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Activated Carbon from Coconut Shells as a Modifier of Urea–Formaldehyde Resin in Particleboard Production

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Abstract: Various methods for the effective modification of urea–formaldehyde (UF) adhesives, aimed at enhancing the performance of wood-based materials, have been continually explored worldwide. The aim of this work was to investigate and evaluate the effect of introducing small amounts (0.25–1.5%) of activated carbon from coconut shells (ACCS) in UF adhesive on the properties of particleboard. The performed investigations of the adhesive mixture's properties showed an increase in both viscosity and reactivity. Moreover, the use of loadings of 0.75% and 1% had a positive effect on mechanical properties such as bending strength, modulus of elasticity, and internal bond. In these variants, a delay in the degradation of the adhesive bonds by water was also observed, as indicated by the lower thickness swelling values measured after 2 h. However, under long-term exposure to water, the modification had no considerable effect on the dimensional stability of the boards. Markedly, the addition of 1 and 1.5% of ACCS resulted in a reduction in formaldehyde content, which can be attributed to the excellent adsorption capacity of activated carbon. Overall, a loading of 1% was found to be optimal, resulting in improved strength, enhanced water resistance, and reduced formaldehyde content.

Keywords: urea–formaldehyde adhesive; particleboard; activated carbon; modifier; coconut shells

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1. Introduction

Particleboard is an example of an engineered wood product that was invented in the first half of the 20th century. In most cases, it is composed of wood (mainly softwood) or other lignocellulosic materials. Even though significant time has passed since its invention, its worldwide production still reaches over 100 million m^3 annually, and it is expected to grow to 122 million m^3 by 2029 [1]. Asia and Europe are the largest producers of particleboard. In Europe, the leaders are Germany, Poland, Italy, Austria, and France [2]. In the case of particleboards used to produce furniture for indoor use, their production has reached almost 30 million m^3 per year in Europe alone [1]. Such high production capacity also requires the supply of a considerable amount of binding agents. Urea–formaldehyde (UF) resin is mainly used to produce boards for indoor applications. It is obtained as a product of the condensation of formaldehyde and urea, which demonstrate very good properties such as high strength in dry conditions, a wide range of curing temperatures, light color, ease of handling, and availability at relatively low prices. Due to these properties, its production accounts for 85% of the production of all amino resins globally [3–5].

Despite the widespread use and sale of particleboards over many years, particleboard manufacturers still face challenges in terms of the increasing requirements regarding their performance. The demands usually include, for example, improved strength and durability,

enhanced water resistance, and reduced flammability and formaldehyde emissions [6–9]. These can be achieved by implementing several strategies involving wood and/or adhesive modification, mixing wood with other materials, etc. Among them, adhesive enhancement seems to be the simplest solution. This was confirmed by Demir [10], who stated that when it comes to reducing formaldehyde emission, adhesive modification is indeed the most frequently chosen approach. Interesting outcomes were previously obtained by using carbonaceous substances as a modifier of formaldehyde-based resins. These included mainly nanoscale materials, such as modified multiwalled carbon nanotubes [11,12], graphene nanoplatelets [13], graphene oxide [14], ethylenediamine-functionalized graphene oxide [15], etc. However, taking into account the price of these types of materials, their practical implementation could be limited at the moment; therefore, it is justified to also look for ideas among carbonaceous bulk counterparts.

