



Gypsum-Based Boards Made from Mixtures of Waste Cellulosic Sources: Part 2. Chemical and Technological Properties

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Abstract

The highest total color difference values (ΔE) were found in the panels produced with the similar proportions of waste paper and OCC into gypsum (A4: 6.09; B4: 5.79). These boards have also show highest CIE whiteness reduction (A4: -33.19; B4: -28.44). With the help of FTIR, some chemical groups are modified to some extent but similar functional groups were observed in surface of boards. For A- and C-type boards, similar TGA degradation trends (spectra) were observed while the B-type panels show markedly higher mass loss, especially at initial temperature stage (100-120 °C). This is probably because of the presence of some non-cellulosic materials (i.e. starch, silica, etc.) and the high rate of lignin in sheet structure of OCC material. The flame propagation characteristic of the surfaces of all test boards shows that the flame did not reach the threshold limit of 150 mm. These indicates, even if the source of fire is not removed, flame did not reach the threshold limit that boards that could be classified as non-flammable material class according to TS EN-ISO 11925-2 standard.

The heat insulation properties of the test sample were found to be lowered with addition of lignocellulosic material to the gypsum structure. It was seen that waste paper ratio in gypsum structure higher than 10% (A3 to A6 boards) show better insulation properties than counterpart OCC (B-types) and secondary fiber based (C-types) gypsum boards. In general, reduction heat transfer in average values of samples attracted attention as compared with control specimens. Interestingly, the boards are indicated best insulation properties as mentioned above, show higher mass loss too. The highest mass loss for A-type boards found to be 3.52% (A6), for B-type boards 3.46% (B6) and for C-type boards 3.28% (C8).

Keywords: Waste paper, FTIR, TGA, gypsum board, heat insulation.

Atık Selülozik Karışımı Kaynaklardan Üretilen Alçı Esaslı Levhalar: 2. Bölüm. Kimyasal ve Teknolojik Özellikler

Öz

En yüksek toplam renk farkı (ΔE) benzer üretim şartlarında atık kağıt ve eski oluklu mukavvalardan üretilmiş levhalarda (A4: 6.09; B4: 5.79) gözlemlenmiştir. Bu levhalara aynı zamanda en düşük CIE whiteness değerleri göstermiştir (A4: -33.19; B4: -28.44). FTIR tekniği yardımıyla levhaların yüzeylerindeki kimyasal gruplar incelenmiştir. A ve C tipi levhaların diagramları benzerlik göstermekle birlikte B tipi levhaların TGA diyagramları oldukça farklı olduğu anlaşılmıştır. Özellikle başlangıç sıcaklık değerlerinde (100-120 °C) B tipi levhaların ağırlık kaybı diğer A ve C tipi levhadan daha yüksektir. Bu durum muhtemelern atık oluklu mukavva (B tipi levha)

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yapısında selülozik liflerden ayrı olarak daha yüksek oranda nişasta, silika, lignin bulunmasından ileri gelmesidir. Deneme levhalarının yüzeyine uygulanan alevin, tüm levhalar için kritik eşik değer olan 150 mm seviyesine gelmediği gözlemlenmiştir. Alev uzaklaştırıldıktan sonra dahi, yüzeydeki yayılma kritik sınır değerine ulaşmamıştır. Buradan üretilen deneme levhalarının TS EN-ISO 11925-2 standardına göre yanma malzeme sınıfına dahil edilebilir.

Deneme levhalarının ısı yalıtım özellikleri incelendiğinde, alçı yapısına lignoselüloik hammaddenin eklenmesinin olumlu katkı yaptığı anlaşılmıştır. Levha yapısında %10 dan daha yüksek atık kağıt eklenmesinin (A3-A6 levhalar) aynı üretim şartlarındaki atık oluklu mukavva (B tipi) ve sekonder lif esaslı (C tipi) deneme levhalarından daha yüksek ısı yalıtımı sağladığı anlaşılmıştır. Genel olarak ısı transfer ortalama değerleri kontrol örneğine göre daha iyi olduğu görülmüştür. İlginç olarak en yüksek ısı izolasyonu sağlayan örnek aynı zamanda en yüksek kütle kaybı göstermiştir. En yüksek kütle kaybı A tipi levhalarda %3,52 (A6), B tipi levhalarda 3.46% (B6) ve C tipi levhalarda 3.28% (C8) olarak ölçülmüştür.

