

Alkali Modification of Cotton (*Gossypium hirsutum* L.) Stalks and its Effect on Properties of Produced Particleboards

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This study evaluates the effect of 1% to 5% NaOH treatments of cotton stalk particles on the chemical composition and physical and mechanical properties of particleboards produced with this material. Gas chromatography and Fourier transform infrared (FTIR) spectroscopy indicated that the extractive, hemicellulose, and lignin content of the particles decreased during the alkali treatments, whereas the cellulose content increased. Thermogravimetric analysis (TGA) indicated that the untreated particles exhibited higher thermal stability than the particles treated with NaOH. The decrease in thermal stability of alkali-treated particles seems to be due to degradation of chemical components. Alkali treatments raised water absorption (WA) and thickness swelling (TS) of the produced particleboards. Except for 1% NaOH, the treatments reduced the modulus of elasticity (MOE), modulus of rupture (MOR), and internal bond (IB) strength in relation to the board made with untreated particles. The 1% NaOH treatment of cotton stalks was more effective and beneficial, leading to particleboards with satisfying MOE, MOR, and IB values that met the requirements for general-purpose particleboards used in dry conditions, as indicated by the TS-EN 312 (2012) standard.

Keywords: Cotton stalks; NaOH treatment; Chemical composition; Thermal stability; Physical and mechanical properties

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INTRODUCTION

The demand for wood products has increased in the last decades due to rapid growth in world population and thus industrialization. Therefore, the use of agricultural and other alternative fiber sources in addition to wood-based fibers, recycling of various raw materials after usage, more efficient technologies, and new and higher quality products will play important roles in regulating the supply and balance in the wood products industry in the future (Oner and Aslan 2002; Yaman and Gencer 2005; Yasar *et al.* 2010a).

Non-wood lignocellulosic resources on earth are available in different forms as agricultural waste. The stalks obtained from cotton production are among the most important agricultural waste with regard to quantity. In Turkey, approximately 2.5 million tons of stalks out of more than 6 million tons from cotton cultivation on an area of 6800 km² can be collected annually. Large quantities of stalks generated by the regular cotton cultivation are either left in the fields or used as fuel (Bascetincelik *et al.* 2005).

Efforts have been undertaken to use non-wood lignocellulosic sources as alternative raw materials in the production of particleboards. Agricultural residues (Guler 2015), eggplant stalks (Guntekin and Karakus 2007), wheat stalks (Mo *et al.* 2003; Halvarasson *et al.* 2005), rice husks (Leiva *et al.* 2007; Ndazi *et al.* 2007a), sugar cane (Lee *et al.* 2006),

cotton stalks (Guler *et al.* 2001; Guler and Ozen 2004), sunflower stalks (Bektas *et al.* 2005; Guler *et al.* 2006), vine pruning waste (Yasar *et al.* 2010b), and peanut husks (Guler and Buyuksari 2011) have been used as raw materials to produce particleboards.

Particles and fibers can be treated to improve certain properties of boards. These treatments include acetylation (Gomez-Bueso *et al.* 1999; Abdul Khalil *et al.* 2007), enzymes (Zhang *et al.* 2003), heat (Boonstra *et al.* 2006), NaOH (Ndazi *et al.* 2007a), boric acid (Var *et al.* 2002; Zaidon *et al.* 2007), and other chemicals (Guntekin *et al.* 2009; Yasar *et al.* 2010b).

Alkali treatment is commonly used to modify the fiber surface in order to increase the proportion of reactive OH groups (Ray and Sarkar 2001; Mwaikambo and Ansell 2002; Ndazi *et al.* 2007a,b). Alkali treatment results in better adhesion between particles and fibers due to the increased surface roughness and the higher proportion of OH groups, which has significant effects on various properties of the boards (Gardner and Elder 1988; Lopattananon *et al.* 2008; Guntekin *et al.* 2009; Yasar *et al.* 2010b; Guntekin 2012).

In this study, treatment in NaOH solutions with different concentrations (1 to 5%) was applied to particles from cotton stalks. The changes in the chemical and thermal properties of the particles were demonstrated, and their effects on the physical and mechanical properties of the boards produced from these particles were evaluated.

