstrating the existence of an optimum moisture content of 15% for the combinations containing a dosage of 2% of this binder. These combinations' pellet lengths after the abrasion and knocking test remained in the range of 88–98% of the initial values. It is remarkable that all the combinations reached the pellet lengths stated by the ISO 17225-6 Standard before and after the abrasion and knocking test. The results obtained after this test highlight the complexity of the relationship between the binder kinds, their dosages or the moisture content and the pellets' durability.

3.4. Water Absorption

Figure 6 shows the water absorption results when the pellet combinations were maintained in a wet chamber at 100% of relative humidity for 4 h.
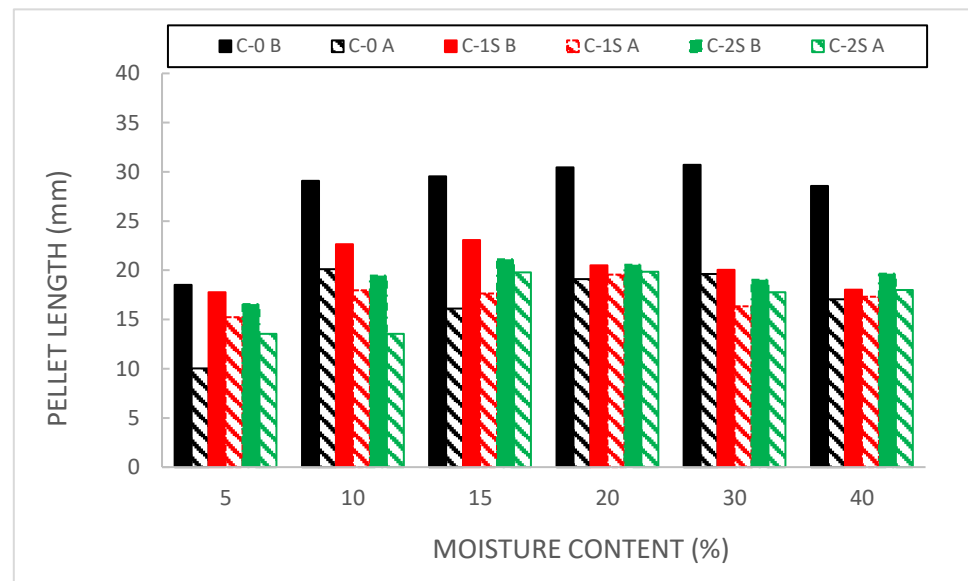
(**a**)
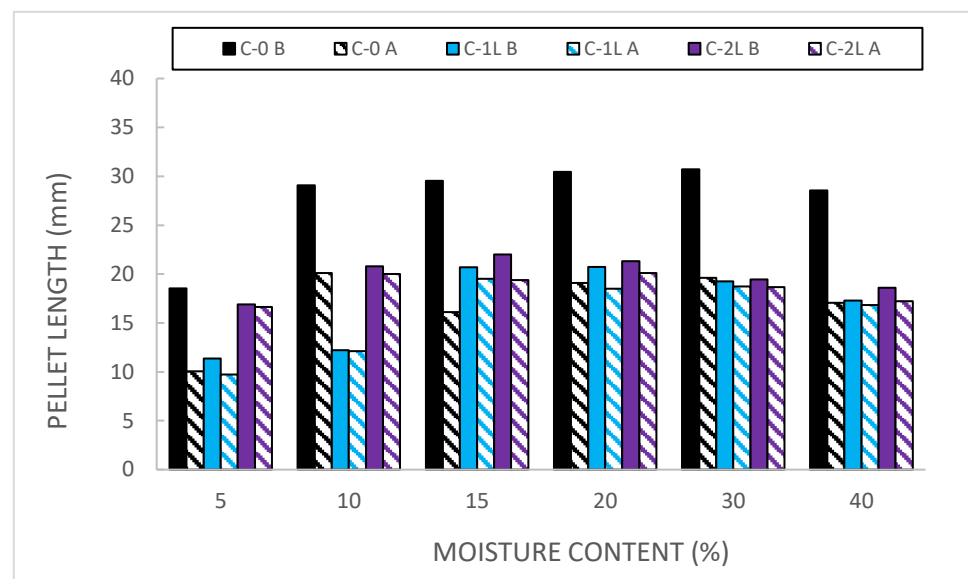
(**b**)

Figure 5. Pellet length before (B) and after (A) the abrasion and knocking test. (**a**) Sugar and (**b**) lignosulphonate containing mixes.