Research aimed at finding applications for activated carbon seems to be an interesting concept in this perspective. Activated carbon is a predominantly amorphous solid characterized by an extraordinarily large pore volume and specific surface area [16]. The unique properties that activated carbon exhibits have made it suitable for use as an adsorbent against a significantly wide range of both gaseous and water-soluble pollutants [17]. Moreover, importantly for this research, activated carbon has the ability to effectively adsorb formaldehyde from both wastewater and the gas phase [18]. Besides being a great absorber, activated carbon has also been used before as a filler for polymer composites such as, for example, both low- and high-density polyethylene [19,20], polypropylene [21,22], epoxy resin [23,24], and poly(lactic acid) [25]. An interesting example of activated carbon is the one obtained from coconut shells (ACCS). Coconut shells are a well-known precursor among various carbon-rich substances. The product of their conversion to activated carbon is usually characterized by a surface area of about 800–1500 m^2/g [26]. Moreover, the advantages of ACCS include environmental factors related to the use of waste biomass, as well as its low cost, non-abrasive nature, safety in handling, low density, superior specific properties, etc. [27,28]. Ergun et al. [29] recently investigated the possibility of modifying UF resin with ACCS in particleboard production. The authors showed an improvement in the particleboard's mechanical properties, but the investigated loading range was between 1.5 and 7.5%. The aim of the present study was to investigate the effect of incorporating small amounts of ACCS into UF resin used for bonding particleboard. Low quantities up to 1% were previously used by Zamani et al. [30], and the authors obtained promising results, showing an improvement in the properties of medium-density fiberboards (MDFs). However, the authors used activated carbon from sanding dust, and, as numerous studies indicate, the source has a significant impact on crucial features of the obtained carbons [31,32], which in turn may considerably affect the outcomes. Moreover, the effects may vary due to the differences between the gluing of wood fibers and the gluing of wood particles.

In summary, the aim of the presented research was to investigate the effects of urea–formaldehyde adhesive modification with small amounts of activated carbon from coconut shells, ranging from 0.25 to 1.5%, on the properties of manufactured interior-grade particleboard.

2. Materials and Methods

Activated carbon from coconut shells (ACCS) was purchased from Aquafloow (Kraków, Poland), who mainly provided the water filters. ACCS was characterized by a specific surface area of 1100 m^2/g, an apparent density of 0.6 g/cm^3, and a dimensional range of 0.43–1.70 mm. To obtain proper homogenization and adjust the mixture to the pneumatic gluing system, only the fractions of 0.7 mm and smaller were collected and used for adhesive modification. The structural analysis of the sample was performed by Fourier transform infrared (FTIR) spectroscopy. For this purpose, a sample of 1 mg was mixed with 200 mg of potassium bromide (Sigma Aldrich, Darmstadt, Germany). The spectrum was recorded using a Nicolet iS5 spectrometer (Thermo Fisher Scientific, Madison, WI, USA) at a range from 4000 to 500 cm^{-1}, at a resolution of 4 cm^{-1}, registering 32 scans.

The morphology of the activated carbon was evaluated using a Zeiss EVO 10 scanning electron microscope (SEM) (Carl Zeiss Microscopy GmbH, Jena, Germany), operating with an accelerating voltage of 20 kV. Moreover, energy dispersive X-ray spectroscopy (EDS) was used to determine the elemental composition (C, O, Na, Mg, K) of ACCS during the microscopic analyses.

Commercially available UF adhesive supplied by Silekol (Kędzierzyn-Koźle, Poland) was used as a binding agent for particleboard manufacturing. The modification of the adhesive was performed by introducing various amounts of ACCS: 0.25, 0.5, 0.75, 1.0, and 1.5%. Pure UF adhesive without activated carbon was used as a reference variant. Each formulation also contained 3% of an aqueous solution of ammonium nitrate (20 wt. %) as a hardener (Chempur, Piekary Śląskie, Poland). The whole mixture was mixed with a CAT-500 homogenizer (Ingenieurburo CAT, M. Zipper GmbH, Ballrechten-Dottingen, Germany) at 1000 rpm for 90 s. To determine the effect of the performed modification on the properties of the adhesive, the parameters used for the industrial quality control of the resins in the production of the particleboards were investigated [33]. Viscosity was measured using a rotary viscometer Brookfield DV-II+Pro (Middleboro, MA, USA) at 100 rpm using spindle no. 4. Furthermore, gel time at 100 °C was determined according to PN-C-89352-3 [34] as the time needed for the adhesive to lose its fluidity. The pH was measured using a multifunctional Testo 206 pH meter (Pruszków, Poland). Each parameter was determined in triplicate.

