The Combined Effects of Alkali Treatment and Ammonium Bicarbonate Addition on Selected Properties of MDF Panels

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Effects of alkali treatment and ammonium bicarbonate were studied relative to selected properties of medium-density fiberboard (MDF) panels. Wood fibers were subjected to alkali treatment with sodium hydroxide or sodium perborate monohydrate (SPM). The MDF panels were produced from untreated and alkali treated fibers with or without ammonium bicarbonate (ABC). The modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding (IB), thickness swelling (TS), water absorption (WA), and limiting oxygen index (LOI) of the MDF were analyzed. The mechanical, physical, and fire resistance properties of MDF samples showed differences depending on the experimental parameters used. The lowest TS and WA values were found in the untreated-control group. The highest value for MOR and MOE was found for the group with the addition of 8% ABC. The IB values generally were higher than the untreated-control value. The highest value was determined in the group ABC8. Remarkably higher LOI values were achieved in the LOI test, especially for the groups with the application of both alkali treatment and ammonium bicarbonate.

Keywords: Medium density fiberboard; Alkali treatment; Ammonium bicarbonate; Mechanical properties; Physical properties; Fire resistance

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INTRODUCTION

The recently increasing demand for wood-based panel composites, especially medium density fiberboard (MDF) panels, has been remarkable. The MDF sector has shown a notable growth trend with the development of technology over time. In the recent years, among panel producers in Europe, Turkey has the largest MDF capacity (Çavdar 2020). For furniture applications, MDF panels are often preferred as a replacement for wood and other wood-based panels such as plywood, particleboard, *etc*. The MDF panels have other uses such as interior trim pieces, interior door skins, mouldings, *etc*. (Stark *et al*. 2010).

Most wood-based products contain various additives. These additives are chemicals that protect wood materials against fire and biological decay, *etc*. Sometimes untreated wooden material cannot be used because of its combustible nature. The flammability of wood materials is reduced by the application of fire retardant treatment (Sandberg 2016). Common fire retardants include compounds of boron, phosphorus, magnesium, halogen, and aluminum, among others (Çavdar 2020).

Carbonates, the salts of carbonic acid, are widely used as minerals in various industries such as glass, construction, agricultural, ceramics, and other chemical industries. As one of type of carbonate minerals, ammonium bicarbonate is used in textiles, plastics, and ceramics. Additionally, it is utilized in fire extinguishers and in fertilizers (Hinkamp 1998). Fire retardant effects of various carbonate compounds such as magnesium carbonate, potassium carbonate, and sodium bicarbonate have been investigated by many researchers (Morgan *et al*. 2007; Ozkaya *et al*. 2007; Bakirtzis *et al.* 2009; Grześkowiak 2012; Kawalerczyk *et al.* 2019).

As one of the widely used methods for modifying fiber properties, alkali treatment provides high quality fibers that have improved wettability and an increased number of reactive sites (Kalia *et al.* 2009). The application of alkali treatment to fiber increases the strength of interfacial bonding between thermosetting resins and fibers (Fiore *et al*. 2015). Moreover, the surface roughness of fibers increases with the alkali treatment and so, fibers make the effective contact with polymers during composite preparation (Song *et al.* 2017). The efficacy of alkali treatment changes depending on some parameters such as alkali concentration, liquor ratio, time, and temperature. The most used alkali chemical is sodium hydroxide (NaOH) for this treatment (Manna *et al.* 2017). Fibers are immersed in NaOH solution within a certain period of time for the alkali treatment method (Li *et al*. 2007). Many studies have been carried out to explore the effects of alkali treatment on the properties of various fibers and composite products (Ray *et al*. 2001; El-Shekeil *et al*. 2012; Ikhlef *et al*. 2012; Saiful *Islam et al*. 2012; Fiore *et al*. 2015; Then *et al*. 2015; Cai *et al.* 2016; Mukaida *et al.* 2016; Ma *et al.* 2017; Hashim *et al*. 2017; Ahmad *et al.* 2018; Jonoobi *et al.* 2018; Boran Torun *et al.* 2019).