EXPERIMENTAL

Material

Cotton stalks were collected from Karaisali, Turkey. The stalks were ground using a hammer mill to 1 to 3 mm and dried naturally in an air-conditioned room for two weeks.

Alkali Treatment of Particles from Cotton Stalks

Cotton stalk particles were soaked in 1%, 3%, or 5% w/v NaOH at room temperature for 24 h. The treated particles were sieved, washed with 10% acetic acid solution to neutralize residual NaOH, and rinsed with excess of water. The rinsed particles were dried in an air-conditioned room for two weeks.

Preparation of Chemical Test Samples and Application of Experiments

The untreated and alkali-treated particles were ground in a Retsch SK 1 mill (Haan, Germany) to 40 to 100 mesh. After grinding, the samples were extracted with cyclohexane:ethanol (2:1 v/v) and then ethanol in a Buchi Extraction System B-811 (Flawil, Switzerland). The content of the dissolved extractives was obtained as a percentage of oven-dried material weight.

The method of Dill *et al.* (1984) was slightly modified to perform acid hydrolysis on the samples extracted by cyclohexane and ethanol. Briefly, 1 g of material (on an oven-dry basis) was primary hydrolyzed in 20 mL of 72% H₂SO₄ at 30 °C for 2 h, diluted by the addition of 360 mL of distilled water, and secondarily hydrolyzed in a JP-Selecta autoclave (SterilMAX, Barcelona, Spain) at 120 °C for 30 min (Yasar *et al.* 2010a). After filtering, insoluble Klason lignin was dried at 105 ± 2 °C and expressed as a percentage of oven-dried material weight. Polysaccharide units (monosaccharides) were collected in the hydrolysate for gas chromatography analysis.

The acid chlorite method introduced by Browning (1967) was used to prepare holocellulose from the samples subjected to extraction with cyclohexane and ethanol. The

ASTM D1103 (1980) method was applied to determine the content of α -cellulose and hemicelluloses. The weight of hemicelluloses was calculated as the difference between α -cellulose and holocellulose; α -cellulose and hemicellulose content was established as a percentage of oven-dried material weight.

The monosaccharide composition was analyzed on a Perkin Elmer Autosystem XL gas chromatograph (Waltham, USA) (Cao *et al.* 1997). The monosaccharide content was expressed as a percentage of oven-dried material weight.

The samples that passed through 40 to 100 mesh were converted to homogenous powder by a ball mill and submitted to Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analyses (TGA). First, 10 mg of homogenous powder were pressed to form a self-supporting pellet by compression of 602 N/mm² after dispersion in a matrix of 1000 mg KBr. FTIR spectra were collected between 4000 and 400 cm⁻¹ with a resolution of 4 cm⁻¹ in a Perkin Elmer BX FTIR spectrometer (Waltham, USA) at room temperature. For TGA, 5 mg of homogenous powder were analyzed on a Perkin Elmer SII Diamond thermograph (Waltham, USA) at a heating rate of 10 °C/min in the temperature range of 25 to 900 °C under nitrogen.

Preparation of Particleboards and Determination of Properties

The untreated and alkali-treated particles were dried at 102 ± 5 °C until maximum 3% moisture content was obtained. Based on the oven-dry particle weight, 10% liquid urea formaldehyde (UF) resin (Orma Company, Isparta, Turkey) was applied as adhesive; 35% NH₄Cl (ammonium chloride solution) (Orma Company, Isparta, Turkey) was used as hardener in which 10% was added based on the adhesive weight. The boards were prepared by spraying the adhesive on the particles in a drum blender. The blended particles were then placed in a 31 × 35 cm wooden box on a metal plate, which was used in carrying the mat to the hot press, and manually formed. The hand-formed mats were hot-pressed at 150 ± 5 °C under 2.5 to 3 N/mm² pressure for 5 min to achieve the 12 mm target board thickness. Experimental boards were conditioned in a chamber at 20 °C and a 65% relative humidity for 48 h. Target density of the boards was 0.5 g/cm³ (Guntekin *et al.* 2009).