Pine wood particles were supplied by the local manufacturer of wood-based materials. The results of the sieve analysis showed that the vast majority of particles fall within the dimensional range between the fractions 1.0 and 2.5 mm. After drying to reach a moisture content of approx. 3%, the particles were glued in a low-speed laboratory gluing machine that was equipped with a pneumatic adhesive spraying system. The following parameters of the boards were assumed: dimensions of 380 mm × 670 mm × 12 mm, gluing degree of 10%, and targeted density of 600 kg/m^3. Two single-layer particleboards were produced for each variant of the adhesive mixture using the following pressing parameters: pressing temperature of 160 °C, unit pressure of 2.5 N/mm^2, and pressing time of 20 s/mm of the final board thickness.

The manufactured particleboards were conditioned for seven days at a relative humidity of 65 ± 2% and a temperature of 21 ± 2 °C. The density of the resultant boards was determined according to EN 323 [35], and the density profile was determined using the density profiler working on the X-ray system GreCon DAX 6000 (Fagus GreCon, Charlotte, NC, USA) with a measurement resolution of 0.02 mm and a measurement rate of 0.05 mm/s. The manufactured particleboards were tested in terms of their mechanical properties such as internal bond (IB), bending strength (MOR), and modulus of elasticity (MOE) according to EN 319 [36] and EN 310 [37], respectively. To investigate the water resistance of the boards, parameters such as thickness swelling (TS) in accordance with EN 317 [38] and water absorption (WA) based on the difference in weight before and after soaking in water were tested. Both parameters were determined after 2 and 24 h of immersion in water. Each of the physical and mechanical properties was determined using 12 samples. The formaldehyde content was measured in triplicate using the perforator method according to EN 120 [39]. The formaldehyde content in a collected aqueous solution was measured according to a well-described standard ammonium acetate and acetylacetone method with the use of a Biosens UV-5600 spectrophotometer (Biosens, Warsaw, Poland) at 412 nm.

The statistical analyses were performed with Statistica 13.3 software, using the one-way analysis of variance (ANOVA). Post hoc comparison was carried out with the HSD Tukey test. An obtained value of $p < 0.05$ was considered to be statistically significant.

3. Results and Discussion

Figure 1 shows the FTIR spectrum of the ACCS, which shows a rather simple course. The spectrum contains only two well-defined peaks at 3463 cm^{-1}, corresponding to the -OH vibrations of the phenolic group; and at 1635 cm^{-1}, which can be attributed to the C=O

stretching vibrations of aldehydes, ketones, and carboxylic acids [40,41]. A similar course was observed in the case of the activated carbon used for the phosphate removal presented by Wang et al. [42] and the activated carbon used for the synthetic dye removal presented by Unugul and Nigiz [43]. The relatively low number of observed peaks may indicate a high conversion temperature, which unfortunately was not declared by the producer. According to Allwar [44], the intensity of the bands between 1234 and 1153 cm^{-1} decreases with increasing temperatures used for the activated carbon preparation.