Sodium perborate monohydrate is one of the available forms of sodium perborates. Along with other sodium perborates, it has commercial importance in various formulations of detergents, medicine, and cosmetics (Kocakuşak *et al.* 1997). Sodium perborate, which has alkali character is also used as bleaching agent in the pulping process (Peşman *et al.* 2013).

Although the main usage of SPM is not as a fire retardant chemical, it was selected for this study of MDF based on the results of another study by Ustaömer (2008). The cited study, involving sodium perborate tetrahydrate combined with other FR chemicals, showed a range of positives effect on the fire resistance of MDF panels (Ustaömer 2008). It is already known that boron compounds are utilized as fire retardants for various materials including wood, wood products, and polymers (Cavdar *et al*. 2015). SPM can be regarded as a promising candidate for such applications because it is both a boron compound and an alkali-based chemical.

The aims of this study were as follows:

(1) To compare the effectiveness of SPM as an alternative alkali chemical to NAOH, which is widely used for alkali treatment, in the fiber treatment.

(2) To investigate the combined effects of alkali treatment (NaOH and SPM), with ammonium bicarbonate as additive chemical on the selected properties of MDF.

EXPERIMENTAL

Materials

In this study, MDF panels were produced from commercial fibers (mixture of pine and beech fibers) supplied by Çamsan Ordu Inc. (Ordu, Turkey). Sodium hydroxide and sodium perborate monohydrate were used as alkali chemicals for fiber pre-treatments. Ammonium bicarbonate (ABC) was used as an additive chemical at two loading levels

(4% and 8%) based on oven-dry fiber weight. Urea formaldehyde (UF) was used as adhesive. Ammonium sulfate (1%) and wax-paraffin (1%) were added to UF adhesive prior to use. The experimental design of the panels is presented in Table 1.

Methods

Fiber pre-treatment and panel preparation

To begin with, 2% (w/v) solutions of alkali chemicals (NaOH and SPM) were prepared, and the fibers were treated with these solutions for 6 hours until they absorbed the solutions completely. After this process, they were washed for neutralization and dried to obtain 2 to 3% moisture content. In the MDF panel preparation, ABC was first added to urea formaldehyde (UF) adhesive (13% loading) together with a wax-paraffin and hardener. The mixture thus obtained was sprayed over the surface of untreated (control) and alkali pre-treated fibers. Following this process the panel mats were formed, and they were pressed using a laboratory hot press (Cemilusta Hot Press; Cemilusta Wood Working Machinery Ind. Inc., Istanbul, Turkey) with 180 °C for 8 min. The final thickness of panels was approximately 8 mm with density of 780 kg/m^3. Twenty seven panels were produced and conditioned in a climate room (20 ºC, relative humidity of 65%). These panels were then cut into the required dimensions for the related tests.

Table 1. Experimental Design of MDF Panels

Panel ID	Alkali Treatment of Wood Fiber	ABC Level (%)
Untreated/Control	Un-treated with alkali	0
ABC4	Un-treated with alkali	*4*
ABC8	Un-treated with alkali	8
NaOH	Treatment with sodium hydroxide(NaOH)	0
NaOH /ABC4	Treatment with sodium hydroxide(NaOH)	4
NaOH /ABC8	Treatment with sodium hydroxide(NaOH)	8
SPM	Treatment with sodium perborate monohydrate(SPM)	0
SPM/ABC4	Treatment with sodium perborate monohydrate(SPM)	4
SPM/ABC8	Treatment with sodium perborate monohydrate(SPM)	8

*ABC: Ammonium bicarbonate; NaOH: Sodium hydroxide; and SPM: Sodium perborate monohydrate.

Mechanical properties

The mechanical properties such as modulus of rupture (MOR), modulus of elasticity (MOE), and internal bonding (IB) of the MDF samples were performed using Zwick testing machine (Model Z50, Ulm, Germany). All tests were carried out according to related standards EN 310 (1993) and EN 319 (1993).