Anahtar Kelimeler: Atık kağıt, FTIR, TGA, Alçı levha, Isı yalıtımı

1. Introduction

The use of alternative cellulosic sources such as; agricultural and forest residues, secondary wood materials (wastes) or fast-growing annual plants have become important issue considering convert these low value materials in value added products. In this regard, there is plenty of literature finding available for industrial applications of that raw materials processing into composite panels or paper-based products (Atchison, 1993; Ndazi et al., 2006; Rials and Wolcott 1997; Youngquist et al., and 1997). However, the products obtained from these alternative sources may have some properties which are different from the characteristics of the conventional wood-based panels. They could, for example, exhibit some degree of resistance to fire, as a better sound and heat insulation material, and could be used as a building material in with inexpensively and effectively (Arslan and Sahin 2016, Sahin et al. 2018; Kaya and Sahin 2018).

Waste paper is a general name given to all kinds of paper, cardboard and paperboard that are completed its use. Due to technological developments and industrial improvements, using waste paper has become important topic for economic and environmental factors (Kaya, 2015). Especially in the last 50-60 years, post-consumer waste paper products have been becomes an important source for the paper industry. Moreover, one of the most important benefits of waste paper recovery is the production of cellulose raw material which is more economical production with the help of less harmful processes in the environment for paper industry (Baipai 2013 and 2018).

However, during the recycling process, some important changes occur in the structure of cellulose. Thereby, cellulose is inherited and the water swelling property is irreversibly reduced. Because intensive hydrogen bonds are formed in crystalline structure, resulting the markedly modification of elasticity and plasticization properties of the fibers (Atalla, 1992; Brancato, 2008; Hubbe et al., 2007). As a result of those changes, the flexibility and plasticity of the fibers is reduced and the hardness/brittleness increases and generally known as '*hornification*' (Kaya, 2015; Minor, 1994). Moreover, when once drying of the cellulosic fibers, re-treatment of the microfibrils with water again not effective as virgin fibers. In addition, without any sorting or grading of post-consumer waste paper products, the recycling process may result in loss of quality of recovered cellulose fibers and process efficiency (Thompson, 1992; Wistara and Young 1999).

The use of waste papers directly or as recycled fibers (secondary fibers) for composite panel industry might not require high-cost papermaking procedure (i.e. re-pulping, energy and capital intensive multi processes) rather than direct utilization as a raw material source for composite panel industry.

However, reduction of forest assets and environmental pressures leads increased sensitivity of people to the environment that make the use of waste paper more attractive. In first part of this study, it was investigate the physical and mechanical strength properties of gypsum-based composite panels that were made from the mixtures of three different cellulosic raw material sources (waste paper/OCC/secondary fiber) at various proportions with gypsum. In the second part of that study, the determination of some chemical and technological properties of those panels have been investigated and given in below.

2. Materials and Methods

2.1. Materials

The detailed description of cellulosic raw materials and gypsum with their specifications, with gypsum-based experimental panel manufacturing procedures could be found first part of this study. However, the board's code numbers and cellulosic additive of waste paper (A); OCC (B) and secondary fiber (C) with gypsum ratio at various proportions have also given in first part of this study.

2.1. Methods

A single flame combustion tests were carried out for determining ignitability of experimental panels. In this regard, a flame combustion test system was conducted according to TS EN-ISO 11925-2. A single-source small flame applied at 45° slope and distance of 20 mm from the middle of samples was initiated. The total test duration was 60s. At the end of test, whether the specimen ignition occurs or not at the flame spreads in the vertical direction more than 150 mm above (the flame application point). A visual observation of the sample was made and results recorded as positive or negative.