The lowest water absorption values were obtained by the mixes with 5% of manufacturing moisture content. This is probably due to the highest density of these combinations, which results in a denser structure and a closed pellet surface. Thus, C-0-5W, C-1S-5W, C-2S-5W, C-1L-5W and C-2L-5W combinations absorbed 1.51%, 1.46%, 1.34%, 1.84% and 1.46% of the initial mass, respectively. For all the mixes, water absorption capacity increased as manufacturing moisture content did, up to 30%, when the maximum of water absorption was reached by all the combinations. Water absorption decreased again for the 40% mixes. LS combinations showed a higher water absorption capacity than the other mixes for the same manufacturing moisture contents. This could be highlighting a higher hygroscopicity of this additive than the sugar and hence a lower durability against environmental conditions. The positive results obtained by the raw CFP samples are remarkable, probably due to the insoluble nature of their constituents that are mobilized to the pellet's surface in the pelleting process.
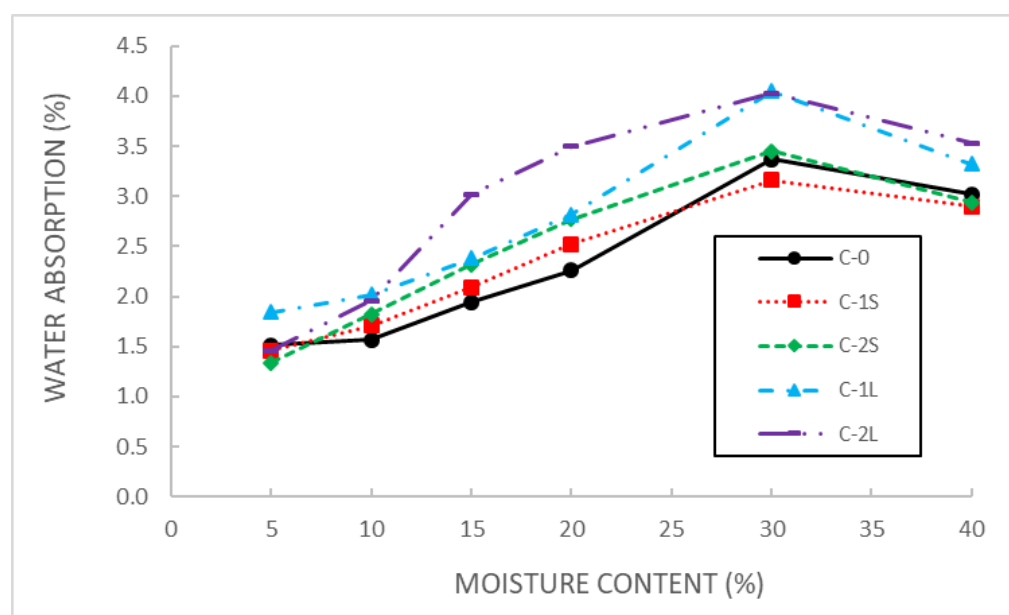
Figure 6. Water absorption.

3.5. Chemical Properties and Combustion Characterization

Table 3 shows the LHV of the different combinations' proximate and ultimate analyses, as well as their ashes composition.

**Table 3.** Combustion characterization of the CF combinations and the ashes' chemical compositions.

Analysis	CFP	CFP + 1% S	CFP + 2% S	CFP + 1% LS	CFP + 2% LS
Lower heating value (MJ/kg)	14.984	14.185	12.718	14.294	13.741
Ultimate analysis (wt%)					
N	0	0.11	0.17	0.13	0.88
C	39.15	38.67	34.68	38.39	33.39
H	5.48	5.33	5.14	5.45	5.05
S	0	0	0	0	0
O	55.38	55.89	60.01	56.04	60.69
Proximate analysis (wt%)					
Moisture	7.83	8.65	6.05	6.5	6.64
Volatiles	54.49	51.17	46.59	51.59	45.81
Fixed carbon	26.97	28.42	27.61	27.6	28.71
Ash	10.71	11.76	19.75	14.31	18.84
Ashes chemical composition (wt%)					
C	16.78	16.25	30.25	13.45	23.65
O	57.93	56.73	49.05	59.19	52.21
Si	5.04	7.15	5.02	7.93	4.29
B	16.42	13.09	10.03	14.4	15.67
Ca	3.83	6.78	5.65	5.03	4.18

The CFP showed an LHV value adequate for its use as biomass fuel [12]. As expected, the lower heating power of the pellets decreased when additives were used [18]. This partial substitution of CFP by less energetic products reduced LHV by 5.3% and 15.1% for the sugar at 1% and 2% of dosage, respectively. Lignosulfonate at the same dosages reduced CFP LHV by 4.6% and 9.3%, respectively, demonstrating its higher energetic power than sugar. Ultimate analysis showed a slight increase in the nitrogen that could potentially increase the pollutant NO_x's combustion emissions of combinations containing binders, particularly for the combination CFP + 2% LS. Despite the CFP + 2% LS combination

presenting 0.88% of N, all the combinations meet the parameters of the 17225-6 Standard. Unexpectedly, lignosulfonate combinations did not show the presence of sulfur, probably because of its low content in lignosulfonate and the low dosages considered.