Physical properties

The physical properties such as water absorption and thickness swelling for 2 h and 24 h soakings of MDF panels were conducted according to the EN 317 (1993) standard.

Fire resistance: Limiting oxygen index (LOI) test

Determining the fire resistances of the MDF samples, the LOI test was carried out under oxygen and nitrogen gases. For this purpose, the MDF samples were tested in a limiting oxygen index chamber (Dynisco, Franklin, MA, USA) according to the related standard (ASTM D2863-13 2013), and minimum oxygen concentrations of these samples were determined as a percentage. Each test for these sample groups was performed with five replicates. The dimensions of test samples used were 15 mm x 100 mm x 8 mm.

Statistical evaluation

The obtained results from mechanical and physical tests in this study were analyzed using SPSS 21.0 statistical package (IBM Corp., Armonk, N.Y., USA). One-way analysis of variance (ANOVA) evaluation followed by a Duncan's post-hoc test method was used to determine statistical significant differences among the MDF groups.

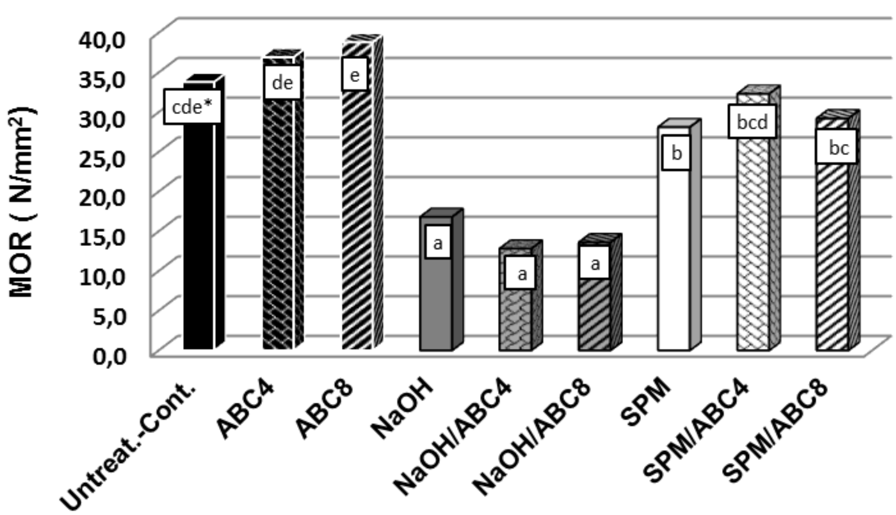
RESULTS AND DISCUSSION

Mechanical Properties

Modulus of rupture and modulus of elasticity

The MOR and MOE values of MDF samples are represented in Figs. 1 and 2. The two figures also show the homogeneity in groups for MOR and MOE values of samples.

As shown in Fig. 1, the MOR values of the samples varied depending on the addition level of ABC, alkali treatment, and type of alkali chemicals. The MOR values of MDF samples ranged from 12.8 to 38.8 N/mm^2. The MOR value for untreated-control samples was 33.8 N/mm^2. The highest MOR values in all groups were found for groups ABC8 and ABC4 to be 38.8 and 36.9 N/mm^2, respectively. The results showed that the increasing addition level of ABC positively affected the MOR values of the groups with ABC without alkali treatment.



* Letters represent each homogenous subset analyzed with Duncan test.

Fig. 1. Modulus of rupture (MOR) values of MDF samples

While moderate reductions were attained in the MOR values of the groups treated with SPM, marked reductions were seen in the MOR values of the groups treated with NaOH, compared to the control group. In particular, NaOH treatment and its combination with ABC led to more negative effect in the MOR values. Therefore, the lowest values were found in the groups NaOH, NaOH/ABC4, and NaOH/ABC8 compared to other groups. In the literature, while most researchers reported improvement of the mechanical properties of various composite materials with the alkali treatment (Then *et al.* 2015; Ahmad *et al.* 2018; Boran Torun *et al.* 2019), some researchers reported the opposite trend that alkali treatment showed less or more reduction in the mechanical properties depend on experimental parameters in their study (Jonoobi *et al.* 2018; Yasar and Icel 2016).