A Shimadzu (IR Prestige-21 series) FTIR spectrophotometer was used to evaluate the chemical groups present in the boards was made from selected samples. Thermogravimetric Analysis (TGA) is carried out using a Perkin Elmer SII instrument in order to measure changes in properties of experimental panels as a function of increasing temperature (with constant heating rate).

The natural weathering tests were conducted on 50x50x10 mm samples from experimental panels were exposed to weathering process for two months, The surface color changes were determined with X-Rite SP68 Spectrophotometer using CIE L*,a*,b* standard (1976).

3. Results and Discussions

The surface color parameters (Δ ; differences) according to the CIE L*a*b* (1976) standard, which were measured before and after natural weathering process under external atmospheric conditions for 60 days, are given in Table 1. It is very difficult to explain all color parameters and relationships to each other. But it is the most commonly used method in the literature to explain total color difference (ΔE) of samples (Sahin and Arslan 2011; Sahin et al. 2011). It was realized that the highest and lowest total color differences were calculated in the experimental panels produced with the same proportions of waste paper-OCC into gypsum matrix. When increase waste paper and OCC into panel structure, an increase trend was found for both (A&B) type boards. The highest color differences of 6.09 and 5.79 for A4 and B4 boards found, respectively. As expected these experimental panels have also show highest CIE whiteness reduction (A4: -33.19; B4:-28.44). But for C-types, the highest total color difference of 4.37 and CIE whiteness reduction of -20.92 were observed in C7 panel.

Table 1. Color properties of experimental panels after natural weathering process (60 days)

Board code	ΔL	Δa	Δb	ΔE	CIE Whiteness
Waste paper/gypsum-based experimental panels					
A1	-1.2	-0.65	-2.18	2.57	7.85
A2	-0.52	-0.12	0.29	0.61	-2.73
A3	-3.17	0.56	3.65	4.87	-2.81
A4	-2.44	0.81	5.52	6.09	-33,19
A5	-3.39	0.91	4.12	5.41	-28.83
A6	-0.67	0.44	2.97	3.08	-16.4
OCC/gypsum-based experimental panels					
B1	-1.2	-0.65	-2.18	2.57	7.85
B2	0.59	-0.22	-0.23	0.67	2.51
B3	1.5	-0.76	2.98	3.42	18.42
B4	-5.48	-0.22	1.87	5.79	-28.44
B5	-4.3	-0.11	3.68	5.66	-22.09
B6	-3.57	-0.63	3.5	5.04	-26.06
Secondary fiber/gypsum-based experimental panels					
C1	-1.2	-0.65	-2.18	2.57	7.85
C2	0.14	-0.59	-1.18	1.33	6.23
C3	-1.62	-0.59	1.27	2.14	-10.29
C4	-3.86	-0.82	-0.82	4.03	-3.87
C5	-1.82	-0.42	0.14	1.87	-3.15
C6	-3.14	-0.39	1.26	3.41	-13.58
C7	-3.69	-0.64	2.25	4.37	-20.92
C8	-0.25	-1.05	-0.17	1.09	0.33

The combined effects of panel density and cellulosic additive level on surface total color difference properties is shown in Figure 1. As explained above, all three cellulosic additives markedly effects on surface color changes some level. However, it has clearly realized that waste paper and OCC addition with increasing panel density markedly effects on color reduction (increasing total color difference) (Fig. 1A& B) while less effect on secondary fiber-gypsum based experimental panels (Fig.1C).