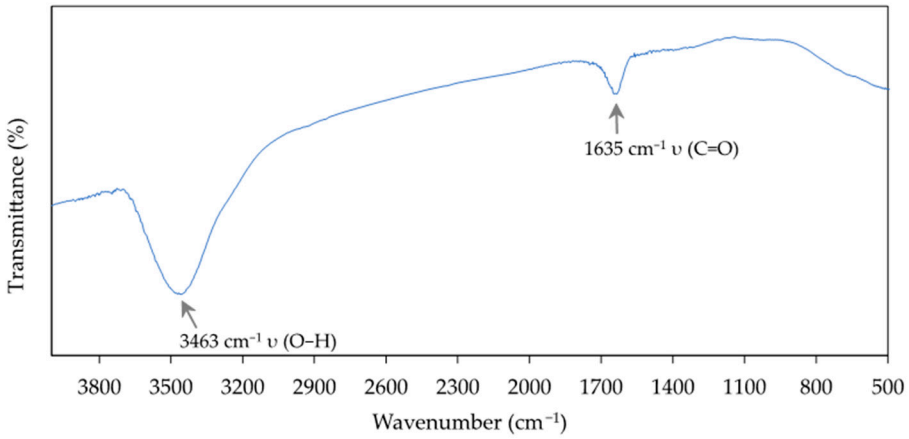

Figure 1. FTIR spectra of activated carbon.

The SEM images taken at different magnifications are presented in Figure 2. As the micrographs taken at lower magnifications show, the activated carbon particles had a regular shape and a rather smooth surface without the cracks, crevices, and large holes noticed previously, for example, by Saka [45]. The images taken at greater magnifications showed the visibly porous morphology of the activated carbon surface. This seems to be advantageous because high porosity indicates a high formaldehyde adsorption capacity [46].

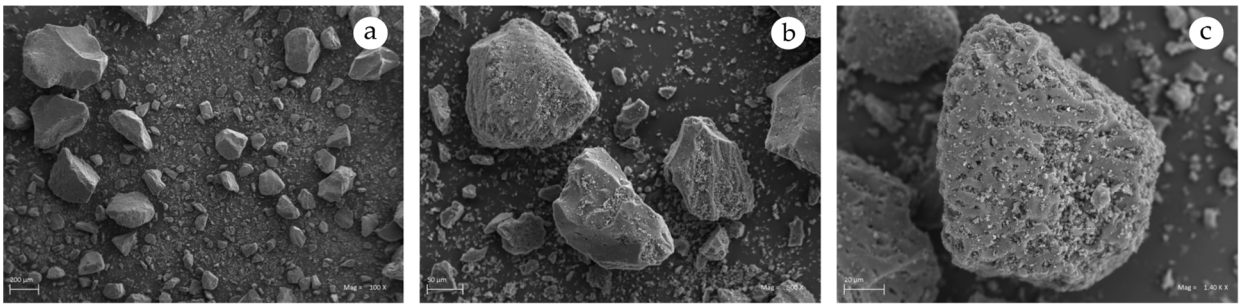

Figure 2. SEM images of activated carbon at different magnifications: (**a**) 100×, (**b**) 500×, (**c**) 1400×.

The results of the EDS analysis showed mainly carbon and trace amounts of oxygen, sodium, magnesium, and potassium (Table 1). This is consistent with the results presented by Aljundi and Al-Dawery [47], who showed that the carbon content ranged between 97.39 and 93.58%, depending on the production temperature. Alkaline metal oxides such as K_2O, MgO, Na_2O, and CaO are usually the source of the inorganic elements found in trace amounts, and their presence can affect, for example, the hydrophilicity of activated carbon [48].

Table 1. Elemental composition of activated carbon.

Weight (%)				
C	**O**	**Na**	**Mg**	**K**
97.6	2.0	0.1	0.1	0.2

The results of the properties of the adhesive mixtures, which are crucial for particleboard production technology, are presented in Table 2. It was found that the addition of activated carbon in the amounts up to 1% did not cause any statistically significant changes in the gel time. However, the maximum addition of 1.5% resulted in a significant acceleration of the gel time by approx. 15% when compared to the non-modified adhesive. This is particularly important because it is the most fundamental property when the pressing technology, i.e., pressing time and pressing temperature, is set, which in turn may affect the production costs and financial efficiency in a considerable way [49,50]. Zamani et al. [30] found that small loadings of activated carbon can contribute to a slight increase in curing reaction enthalpy, which is an indication of improved reactivity as well. The reason could be the ability of ACCS to absorb water both in the surface and subsurface pores [51,52]. As a result, the curing process of the UF adhesive is promoted. This is particularly beneficial because increasing reactivity could potentially shorten the pressing time, which in turn, taking into account the energy consumption of this process, could increase the financial efficiency of production. Furthermore, the addition of activated carbon in an amount exceeding 0.25% resulted in a statistically significant increase in viscosity. In the variant with the maximum modifier content, the viscosity increased by approx. 11%. For comparison, in another study, the addition of 1.5% of ACCS resulted in increased viscosity by approx. 6% [29]; however, this may depend on the properties of the carbon itself, the fraction used, etc. Besides the water absorption mentioned before, the addition of any organic or inorganic filler, even in small quantities, leads to a thickening of the UF adhesive, which usually also means an increase in viscosity [53]. Moreover, it was found that the addition of activated carbon did not significantly affect the pH of the adhesive mixtures.