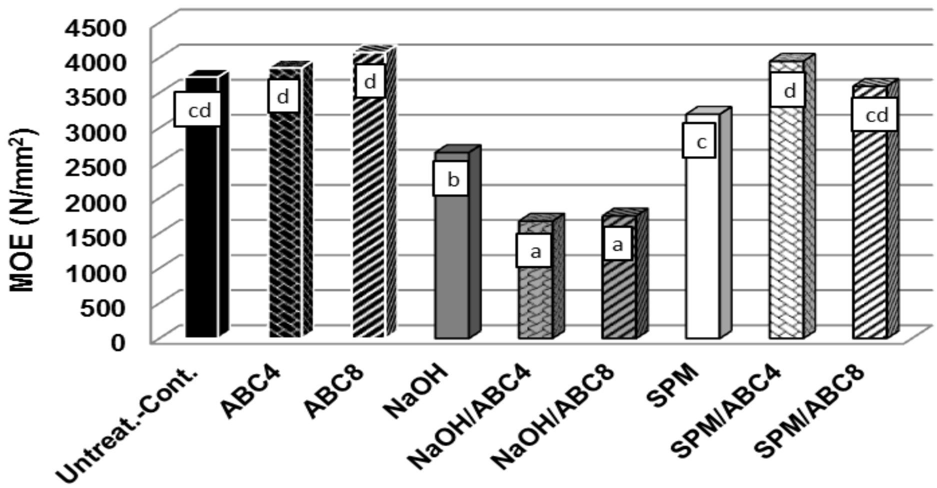


Fig. 2. Modulus of elasticity (MOE) values of MDF samples

As illustrated in Fig. 2, the MOE values of the MDF samples showed a similar trend with the MOR values. These values also changed depending on experimental parameters. The highest value in all groups was found in the group ABC8 to be 4080 N/mm^2. The lowest value was found for group NaOH/ABC4 to be 1670 N/mm^2. Similarly, the MOE values showed a decreasing trend with the application of alkali treatment. Especially, the distinct decreasing trend was seen in the groups with only NaOH treatment and its combinations with ABC (NaOH/ABC4, and NaOH/ABC8). However, SPM treatment and its combination with ABC did not cause significant decreases in MOE values and gave higher MOE values. Additionally, both MOR and MOE values showed statistically significant differences ($P \leq 0.05$), depending on the experimental parameters.

Internal bonding

Figure 3 shows the IB values of MDF samples. The figure also shows the homogeneity groups for IB values of MDF samples. As is evident from Fig. 3, these values showed variability depending on the alkali treatment, the type of alkali chemicals, and the addition level of ABC. The IB values of MDF samples ranged from 0.36 N/mm^2 to 0.97 N/mm^2. The IB value of the control group was 0.37 N/mm^2. Generally, the IB values were higher than the untreated-control value. The groups (ABC4, ABC8) without alkali treatment resulted in higher values relative to other groups. Increasing levels of ABC notably improved the IB values in these groups, and the highest value was obtained for group ABC8 as 0.97 N/mm^2. In addition, treatment with NaOH alone gave the second highest value (0.59 N/mm^2) compared to treatment of SPM.