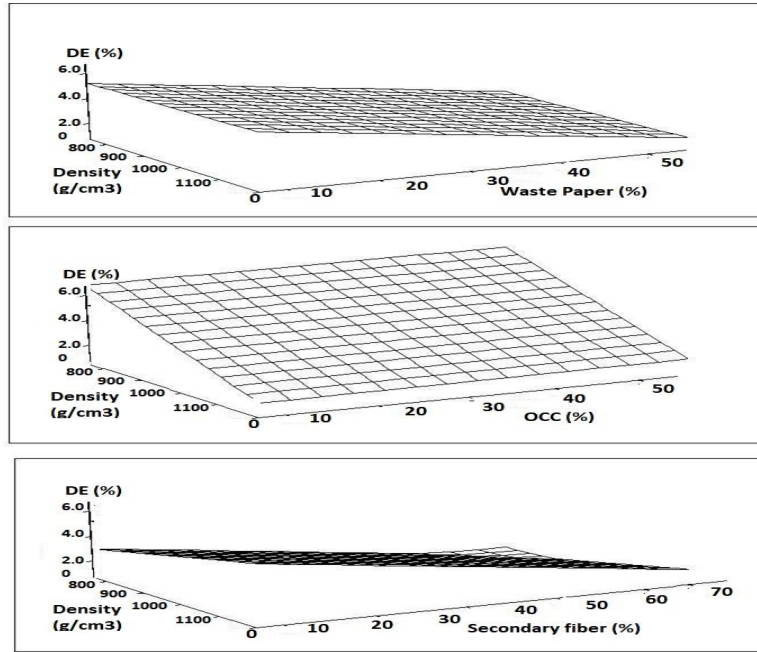


Figure 1. Cellulosic additive and board's density effects on surface color properties of experimental panels (A: A-types; B: B-types; C: C- types)

The comparative Fourier Transformation Infrared Spectroscopy (FTIR) spectra presented in Figure 2. As it is known, FTIR technique is a widely used method in determining the solid material surface functional groups. In this study, it was aimed to determine the functional groups on the surfaces of the experimental panels produced by adding different proportions of cellulosic raw materials to the gypsum structure. Generally, peaks in the range of 1030-1060 cm^{-1} and 1145-1162 cm^{-1} are assigned to C-O and C-O-C tension in polysaccharides (cellulose & hemicellulose). However, 1500-1610 cm^{-1} are C=O and COO-symmetric tension vibrations in aromatic rings that considered to a characteristic peak for lignin components (Pandey, 2005; Demir, 2019). It was proposed that the peaks in the range of 1360-1380 cm^{-1} were C-H degradation in polysaccharides and the change in that peak indicator for the change in the hydrophilic property of cellulosic material (Can and Sivrikaya 2017). Similarly, C-OH bands in the range of 2900 through 3100s cm^{-1} indicator of -OH groups in cellulosic materials that some changes (decrease or increase) in that region were shown property changes. In our study, the structure of lignin and hemicellulose looks like modified some degree in water-gypsum mixture environment. Those in a weak alkaline condition could be affects some cell wall compenents especially polysacahardies and oligomers. While the gypsum is freezing, its volume is narrowed to around 7% that called *calcination* to evaporate the water of the gypsum (Demir, 2019), in the open air, in a weak alkalen conditions could be affects some cell wall compenents. Although it is difficult to explain all the chemical changes in the board matrix structure with the help of FTIR but similar functional groups were observed in FTIR spectra as seen in Figure 2.

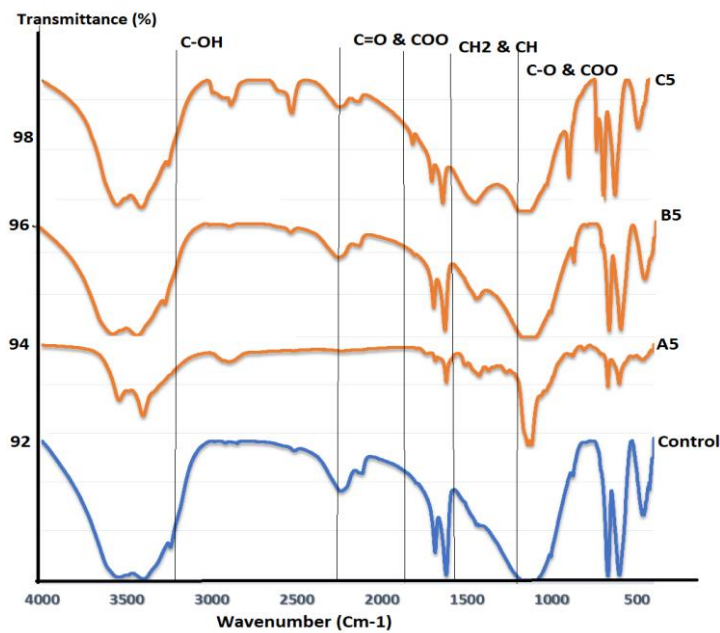


Figure 2. Fourier Transformation Infrared Spectroscopy (FTIR) of experimental panels