Table 2. Properties of UF adhesive mixtures modified with ACCS.

Activated Carbon Content (%)	Gel Time (s)	Viscosity (mPa·s)	pH
0.00	92 ± 4^{b}	282.7 ± 3.5^{a}	6.21 ± 0.04^{a}
0.25	95 ± 4^{b}	285.0 ± 2.6^{a}	6.20 ± 0.01^{a}
0.50	94 ± 7^{b}	302.3 ± 3.5^{b}	6.21 ± 0.03^{a}
0.75	99 ± 5^{b}	305.0 ± 2.6^{bc}	6.20 ± 0.03^{a}
1.00	94 ± 5^{b}	314.0 ± 6.2^{c}	6.20 ± 0.02^{a}
1.50	79 ± 2^{a}	314.3 ± 4.0^{c}	6.21 ± 0.03^{a}

a, b, c ...—homogeneous groups according to the Tukey test.

The appearance of the produced particleboards is shown in Figure 3. No effects from the addition of activated carbon up to 1.5% were observed. On the contrary, in the case of the greater carbon loadings, the obtained boards darken and become grayer [29]. Therefore, with no side effects compromising the aesthetics of the material, this can be considered as the advantage of smaller loadings.

Density is one of the most important parameters of particleboard. It can noticeably affect the vast majority of a board's properties and can be used to control its targeted functional properties [54]. As shown in Figure 4a, the addition of activated carbon had no significant effect on the obtained density value ($p > 0.05$). The average result for each variant was very close to the assumption, and the difference did not exceed 5 kg/m^3. Moreover, the most common U-shaped course of the density profile was observed for manufactured particleboards (Figure 4b). This phenomenon is highly beneficial because denser subsurface layers contribute to a more efficient transfer of both tensile and compressive stresses during bending [55]. However, the implemented adhesive modification did not affect the density profile.

Figure 3. Appearance of the laboratory-made particleboards bonded with UF adhesive modified with ACCS.

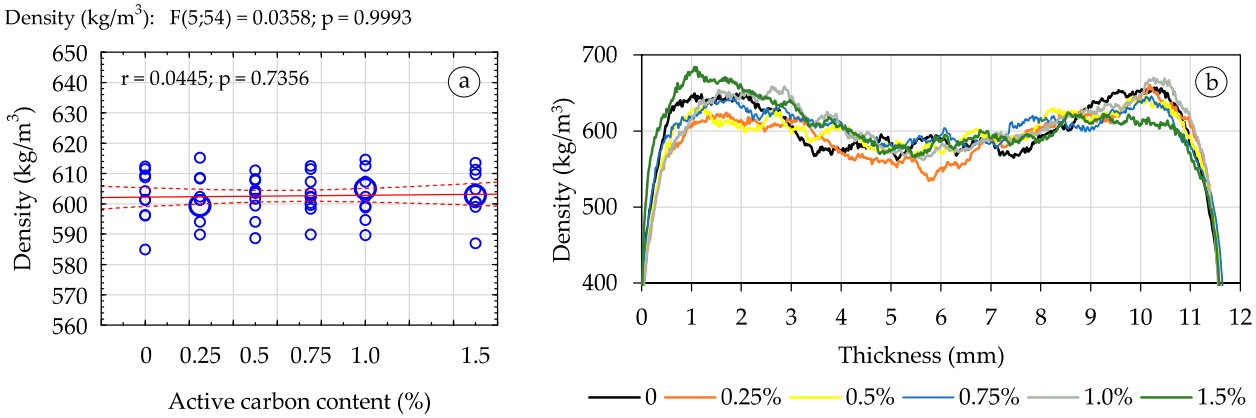

Figure 4. The density of the particleboards: (**a**) average density, (**b**) density profile.