All the pellet combinations showed similar combustion characteristics, highlighting their potential as a green energy source compared to other non-conventional biomass fuels [18,42–46]. All the combinations showed adequate combustion characteristics, except those with high ash content due to the presence of Boron salts in their composition. These oscillated in the range of 10.71–18.84, much higher than the 10% required by the ISO 17225-6 Standard for non-woody type B pellets. The use of additives decreased the CFP volatile matter content and increased the fixed carbon that could facilitate a more stable combustion. Figure 7 shows the thermogravimetric analysis of the raw CFP and additives containing pellets.

Thermogravimetric curves (TG) show the typical four stages throughout the combustion processes: dewatering, volatilization and burning, char burning and burnout. As the DTG curve depicts, the combustion of CFP generates two exothermic peaks due to the burning of volatile and fixed carbon, the fixed carbon peak being higher than the one due to volatile burning. DTG curves corresponding to the combinations with additives show a decrease in the volatile peaks that suggests a higher proportion of fixed carbon in these combinations, in accordance with the proximate analysis test results. This reduction in the volatiles could be related to a more stable combustion that could provide other beneficial consequences by using sugar or lignosulfonate for pellet manufacturing. DTA curves showed a decrease in the mass loss rate in the volatile stage for the binder-containing combination compared to the raw CFP one, which also could be considered an indicator of a more stable combustion. Finally, the ash's chemical composition was analyzed by XPS. The sample's spectrum showed two contributions at 192.5 eV and 193.7 eV that demonstrated that boron remains are an important constituent in ashes generated by all the combinations, as shown in Table 3. Considering the calcining temperature, the main boron constituent would be B_2O_3. This demonstrates the possibility of recycling CFP ashes as a boron source.
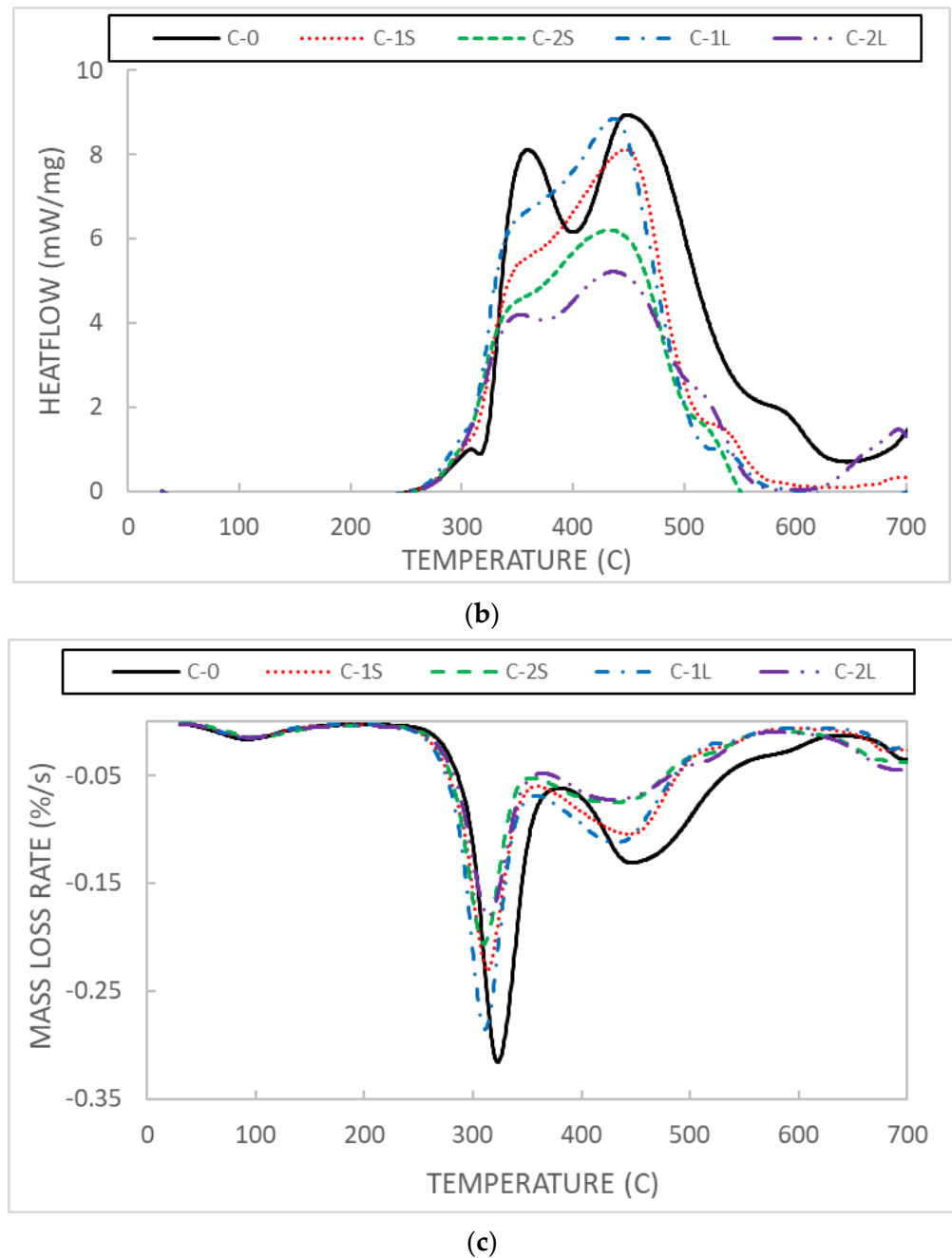