However, one of the most commonly used approaches is TGA (Thermal Gravimetric Analyzer) method for determining temperature-time degradations of the components of the composite materials (Kaya, 2015). In this regard, temperature-dependent TGA mass-change micrographs at the temperature range of 25-900 °C is shown in Figure 3.

The degradation values obtained in the TGA graph are explained in four different temperature zones. Zone 1 is assigned to be a heating zone and approximately 7-10% mass loss is caused by drying due to the moisture inside the material up to 100-110 °C. Zone 2 is considered to be the water is completely removed from the cell wall in the cellulosic fibers between 110-250 °C. Above this temperature, the cellulosic material is considered to degrade significantly. Zone 3 is assigned to completely break down of cellulosic substances as rapidly in the 300-360 °C range and the mass loss occurs approximately 75-80%. At these temperature levels, the constituents have become solid to gas state rapidly and cell structures significantly degraded. Zone 4 is considered to some non-organic residues such as; ash or char and the mass loss of the sample reached to above 90% at the temperature range of 400-900 °C.

When Figure 3 carefully reviewed, it was realized that both A5 and C5 (Fig. 3 A&C) experimental panels looks like similar degradation trends. However, the graph of B5 (Fig. 3B) show a very high mass loss, especially at initial temperature ranges (100-120 °C). It is presumed that this is probably due to the presence of some non-cellulosic materials in the structure of OCC and the high rate of lignin in sheet structure. Because both A- and C- type panels produced from fully bleached kraft paper particles (A) and recovered fibers (C) while OCC typically contains higher lignin in sheet structure with some non-cellulosic additives such as starch, sodium silicate and hemicellulose substances that easily degraded with temperature rather than cellulose (Fengel and Wegener, 1984). Moreover, corrugated cardboards consist of at least three sheets (one corrugated layer) with two surface plain papers. Various types of papers could be useful at inside or outer surfaces (liner papers). Some example of these papers as; kraft liner, test liner, NSSC and fluting etc. (Biermann, 1993; Smook, 1994). Hence, corrugated boards have different properties and characteristics compare to office papers. Due to fully bleached kraft waste paper particles (A) and recycled office paper fiber (secondary fibers) (C) utilized in this study, they are typically containing more than %90 cellulose in their sheet structure. It is well known that cellulose is more resistant temperature degradation than other lignin, hemicellulose and starch (Biermann, 1993; fengel and Wegener, 1984).

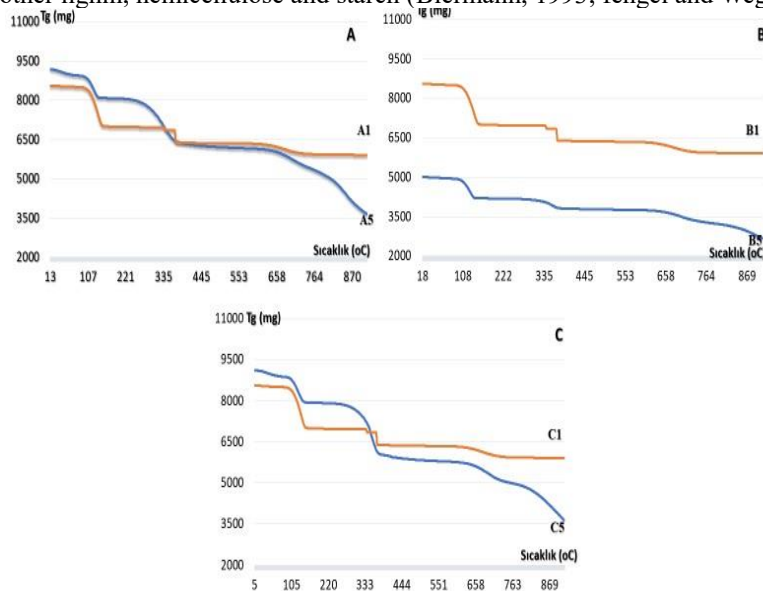


Figure 3. TGA spectra of gypsum based experimental panels (A: Waste paper-gypsum; B: OCC-gypsum; C: secondary fiber-gypsum).