Standards TSE-EN 310 (1999), TSE-EN 317 (1999), and TSE-EN 319 (1999) were applied to determine MOE, MOR, IB, thickness swelling (TS), and water absorption (WA) of the particleboards.

RESULTS AND DISCUSSION

The proximate analysis of untreated and NaOH-treated particles produced from cotton stalks are presented in Table 1. The extractive content in cotton stalks exposed to NaOH solution of 1%, 3%, and 5% decreased by 5%, 35%, and 38%, respectively, compared with those that were not treated. Troedec *et al.* (2008) and Carvalho *et al.* (2010) reported that alkali treatment removes extractive content, oils, and waxes from fibers.

Compared with untreated cotton stalks, the lignin content in the stalks decreased by 4%, 27%, and 36%, respectively with the three NaOH treatments (1%, 3%, and 5%, respectively). Some studies emphasize that lignin can be decomposed from plant fibers by alkali treatment only at high concentration, high temperature, and for a long time (Albano *et al.* 2001; Ray and Sarkar 2001; Rajulu *et al.* 2002), while others report that lignin decomposes at high rates already at low concentration and at room temperature (Ndazi *et*

al. 2007a,b). The findings here are consistent with studies showing that lignin decomposes in noticeable amounts in a low temperature, low concentration treatment.

Table 1. Chemical Components of Cotton Stalk Samples

Component	Untreated	1% NaOH	3% NaOH	5% NaOH
Extractives	5.24 (0.04)*	4.97 (0.06)	3.41 (0.03)	3.26 (0.04)
Lignin	23.27 (0.06)	22.42 (0.05)	16.95 (0.03)	14.82 (0.03)
Hemicelluloses	23.29 (0.05)	21.93 (0.05)	14.97 (0.04)	13.41 (0.03)
Cellulose	42.33 (0.06)	44.92 (0.06)	57.68 (0.07)	61.09 (0.08)

*: Standard deviation

The α -cellulose content in the cotton stalks increased by 6%, 36%, and 44%, respectively, with the treatments, while the hemicellulose decreased by 6%, 36%, and 42%, respectively, compared with untreated cotton stalks. The increase in α -cellulose content can be explained by the relative decrease in the lignin, hemicellulose, and extractives, which are more unstable in alkali treatment. Moreover, cellulose is more resistant to alkali treatment than lignin, hemicellulose, and extractives (Gassan and Bledziki 1999; Ray and Sarkar 2001; Ndazi *et al.* 2007b; Troedec *et al.* 2008; Carvalho *et al.* 2010).