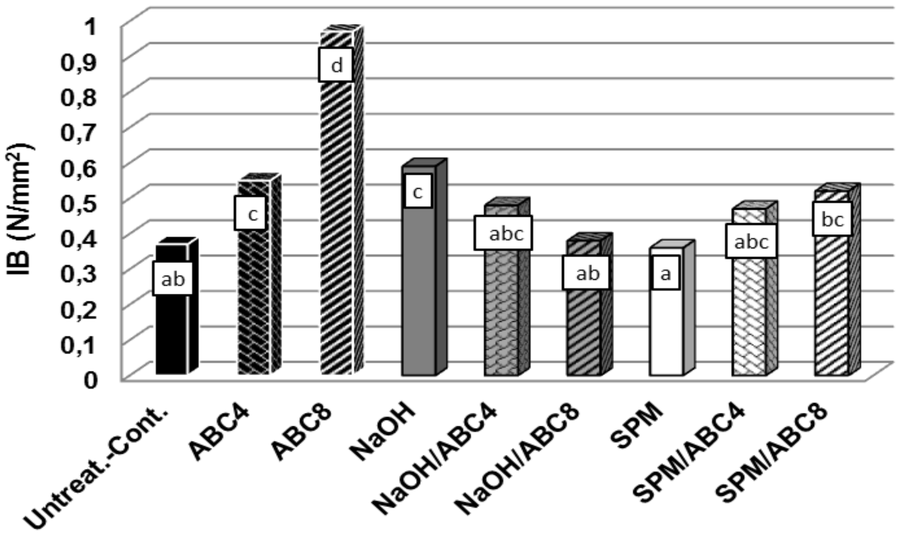


Fig. 3. Internal bonding (IB) values of MDF samples

The combination of NaOH treatment with ABC showed reducing trend in the IB values with the increasing level of ABC. On the other side, the combination of SPM treatment with ABC showed an increasing trend in values with the increasing level of ABC. These findings also suggest that there was more a positive effect in the IB values compared to the other mechanical properties (MOR and MOE). The IB values also showed statistically significant differences ($P \leq 0.05$) depending on the addition level of ABC, alkali treatment, and type of alkali chemicals.

Physical Properties

Thickness swelling and water absorption

The thickness swelling (TS) and water absorption (WA) values of MDF samples for 2 h and 24 h soaking periods are represented in Figs. 4 and 5. These two figures also show the homogeneity in the groups of MDF samples.

Figure 4 shows that the TS values for 2 h and 24 h periods varied depending on experimental parameters. These values of MDF samples ranged from 8.0% to 19.9% and 17.7% to 30.1%, respectively for 2 h and 24 h. While the lowest values of TS for 2 h and 24 h were found in their control groups, the highest values were determined in group SPM/ABC8. Furthermore, the lower results were found in the groups with only NaOH treatment and NaOH treatment/ABC compared to the groups with only SPM treatment and SPM treatment/ABC. Especially, the increases for the TS values (2 h) in the groups with only ABC were determined to be lower than other groups.

As shown in Fig. 5, a similar increasing trend was observed for the WA values. The WA values obtained after 2 h and 24 h ranged from 31.8% to 69.6% and 53.9% to 88.3%, respectively. When comparing all groups, the highest WA values for 2 h and 24 h were in the group SPM/ABC8, the lowest WA values were in untreated- control groups of MDF samples. Similar to the TS values, the increasing trend was found to be slightly higher for especially groups with only ABC compared to the WA values for groups with other combinations. The alkali treatment with NaOH and SPM did not show any positive effects in WA values, especially by its combined use with ABC. Furthermore, the increasing level of ABC caused an increase in the WA values. The statistical results represented that the both TS and WA values showed statistically significant differences (P≤ 0.05) depend on the addition level of ABC, alkali treatment, and type of alkali chemicals.