TGA analysis at four different temperature degradation and mass loss properties are given in Table 2. It was divided four different temperature level as; starting temperature (Tb) 122 °C, first maximum temperature (Tm1) 406 °C, second maximum temperature (Tm2) 650 °C and last final temperature (Ts) 692 °C. When the Table 2 carefully overviewed, the lowest mass loss at all temperature levels was in the boards produced from the OCC-gypsum mixture. It has also realized that the initial degradation temperature (Tb) for all boards close to each other (115-122 °C) while there is very different last final temperature (Ts) levels (692-891 °C) were observed for experimental boards.

Table 2. The TGA analyses of experiemntal panels

Board code	Tb (°C)	Mass loss (%)	Tm ₁ (°C)	Mass loss (%)	Tm ₂ (°C)	Mass loss (%)	Ts (°C)	Mass loss (%)
A1-B1-C1	122	7.0	406	8.0	-	-	692	12.7
A5	119	5.0	329	11.0	700	36	850	45
B5	115	4.75	348	4.84	690	24	883	31
C5	117	4.0	347	12	676	35	891	49

According to the TS EN-ISO 11925-2 standard, the flame propagation characteristic of the surfaces of the experimental panels as a result of combustion tests with single flame source is shown in Figure 4. It was observed that the burning pattern on the surface of all test boards produced by adding three different cellulosic raw material sources to the gypsum structure did not reach the threshold limit of 150 mm that specified in the standard value. The test boards showing the closest behavior to this limit were observed in secondary fiber-added experimental panels (C6, C7 and C8). This is expected because gypsum is considered to be A class material as a non-flammable class. In other words, these non-flammable materials do not carry out any flame, even if the source of fire is not removed. The findings and the appearance in Figure 4 support this hypothesis. But some flammable materials in gypsum may result some level flammable behavior as realized in this study. But the test boards still a non-flammable class (A) while flame did not reach the threshold limit of 150 mm.

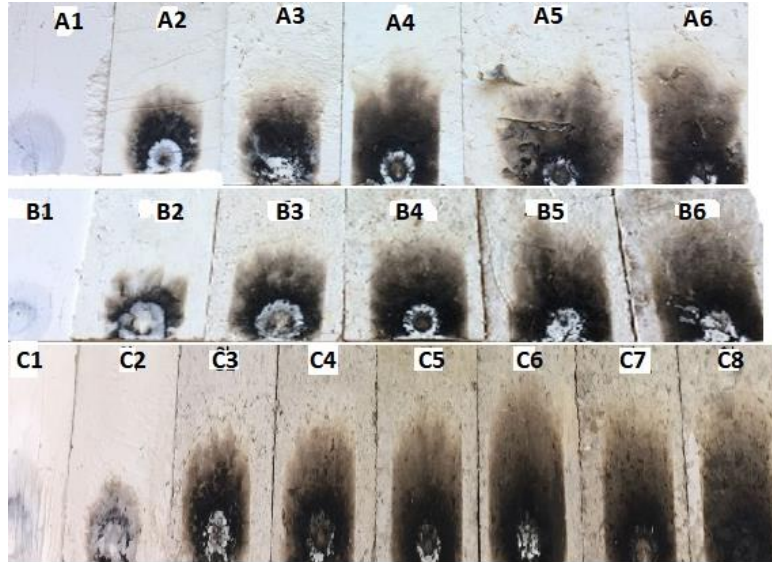


Figure 4. The flame propagation characteristic of experiemental panels.