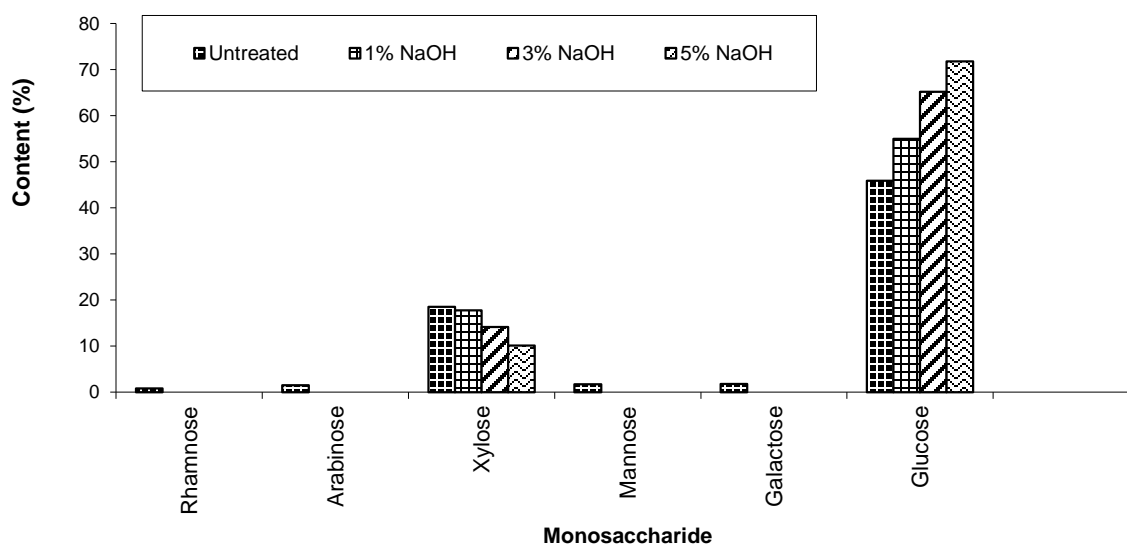


Fig. 1. Monosaccharide composition of cotton stalk samples

The monosaccharide composition of the cotton stalk samples is shown in Fig. 1. The content of rhamnose, arabinose, xylose, mannose, galactose, and glucose in untreated samples was 0.82%, 1.48%, 18.5%, 1.67%, 1.74%, and 45.8%, respectively. The alkali-treated cotton stalks showed no content of rhamnose, arabinose, xylose, mannose, or galactose. The xylose (pentose) content in treated cotton stalks decreased by 4%, 23%, and 45%, respectively with increasing NaOH concentration, while the glucose content increased by 20%, 42%, and 57%, respectively, all results compared with the untreated stalks. The increase in the glucose content indicated the relative increase in the cellulose content in the treated samples. Previous studies demonstrated that hexose in hemicellulose was more prone to decomposition than pentose when treated with alkali (Sarkar *et al.* 1948; Gassan and Bledzki 1999).

