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3 **Properties of Gypsum Based Boards Made**
4 **From Mixtures of Wood and Rice straw**

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8 **ABSTRACT**
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Aims: It was investigated to produce gypsum-based experimental composite panels with red pine wood/rice straw particles in the mixture up to 40/60 (w/w) in gypsum-water mixture. In this case, the fillers (additives) could be observed more closely.

Methodology: The red pine wood chips and the rice straw (stalks) have been turned into suitable particle dimensions. The experimental boards were cut to determine the IB (Internal bond), MOE–MOR (Modulus of Elasticity and Rupture), and TS (thickness swelling after 24 hours immersion in water). A standard flame combustion test system was conducted according to TS EN-ISO 11925-2. For surface chemical analyses, FTIR was used to evaluate the chemical groups present in the board surface. The thermogravimetric analysis (TGA) was conducted for measuring changes in boards as a function of increasing temperature. The natural weathering tests were conducted that were exposed to outdoor for two months then color and surface hardness tests conducted for determining property changes.

Results: The rice straw had negative impact on thickness swelling (TS) properties of boards in water. The highest TS value of 47.66% was observed in the board that produced from 60/40 (w/w), wood/rice straw mixture (DE4). However, the addition of rice straw to the wood/gypsum mixture has a lowering effect on the internal bond (IB) and bending strength (MOR) properties of experimental boards some level. The maximum IB strength of 0.06 N/mm² and MOR of 2.77 N/mm² found control sample (DE0). However, the highest MOE value of 553 N/mm² was calculated on the DE4 board, which was produced by adding 40% rice straw to the wood/gypsum mixture. In similar trend was also realized for surface hardness properties that the highest hardness (Shore D) value of 44 was observed on the control sample (no rice straw added) while the lowest value of 22 found for DE5 board produced from the mixture of 50/50 (w/w), wood/rice straw. For surface optical properties, the highest total color difference (DE: 8.15), Whiteness reduction (CIE whiteness: -35.48) and Yellowness increasing (E313: 7.03) were found to be control sample (DE0). Some chemical groups are modified to some extent but all relevant chemical groups observed on the FTIR spectra. The addition of rice straw and wood particles to the gypsum structure has a positive effect on the heat transfer properties. The highest insulation value (39.1 °C after 300 second heat application) while the lowest mass loss value of 2.01% was measured in same board (DE6) that produced with the rice straw ratio of 60% in wood-gypsum structure.

Conclusion: It is clear that the addition of rice straw to the wood/gypsum mixture adversely affected the strength properties negatively, but with using some longer fibers may improve the strengths of panels. However rice straw in wood chip/gypsum mixture helps to improve heat resistance (insulation) properties some level. Moreover, the addition of rice straw to wood/gypsum mixture effects on extending hardening time. this is probably gypsum-rice straw interaction that could not effectively wet and penetrate the rice straw surface due to the hydrophobic wax and inorganic silica on the outer surface of rice straw.

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Keywords: Rice straw, gypsum board, red pine, strength properties, heat insulation

1. INTRODUCTION

Since human being, wood has been important raw material sources for housing, energy and decorative purposes. However, in modern time, it has become to utilized for many engineeringly made products such as pulp and paper products, composite panels, furnitures and some chemicals [1,2].

However, natural forest lands have become shortage due to increasing demand on wood-based materials. The consumers have also become more sensitive for preservation and destruction of natural forests. Thereby, woody like alternative new raw sources have been studied throughout the world and many agricultural (residues or wastes), forest (residues, low value woody materials), non-wood materials (annual plants), etc., have been found to useful into value added products instead of wood [3-5]. Moreover, there are numerous literature information for utilization of non-wood sources into processing of products [6-11]. The chemical and physical information on those raw materials and processing into composite manufacturing could be found elsewhere [3,8,9].