For determining thermal insulation properties, the temperature values passing to the back surfaces were measured in accordance with DIN 4102 standard for 30 seconds intervals and for a total of 300 seconds. For this test, measured values are shown as a thermal insulation value with a total mass loss in Table 3. Generally, in all panel types and production conditions (A1 to A6; B1 to B6; C1 to C8), it has been observed that the addition of lignocellulosic material to the gypsum structure has a negative effect on the heat transfer properties of the test boards (improving thermal insulation properties). The highest insulation properties (lowest heat transfer rate) in all three types of boards are found to be 68.2 °C for A6, 76.2 °C for B6, 79.4 °C for C8 boards, respectively. It is realized that waste paper ratio higher than 10% (A3 to A6) show better insulation properties than both counterpart OCC (B- types) and secondary fiber based (C-types) gypsum experimental panels.

However, mass loss values (burning behavior characteristics) which occurred as a result of the process with the single flame source of the boards for 5 minutes (300 seconds) also presented in Table 3. Generally, the increase in the ratio of the lignocellulosic additive into gypsum effects on mass loss increasing. The highest mass loss on these three different type boards were measured as 3.52% for A6 board, 3.46% for B6 board and 3.28% for C8 board. Interestingly these boards are also indicated best insulation properties as mentioned above. With having this measurement, it could be concluded that the addition of waste paper, OCC and secondary fibers into gypsum matrices have improved heat insulation properties of some level. However, values for mass lost particularly high mainly due to increasing cellulosic raw material content. This is probably gypsum that could be interaction waste paper and OCC particles to form some mechanical bonding, which resulted in easy removing of gypsum particles with those esay burned cellulosic materials compared with only gypsum based panels (control). Thereby, cellulose might be easily burned while it has effects on gypsum lost during degradation of those easily burned materials. The result found in above support this hypthoses.

After the determining thermal insulation properties with DIN 4102 standart a total of 300 seconds, the surface apperance of experimetal test boards are presented in Figure 5. It can be seen that although some degradation and char formed, all these panels look like acceptable heat insulation properties with some level mass loss.

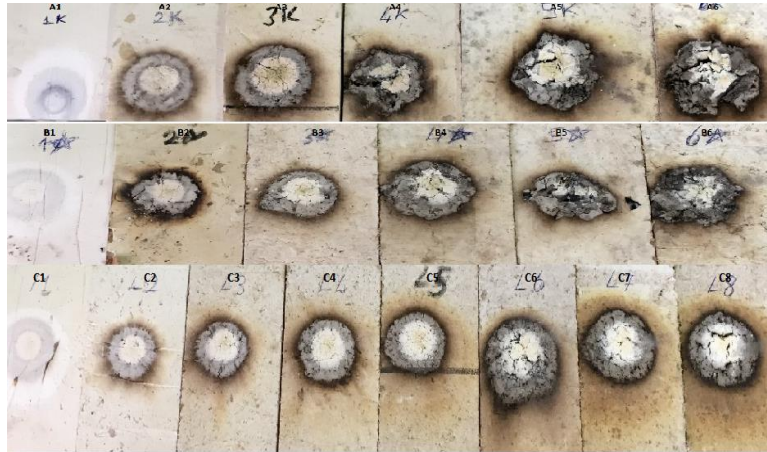


Figure 4. Combustion behavior of test boards for total of 300 seconds.