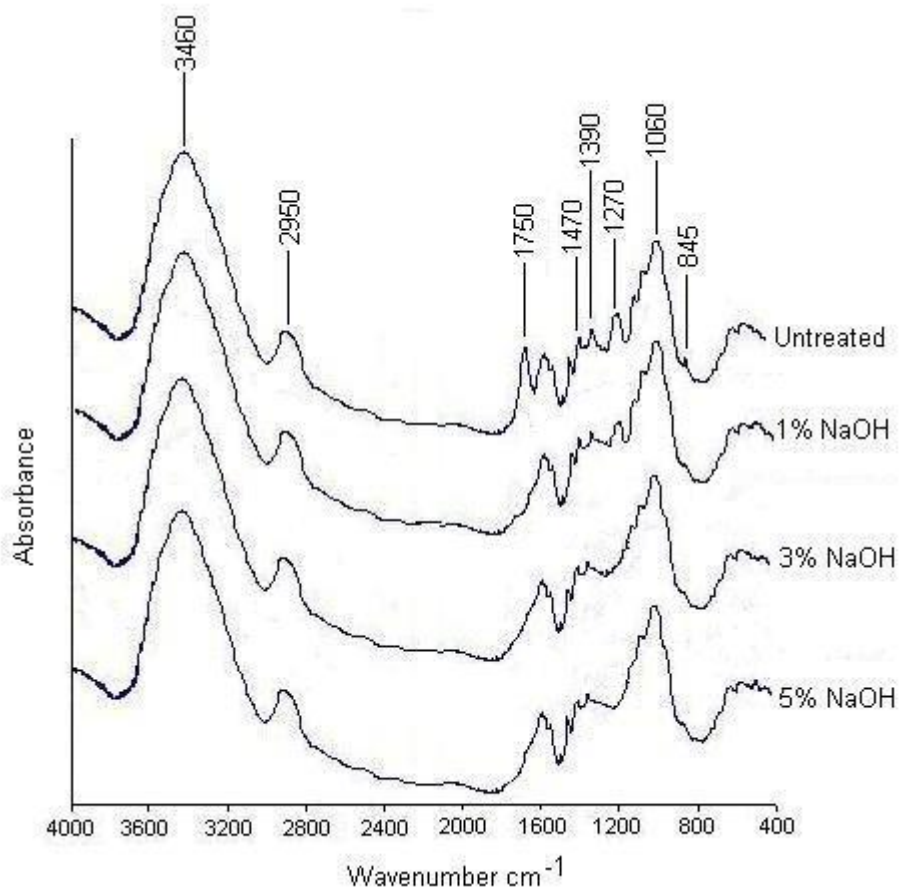


Fig. 2. Infrared spectra of cotton stalk samples

Figure 2 shows the FTIR spectra of untreated and alkali-treated samples with the wavenumber range from 4000 to 400 cm^{-1} . The bands at 3460, 1750, 1470, 1390, 1270, 1060, and 845 cm^{-1} were used to evaluate the main chemical components including cellulose, hemicelluloses, and lignin. The intensities of these bands were compared after dividing by the intensity of the band at 2950 cm^{-1} , which was ascribed to C-H stretching in methyl and methylene groups (Tsuboi 1957) and used as an internal standard (Sinha and Rout 2009; Mahato *et al.* 2014) (Table 2). The band at 3460 cm^{-1} was attributed to H-bonded H-O stretching (Tsuboi 1957; Pandey 1999 and 2005). The value increase in this band in alkali-treated samples indicated increased concentration of OH groups. The band at 1750 cm^{-1} represents the C-O- stretching of the carboxyl and acetyl groups of hemicelluloses (Liang *et al.* 1960). This band appeared only in the spectrum of the untreated sample, probably due to the elimination of acetyl groups in hemicelluloses in alkali-treated samples (Sarkar and Mazumdar 1955). The band at 1470 cm^{-1} was associated with the CH_3 deformation in lignin (Sinha and Rout 2009; Mahato *et al.* 2014). The band intensity at 1470 cm^{-1} was consistently reduced in the spectra of alkali-treated samples, indicating the degradation of lignin due to alkali treatments. The band at 1390 cm^{-1} was attributed to the C-H deformation in lignin, cellulose, or xylan (Sinha and Rout 2009; Mahato *et al.* 2014). This band was higher in intensity in the untreated sample than in the alkali-treated samples. The band at 1270 cm^{-1} was ascribed to the C-O stretching in the acetyl groups in xylan (Sinha and Rout 2009; Mahato *et al.* 2014). The intensity value of this band was higher in the untreated sample but not present in the spectra after 3% NaOH

treatment due to the removal of acetyl groups in hemicelluloses. The band at 1060 cm^{-1} was related to the aromatic C-H in plane deformation and C-O deformation for primary alcohol in lignin (Sinha and Rout 2009; Mahato *et al.* 2014). The intensity value of this band in the spectra decreased consistently due to alkali treatments. The band at 845 cm^{-1} was associated with the aromatic C-H out of plane vibration in lignin (Sinha and Rout 2009; Mahato *et al.* 2014). This band was detected only in the untreated sample. The intensity changes for both bands (1060 and 845 cm^{-1}) are explained by lignin degradation in the alkali-treated samples.

Table 2. Absorbance Intensity Ratio (A_i/A_{2950}) of Infrared Spectra of Cotton Stalk Samples

Position of Band (cm^{-1})	Untreated	1% NaOH	3% NaOH	5% NaOH
3460	1.195	1.209	1.212	1.213
2950	1.000	1.000	1.000	1.000
1750	0.983	-	-	-
1470	0.994	0.957	0.876	0.863
1390	1.025	1.016	0.953	0.951
1270	1.039	1.022	-	-
1060	1.136	1.125	1.112	1.110
845	0.843	-	-	-