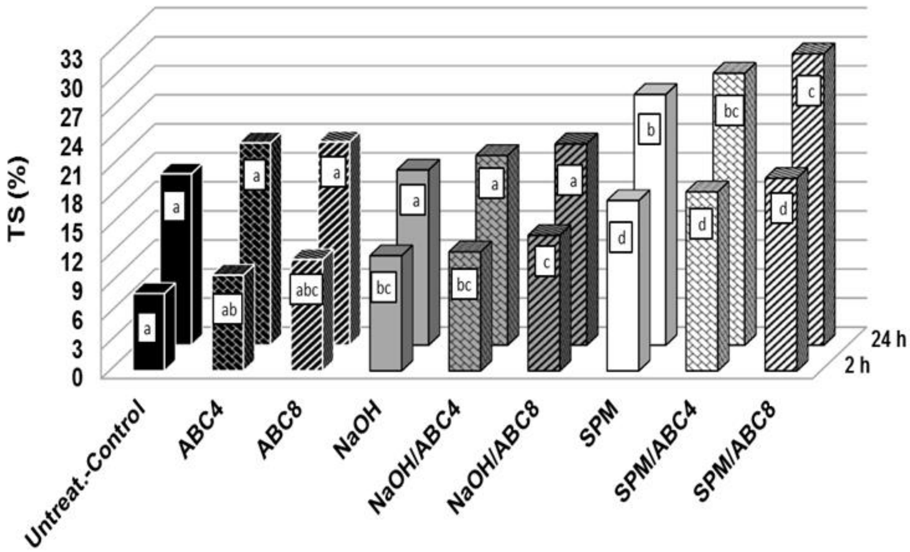


Fig. 4. Thickness swelling (TS) values of MDF samples for 2h and 24 h

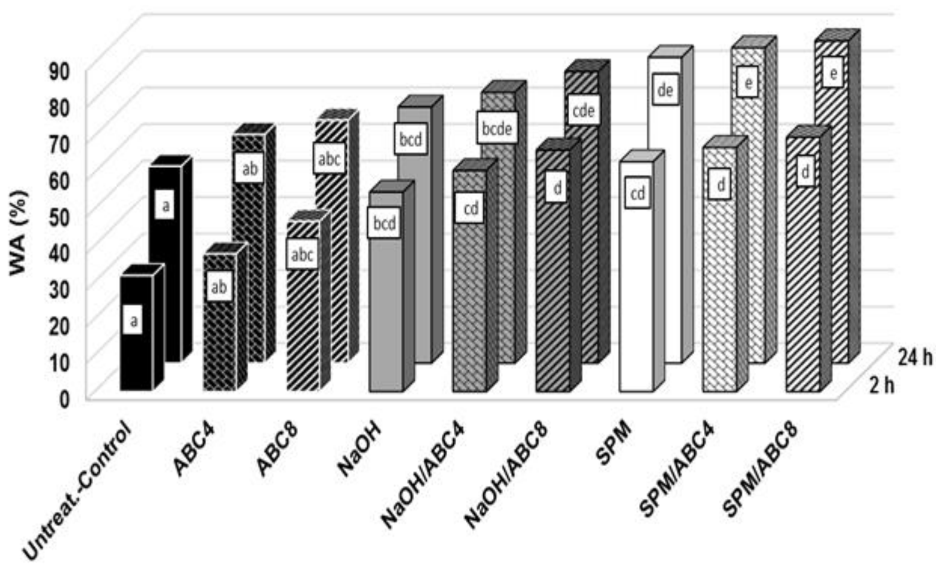


Fig. 5. Water absorption (WA) values of MDF samples for 2h and 24 h

A similarly increasing trend for TS and WA values could be due to the structural properties of NaOH, SPM, and ABC. The SPM treatment showed a more negative effect on the TS and WA values. It is reported that SPM is hygroscopic (Croud 1999). Therefore, its hygroscopic character could negatively affect the TS and WA values. It is also reported that alkali types and alkali concentrations affect the swelling reaction of fibers (Manna *et al.* 2017) and swelling degree (Hashim *et al.* 2012). In some other studies, a similar increasing trend of the TS and WA values of studied materials was obtained. Yasar and

Icel (2016) treated the cotton stalk particles with NaOH and produced particleboard from these treated particles. They found that alkali treatment increased TS and WA values of the particleboard samples. Boran Torun (2019) found that alkali treatment generally caused increases in the TS and WA values of composites made chestnut cupula and recycled polyethylene.