Table 3. Heat insulation and mass loss properties of test boards

Time (Second)	0	60	120	180	240	300	Mass loss (%)
Waste paper/gypsum-based experimental panels							
A1	16.9	64.9	93.0	124.9	133.7	135.4	1.64
A2	14.4	51.9	78.3	100.9	105.2	148.3	1.85
A3	12.2	20.2	51.7	60.8	67.2	68.7	2.35
A4	12.6	22.2	61.0	73.0	78.2	78.3	2.37
A5	13.6	16.9	41.4	66.6	72.7	74.6	2.48
A6	10.4	15.6	30.4	57.8	67.6	68.2	3.52
OCC/gypsum-based experimental panels							
B1	18.1	93.3	127.1	137.9	160.8	161.0	1.51
B2	15.7	51.0	83.8	96.1	100.5	125.1	1.35
B3	14.1	24.6	61.5	66.7	75.9	90.8	1.49
B4	12.4	30.4	62.7	72.4	72.8	80.4	2.75
B5	11.7	26.4	58.3	66.4	75.9	98.0	2.9
B6	9.4	26.1	39.6	73.2	85.1	76.2	3.46
Secondary fiber/gypsum-based experimental panels							
C1	17.2	52.8	78.2	86.6	112.3	123.8	1.32
C2	15.5	22.3	50.4	71.2	78.6	84.9	1.61
C3	15.1	26.5	48.7	73.8	82.5	104.0	1.56
C4	13.9	24.5	69.7	86.7	89.9	107.4	2.2
C5	13.5	30.8	60.7	80.6	86.9	98.1	2.15
C6	28.3	45.3	76.4	84.2	86.6	87.6	3.09
C7	13.2	17.0	38.1	61.8	67.9	84.7	3.13
C8	12.2	17.7	53.6	70.6	79.9	79.4	3.28

Figure 5 show correlation effects of panel density and cellulosic additive level on mass loss properties of experimental panels. As mentioned above, cellulosic additives effects on panels insulation properties positively but mass loss is also increased. However, it could be seen that in all conditions and additional level of these raw materials, increasing panel density and raw material sources increasing effects on mass loss (Fig 5. A,B and C). This trend looks like more-less similar for all three type experimental boards.

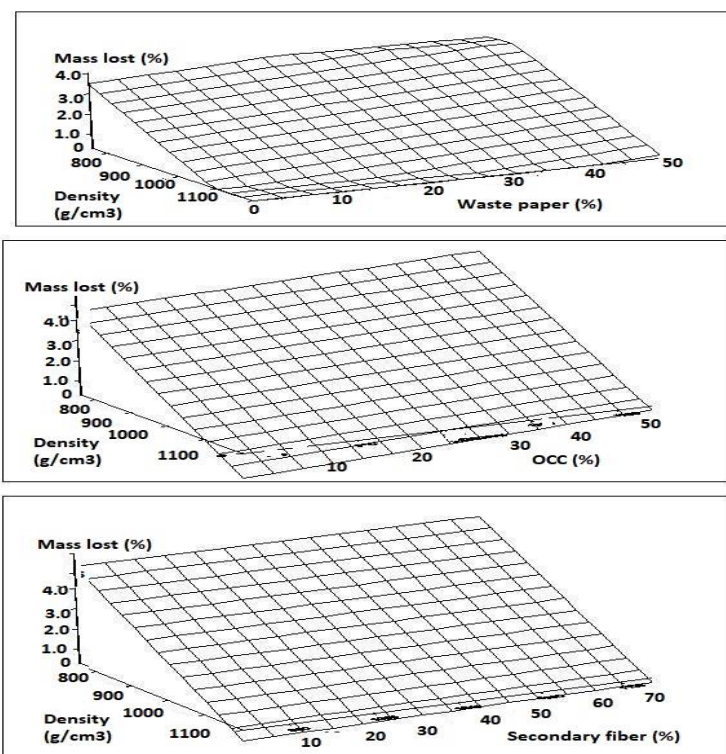


Figure 5. Cellulosic additive and board's density effects on mass lost of test boards (A: A-types; B: B-types; C: C- types)

4. Conclusions

The experimental result show that the production of gypsum-based panels technically feasible. However, there are many reasons for manufacturing gypsum-based composite materials that are stronger, lighter, or less expensive when compared to traditional materials. However, since increasing demand on wood material, this is leading to the need to investigate sustainable materials to replace existing ones. This is possible to use low value lignocellulosic materials in gypsum structure. Hence, paper wastes may exhibit some advantages, such as availability, low cost, non-hazardous nature, and low density. Although the waste paper-gypsum compatibility looks like low, but addition of some chemical substances could be improved for that to provide adequate bonding capacity to gypsum-based composites.

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