The results of mechanical properties such as IB, MOR, and MOE are summarized in Figure 5. The outcomes of the conducted analysis have shown that the modification of UF affected each strength parameter in a statistically significant way ($p < 0.05$). Based on the IB results, it was found that the addition of ACCS in amounts of 0.25 and 0.5% did not cause significant changes. Perhaps, in this case, the loading was too small to affect the strength of the entire board. Markedly, the increase in the ACCS content to 0.75 and 1% resulted in a statistically significant improvement. In these cases, the average value was higher by approx. 25% compared to the non-modified board. However, a further increase in the loading to 1.5% resulted in a decrease in IB to the level of the reference board.

The analysis of the average MOR and MOE values revealed a similar tendency. Particleboards characterized by increased IB also exhibited improved MOR and MOE values, respectively. The reason was most likely the enhancement of the bonding quality caused by the introduction of ACCS. The reasons for the positive effect of the activated carbon addition may be the increase in the degree of polycondensation due to its tremendous specific surface area and reactivity, as well as a thickening effect that usually improves the morphology of the cured bond lines and, consequently, also the strength of manufactured wood-based materials. On the other hand, the lack of further enhancement and the reduction in strength to the level of the reference board may indicate that the loading of 1.5% was too high in this case. Activated carbons demonstrate the tendency to agglomerate [56]. Considering that only a small fraction was used to modify the adhesive, it can be assumed that

the reason was the agglomeration of the modifier's particles, which negatively affects the quality of the adhesive bonds and hinders the transfer of stresses within the bond [57]. As part of future work, it would be interesting to investigate how a chemical functionalization of the activated carbon surface by the attachment of functional groups, which can improve dispersion within the polymer matrix, could affect the recommended loading range. In the case of the variant containing 1.5% of ACCS, the effect was different from the significant improvement shown by Ergun et al. [29]; however, this only proves that many factors, such as, for example, the dimensional fraction of filler affecting the agglomeration [58], can influence the obtained results.