Fire Resistance

LOI evaluation

The LOI levels of MDF panel samples are represented in Table 2. The table also represents the results of observation for smoke generation during and after tests.

Based on the results in Table 2, it is evident that all the experimental parameters such as addition level of ABC, alkali treatment, and type of alkali chemical impacted the LOI levels of MDF samples. The LOI levels increased, depending on these parameters. The obtained LOI levels of the MDF samples ranged from 30.5 to above 48. While the lowest value was noted to be 30.5 in the untreated - control group, the highest values were noted to be above 48 in the groups NaOH/ABC8, SPM/ABC4, SPM/ABC8.

As can be seen in Table 2. following by control LOI level, the other lower LOI levels (31 and 32.3) were determined with the addition of ABC (4% and 8%) without alkali treatment.

Table 2. LOI Results for Samples

Panel ID	O_2 Concentration (%)	Smoke generation during the test	Smoke generation after the test
Untreated-Control	30.5	-	+
ABC4	31	-	+
ABC8	32.25	-	+
NaOH	41.5	-	+
NaOH /ABC4	47	-	-
NaOH /ABC8	*>48	-	-
SPM	47	-	-
SPM/ABC4	*>48	-	-
SPM/ABC8	*>48	-	-

*These values were higher than 48 and stated to be “ >48” in the Table 2, due to the samples were not burned even at max LOI concentration (48) in the fire test equipment.

These lower values express the lower fire resistance compared to other groups having the higher LOI levels. However, the results indicated the treatment with both of alkali chemicals (NaOH and SPM) provided notable increases in LOI levels and improved the fire resistance of samples. Especially, SPM treatment resulted with higher LOI value compared to NaOH treatment. These LOI values were found to be 41.5 and 47, respectively, for groups NaOH, and SPM. More remarkably, distinct increases in the LOI levels were achieved with the both alkali treatment and its combination of ABC. Furthermore, group SPM/ABC4 resulted in slightly higher LOI values compared to the group NaOH/ABC4. In particular, the obtained improvement in the LOI levels with SPM could be attributed to the boron content in the structure of SPM. As reported, many boron compounds are effective fire retardants for cellulosic materials (Baysal *et al*. 2007). Additionally, the fire retardant effect of another sodium perborate compound (sodium perborate tetrahydrate) on the MDF samples was determined in a study by Ustaömer (2008).

According to the Table 2, it is evident that smoke was not observed in all groups during the test. However, smoke was observed in the groups (untreated-control, ABC4, ABC8, NaOH) after completion of the tests. Additionally, it was observed that the burning rates of the groups NaOH/ABC4, NaOH/ABC8, SPM, SPM/ABC4, and SPM/ABC8 were low compared to those of the other groups.

CONCLUSIONS

1. Alkali treatment, ammonium bicarbonate (ABC) addition, and the combination of alkali treatment with ABC showed different efficiencies on the selected properties of the medium-density fiberboard (MDF) panels, depending on experimental parameters.
2. The experimental parameters negatively affected the thickness swelling (TS) and water absorption (WA) values. The modulus of rupture (MOR) and modulus of elasticity (MOE) values were generally reduced with the combinations of alkali treatment and ABC. However, the experimental parameters showed more positive effect on the IB values compared to MOR and MOE values. The most IB values were higher than the control value. Especially, an obvious improvement in the IB values was obtained with the addition of ABC.
3. The high flame retardant performances by improving limiting oxygen index (LOI) levels were achieved with the experimental parameters. Remarkably high LOI levels were obtained with the alkali treatment and combination of alkali treatment with ABC. The highest LOI levels were obtained with the groups NaOH/ABC8, SPM/ABC4, SPM/ABC8. Especially, the SPM treatment resulted more higher LOI value compared to NaOH treatment.
4. Alternatively, SPM could be utilized as one of the alkali based chemicals for treatment of fiber.

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