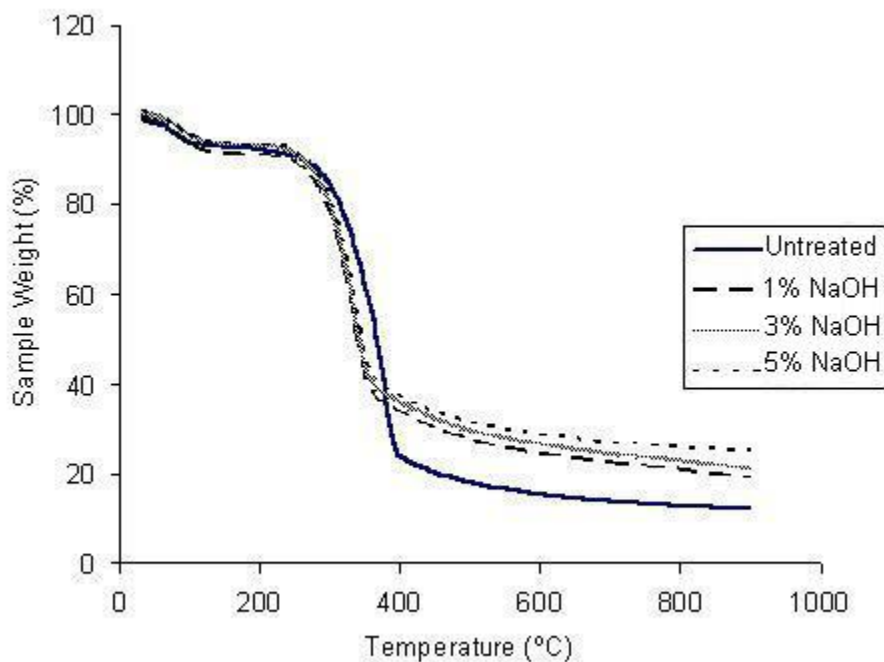


Fig. 3. Effect of alkali treatment on thermal stability of cotton stalks.

During the thermal degradation of plant biomass, water and part of the extractive content is eliminated at approximately $225\text{ }^{\circ}\text{C}$ (Turner and Mann 1981). Once different forms of hemicellulose are decomposed, the actual degradation at temperatures above approximately $225\text{ }^{\circ}\text{C}$ continues with the degradation of extractives, lignin, and cellulose (Turner and Mann 1981; Meszaros *et al.* 2007).

TG and DTG thermographs obtained from the TGA of untreated and alkali-treated cotton stalk particles are shown in Figs. 3 and 4. The elimination of water and primary volatile compounds in the samples was completed at up to 155 °C. During the secondary stage of decomposition starting at 234 °C the hemicellulose decomposed. The degradation in the alkali-treated samples was completed in a single stage from 234 °C to 376 °C / 390 °C, while there was a transition in the untreated sample at around 352 °C. The actual degradation took place in two stages between 234 °C and 413 °C.