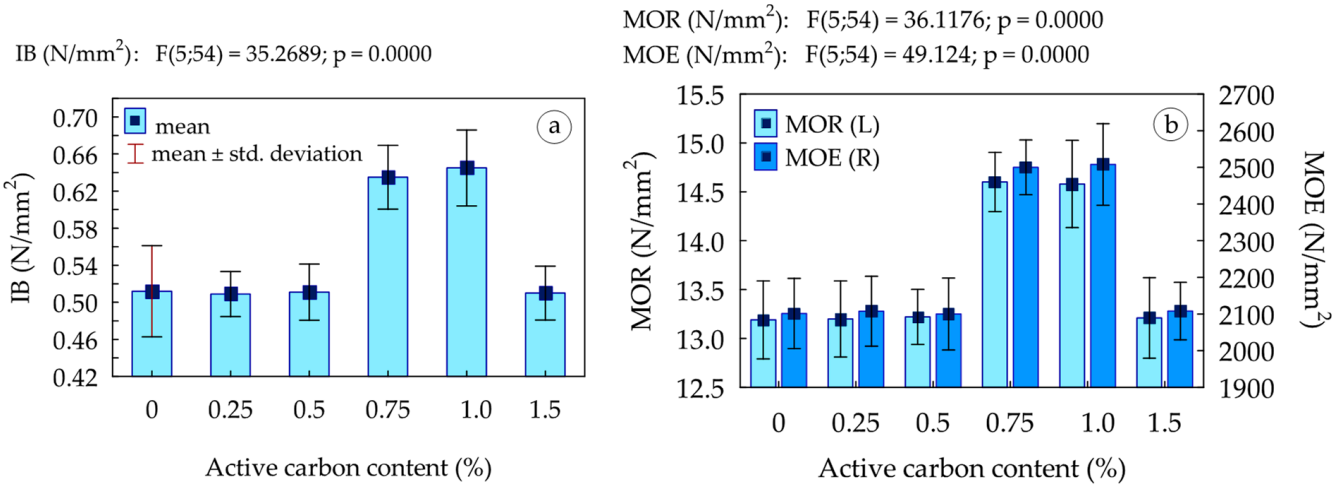

Figure 5. Mechanical properties of the particleboards: (**a**) internal bond; (**b**) bending strength (MOR) and modulus of elasticity (MOE). Error bars represent the standard deviation.

The results of TS and WA, indicating the overall water resistance of the manufactured particleboard, are presented in Figure 6. It was found that the applied modification with ACCS affected only the TS results determined after 2 h of soaking ($p < 0.05$). In the case of variants containing 0.75 and 1%, the recorded value of TS was reduced by approx. 16% compared to the reference particleboard. Considering that this phenomenon was observed only for variants characterized by the improved IB, which is a basic indicator of adhesive performance in the particleboard, the reason for the improved water resistance was most likely the reinforcement of the adhesive bonds [59–61]. However, it seems that 24 h was enough time for a hydrolytic degradation of the UF adhesive bonds to progress, which led to the average TS values at the reference board level [62,63]. Nevertheless, the increase in swelling and water absorption observed for the variant containing 1.5% ACCS in the previous study was not observed [29]. This proves again that further research aimed at investigating the effects of factors such as the activated carbon preparation method, activated carbon characteristics, dimensional fraction, homogenization conditions, pressing conditions, and initial properties of adhesive on the expected outcome is still needed. However, it can be concluded that, in this case, the addition of ACCS delayed the degradation of UF adhesive bonds, which was shown by short-term improvement, but had no effect under long-term exposure to water.

The results of the formaldehyde content analyses are shown in Figure 7. Statistical analysis showed that the modification had a significant effect on the formaldehyde content ($p < 0.05$), but the effect varied depending on the amount of added modifier. It was found that a significant reduction was observed only for variants containing 1 and 1.5% of ACCS. Compared to the reference board, the recorded formaldehyde content was lower by approx. 16%. This effect was caused by the ability of activated carbon to adsorb formaldehyde, attributed to its high porosity and specific surface area [46]. A detailed description of the mechanism of formaldehyde adsorption by activated carbon can be found in the recent paper by Kang et al. [18]. This effect is consistent with the results of research on MDF

boards performed by Darmawan et al. [64] and Zamani et al. [30], who also observed the reduction in formaldehyde release. This is beneficial due to the harmful nature of the formaldehyde emitted and the regulations of emissions that follow the growing awareness of health risks that may occur from long-term exposure [65].