(a)

Figure 7. *Cont.*

(b)

(c)
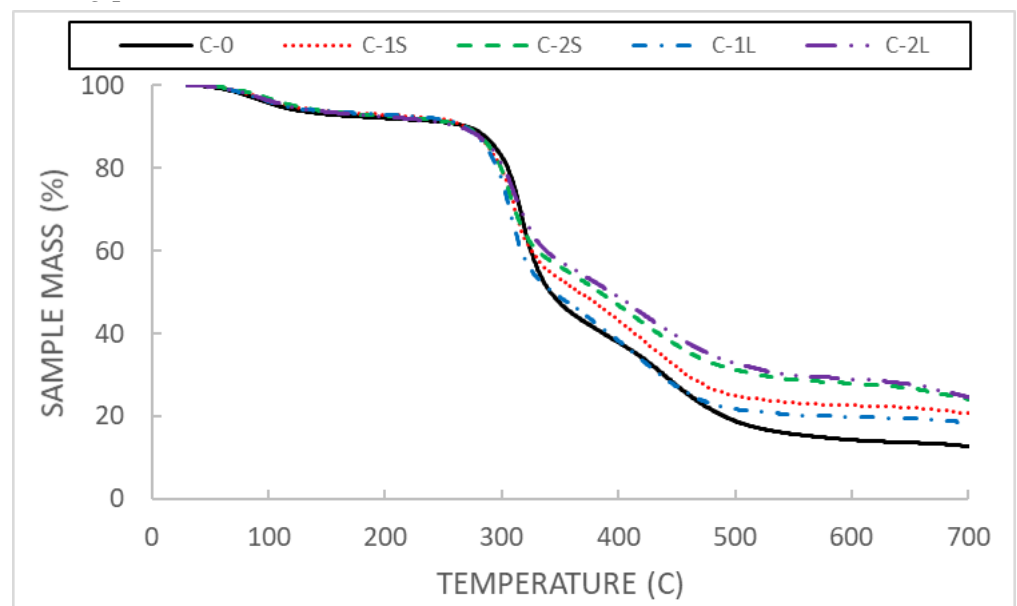
Figure 7. Thermogravimetric analysis of the samples. (**a**) TG, (**b**) DTG and (**c**) DTA.

4. Conclusions

The experimental investigation carried out has demonstrated the suitability of CFP for the manufacturing of pellets of biomass solid fuel based on the following conclusions:

1. CFP produced pellets with a manufacturing performance higher than 97.5%. The use of sugar or lignosulfonate did not increase this performance except for CFP moisture content below 10%, demonstrating that for higher moisture contents the effect of the binders over the manufacturing performance was negligible.

2. Pellet density and moisture content showed an indirect relationship with no evident differences among the combinations, based on the use of additives or the dosages considered.

3. Mechanical durability test results were contradictory. Moisture contents between 10% and 30% were optimum to obtain pellets with the lowest loss of fine particles. In this

range of moisture contents, no differences were observed between combinations with or without any one of the binders and the dosages tested. On the other hand, the use of binders demonstrated effectiveness in obtaining more resistant pellets against handling cracking.

4. The use of binders reduced the resistance of pellets against environmental conditions. This effect was clearer when using lignosulfonate, whose combinations demonstrated a higher hygroscopicity than those of sugar or CFP.

5. The use of binders reduced the pellets' energy power and could increase their pollutant potential and increase the ash content. On the other hand, binders would improve the combustion properties of this biomass fuel.

6. Boron remained in the ashes after combustion. This would allow an effective method of valorization for the CFP as well as the recovery of this product from among the ashes generated.

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