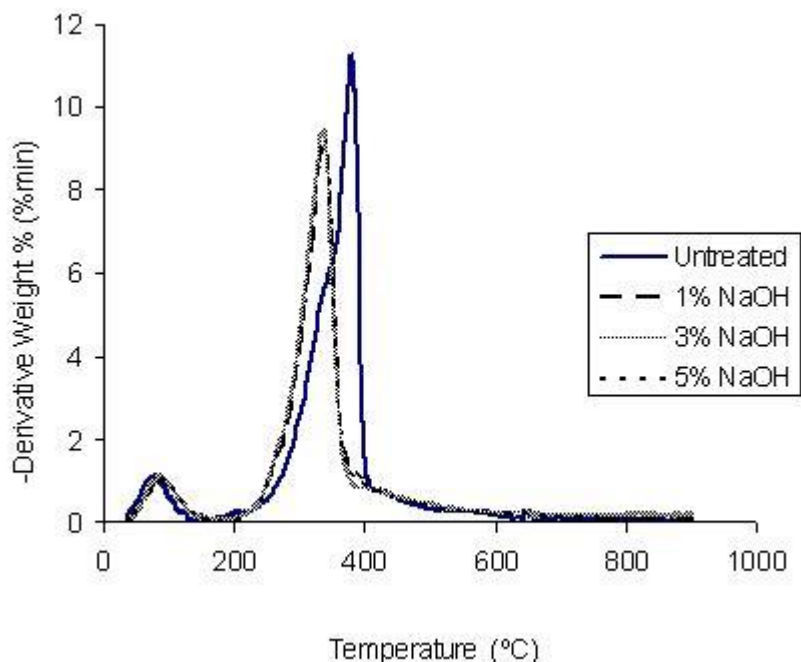


Fig. 4. Effect of alkali treatment on the decomposition rate of cotton stalks

The shoulder at around 352 °C was not observed in the decomposition curves of the alkali-treated samples, but it was observed in the untreated sample. This result suggested that thermal degradation occurred in two main reaction zones for the untreated sample. Similar curves of the thermal degradation of lignocellulosic fibers have been reported earlier (Mansaray and Ghaly 1999; Alvarez and Vazquez 2004; Ndazi *et al.* 2007b). The first reaction range at around 352 °C in the untreated sample resulted in the final degradation of hemicelluloses (Alvarez and Vazquez 2004), conversion of volatile products (Mansaray and Ghaly 1999), and splitting of aliphatic chains of lignin and C-C bonds of the aromatic ring (Le Van 1989). The first reaction range was not observed in the treated samples due to significant decomposition of hemicellulose, lignin, wax substances, and natural oils already during the alkali treatment (Meszaros *et al.* 2007; Ndazi *et al.* 2007b).

The second reaction range from 352 °C to 413 °C took place in the untreated sample; cellulose, lignin, and extractives were decomposed (Le Van 1989; Alvarez and Vazquez 2004; Meszaros *et al.* 2007). The maximum degradation of treated samples between 234 °C and 390 °C was mainly related to cellulose decomposition, as hemicellulose, lignin, and extractives were decomposed to a significant extent through alkali treatment, and some extractives were thermally degraded before 234 °C (Ndazi *et al.* 2007b). Therefore, the difference between the maximum degradation temperatures of the untreated sample and the alkali-treated samples was a minimum of 23 °C.

Table 3. Thermal Degradation Temperatures and Residue Weight of Cotton Stalk Samples

Sample	T_0 (°C)	T_1 (°C)	T_f (°C)	Residue at 900 °C (%)
Untreated	234	352	413	14.38
1% NaOH	234	-	390	19.21
3% NaOH	234	-	380	21.78
5% NaOH	234	-	376	25.64

Table 3 shows the thermal transition in the untreated and alkali-treated cotton stalk particles. T_0 represents the temperature at which degradation started, while T_1 represents the temperature at which the first stage of degradation was finalized and the second stage started in the untreated sample. T_f is the final temperature of thermal degradation and represents the temperature at which degradation was finalized and mass residues were formed. A part of the residue is in the form of cellulose that is exposed to chemical transformation and rearrangement during the thermal degradation. This char part of the residue is attributed to formation of a condensed polycyclic aromatic structure from cellulose (McGrath *et al.* 2003). The continuous increase in the alkali concentration during the treatment and continuous reduction of lignin, hemicellulose, and extractives in the samples led to the relative increase in cellulose content. Therefore, the continuous increase in the amount of residues in the treated samples is explained by the relatively continuous increase in cellulose content.

Table 4. Physical and Mechanical Properties of Boards Produced with NaOH-Treated and Untreated Particles of Cotton Stalks

Board Type	WA (%)	TS (%)	MOE (N/mm ²)	MOR (N/mm ²)	IB (N/mm ²)
Untreated	97 (3.1)*	23 (0.8)	1322 (58)	8.1 (0.5)	0.34 (0.03)
1% NaOH	104 (4.6)	26 (0.4)	1812 (30)	13.2 (0.7)	0.32 (0.02)
3% NaOH	112 (4.9)	34 (1.3)	1314 (21)	8.0 (0.4)	0.21 (0.03)
5% NaOH	126 (7.9)	42 (2.8)	1071 (35)	5.2 (0.4)	0.18 (0.02)

WA: water absorption, TS: thickness swelling, MOE: modulus of elasticity, MOR: modulus of rupture, IB: internal bond strength, *: standard deviation

The physical and mechanical properties of boards produced with NaOH-treated and untreated particles of cotton stalks are summarized in Table 4. Boards from treated particles presented higher WA and TS compared to untreated particles. The requirements of maximum swelling of 16% for load-bearing applications (TS-EN 312 2012) were not achieved. Alkali treatments cause a consistent decrease in the extractives, hemicelluloses, and lignin, whereas the cellulose content increased continuously in the particles. Alkali treatments cause swelling of the cellulose crystalline structure, which may promote water entrance (Joseleau *et al.* 2004; Khedari *et al.* 2004; John and Anandjiwala 2008). Furthermore the wax and oil substances in lignocellulosic fibers, which form as a thin film and prevent water from penetrating the board (Bekhta and Hiziroglu 2002), were removed effectively by NaOH. The higher WA and TS with the NaOH-treated particles may be partially also attributed to the lower lignin content, which is a hydrophobic constituent. Despite the reduction in hemicelluloses during alkali treatments, the rise in cellulose increased the content of the polysaccharides, which were hydrophilic components (Fengel and Wegener 1984). They may have contributed to the higher WA and TS as well.