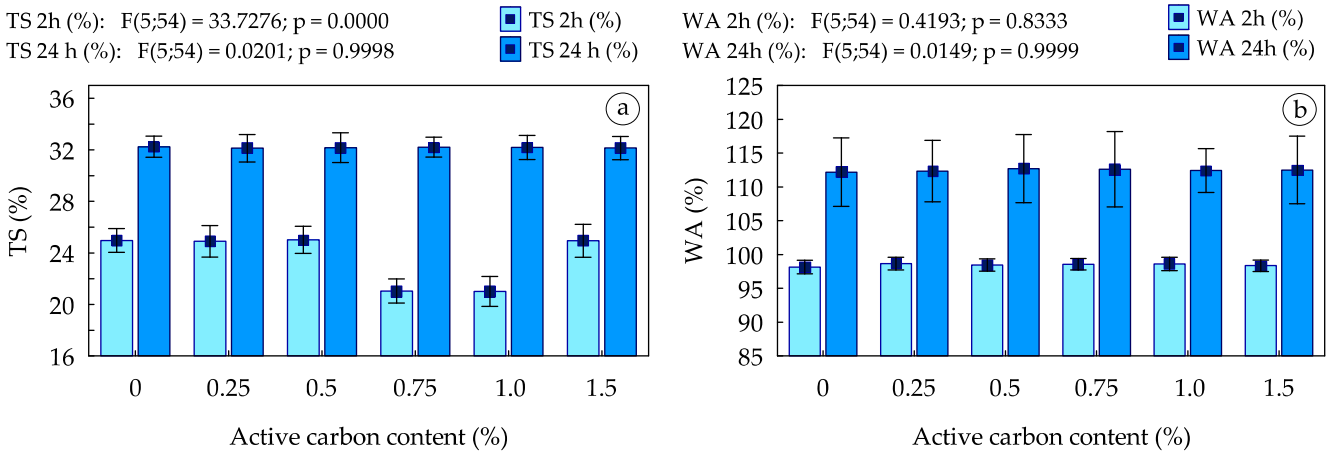

Figure 6. Water resistance of particleboards after 2 and 24 h of soaking in water: (**a**) thickness swelling, (**b**) water absorption. Error bars represent the standard deviation.

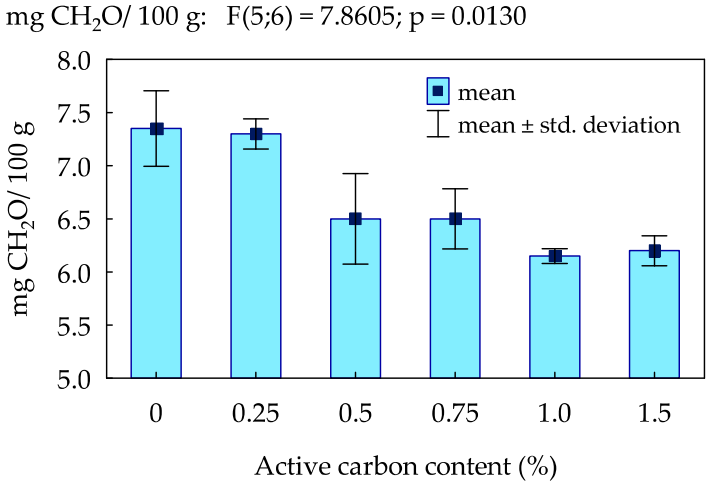

Figure 7. Formaldehyde content in manufactured particleboard.

4. Conclusions

Based on the conducted research, it was found that after adjusting the amount, activated carbon from coconut shells can be used as an effective modifier of UF resin. The introduction of activated carbon significantly changes the properties of the adhesive mixture. As the loading increases, the viscosity of the adhesive increases; moreover, the maximum addition of 1.5% also increases the reactivity of the resin. The use of activated carbon in an amount of up to 1.5% does not significantly influence the aesthetic properties and density of the manufactured particleboards. However, it can affect both the physical and mechanical properties of the resultant materials. In the cases of the 0.75 and 1% loadings, the produced particleboards show better mechanical properties (improved internal bond, bending strength, and modulus of elasticity) and delayed degradation in water, as indicated by lower valuesof thickness swelling measured after 2 h of soaking in water. In the case of long-term exposure to water, the modification has no effect. A comparison of the obtained results with the results of other works also indicates that the use of activated carbon as a UF resin modifier is a complex issue, and the expected effect may vary depending on many factors. Furthermore, in the cases of the additions of 1 and

1.5%, a reduced formaldehyde content in the boards is also noted. Based on all of the results, it was concluded that the variant containing 1% activated carbon from coconut shells can be considered optimal, for which increased strength, enhanced water resistance, and reduced formaldehyde content were observed.

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