Compared with boards produced with untreated particles, boards made with 1% NaOH-treated particles had higher MOE and MOR values. The MOE and MOR of particleboard with 3% NaOH-treated particles had no significant difference from the untreated boards. However, MOE and MOR were significantly lower for particleboards obtained after 5% NaOH treatment. Particleboards should have minimum MOE and MOR values of 1800 N/mm² and 10.5 N/mm², respectively, for interior fitment particleboards (including furniture) and general-purpose applications in dry conditions (TS-EN 312 2012). The results of the study demonstrated that only boards produced with 1% NaOH-treated particles met the minimum requirements. Boards made with untreated and 1% NaOH-treated particles met the minimum IB strength requirement of 0.28 N/mm² for general-purpose end use (TS-EN 312 2012). The decreases in MOE and MOR caused by soaking with 3% and 5% NaOH were probably due to decreases in the degree of crystallinity and increases in the amorphous regions during treatments (Joseleau *et al.* 2004; Khedari *et al.* 2004; John and Anandjiwala 2008). The results of thermal analysis indicated that there was considerable chemical degradation of the cell wall components when more than 1% NaOH treatment was applied carries to the drop in MOE and MOR values. Furthermore, lignin, wax, and oil were removed from the material surface during treatments with the higher concentration of NaOH, which led to insufficient adhesive coverage on the surface and weak strength performance (Bekhata and Hiziroglu 2002). Except for 1% NaOH, the treatments decreased the IB compared to the board produced with untreated particles; no significant difference in IB was detected between the boards produced with untreated and 1% NaOH treated particles. The decrease in IB strength can be explained by decreased wax, oil, and lignin components after alkali treatment. The wax and oil, which diminish the wettability of the lignocellulosic fiber, form a thin film on the surface of the fiber and influence the bonding properties of the board (Bekhata and Hiziroglu 2002; Lee *et al.* 2004). Lignin is a natural adhesive that generates better adhesion between the particles in the board, resulting in improved bonding and dimensional stability (Joseleau *et al.* 2004; Khedari *et al.* 2004). The inverse relationship between lignin content and NaOH concentration may have contributed to the poor adhesion among particles, consequently lowering internal bonding. Generally, the results of mechanical properties are consistent with observations by Mukherjee *et al.* (1993), who found that the use of more than 1% NaOH on lignocellulosic fibers weakens the fibers, resulting in poor mechanical properties. Although bending properties of the board are also related to the IB strength, 1% NaOH treatment slightly lowers the IB values and higher concentrations yielded significantly lower results.

CONCLUSIONS

1. Cotton stalks as renewable material have the potential to be used in the production of composite panels. To achieve good mechanical properties, the cotton stalks must be modified.
2. Treatments with NaOH solution of different concentrations (1%, 3%, and 5%) affected the chemical composition and thermal stability of cotton stalk particles. With higher concentration the extractive, hemicellulose, and lignin content of particles decreased, whereas the cellulose content increased. A reduction of thermal stability due to alkali treatments also indicated the chemical degradation of particles.

3. Compared to boards produced with untreated particles of cotton stalks, boards made with 3% and 5% NaOH-treated particles provided lower mechanical properties. The drop in mechanical properties was explained by a stronger chemical degradation of the cell wall components and deformation of the structure, as also manifested by a decrease in thermal stability.
4. Only boards produced with 1% NaOH-treated particles met the requirements of the TS EN 312 (2012) standard and can be utilized for general purposes in dry conditions. The study concluded that the boards made with cotton stalk particles without treatment can be used as insulating material in buildings because such materials are not subjected to mechanical stress. To obtain the mechanical properties (MOE, MOR, and IB) required for general use, cotton stalk particles must be modified by 1% NaOH treatment before the production of boards.

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