However, rice stalk or straw is one of the abundant lignocellulosic waste materials in the world. It was stated that 709.2 million tons of wheat straw and 673.3 million tons of rice stalks were exposed worldwide and that large amount of lignocellulosic raw material could be used in the production of composite panels [4]. In more recent study, it was proposed that rice is the third most important grain crop in the world behind wheat and corn. According to FAO statistics, world annual rice production in 2007 was about 650 million tons while every kilogram of grain harvested is accompanied by production of 1–1.5 kg of the straw [12].

In recent study, it was used the urea-formaldehyde glue as a binder for producing cotton waste-based panels with red pine chips and fibers, separately. It was concluded that in some preparation conditions, it is possible to produce particle- and fiberboards with cotton-based waste materials at acceptable level [13].

However, gypsum is one of the oldest construction materials throughout human beings. The gypsum based construction and ornamental materials were found ancient civilizations (i.e. Sumerian, Seljuk, Ottoman Aztec, Egyptian, Greek and Roman so on). It was used intensively in Renaissance architecture period in Europe due to its easy shape in interior and exterior spaces by architects and painters [14].

In this study, it was investigated to produce gypsum-based experimental composite panels with red pine wood/rice straw particles in the mixture up to 40/60 (w/w) in gypsum-water mixture. In this case, the fillers (additives) could be observed more closely. Thus, it is possible to produce gypsum-based experimental panels by selecting the most suitable processing conditions.

2. MATERIAL AND METHODS

The red pine wood chips were supplied from a local **timber** company where processing log to **timber**, Isparta-Turkey. The rice straw (stalks) was supplied from Can-Biga region of Turkey. Both raw materials have been carefully cleaned from dust, bark and other substances then turned into particles through scissors and screened to suitable particle dimensions. **The 10-50 mm particles** were utilized for adding to gypsum/water mixture.

63 They were then dried at atmospheric conditions until at least a 10-12% moisture content was
64 obtained.

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66 The commercial grade perlite plaster type Gypsum, used as binder agent, supplied by a
67 local company, Isparta, Turkey. After manufacturing, the experimental boards, they were
68 conditioned at 20 °C and 65% relative humidity. The detailed description of cellulosic raw
69 materials, gypsum with their specifications, and manufacturing process could be found
70 elsewhere [14].

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72 A total of 14 boards (two for each condition) were made. The experimental procedure for
73 manufacturing experimental particle boards as;

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- **Press temperature (°C):** Ambient temperature
- **Pressing time (day):** up to 14
- **Press pressure (N/mm²):** 0.1-1.0
- **Wood particles/rice stalk ratio (w/w,%):** 100-40/0-60
- **Board dimensions (mm):** 400x400x10 cm.
- **Target density (gr/cm³):** 0.75 (± 0.1).

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81 It is important to note that the compatibility of gypsum, red pine particles and rice straw are
82 important but not considered in this study. However, some certain chemicals may also be
83 used to shorten the curing time and improve compatibility of these substances into gypsum.
84 Therefore, only the pure effects of wood/gypsum/rice straw compatibility are considered.

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86 After reaching full strength at ambient temperature, the experimental boards were
87 conditioned at 20 °C and 6 % relative humidity and samples were cut to determine the IB
88 (Internal bond), MOE–MOR (Modulus of Elasticity and Rupture), TS (thickness swelling after
89 24 hours immersion in water), in accordance with TS EN 310 (1999), TS EN 319 (1999) and
90 TS EN 317 (1999), ASTM D 1037, respectively [15-17].

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92 A standard flame combustion test system was conducted according to TS EN-ISO 11925-2.
93 A visual observation of the sample was made either or not the flame spreads in the vertical
94 direction more than 150 mm above (the flame application point) [19-21]. For surface
95 chemical analyses, FTIR spectrophotometer (A Shimadzu (IR Prestige-21) was used to
96 evaluate the chemical groups present in the board surface. For thermogravimetric analysis
97 (TGA), Perkin Elmer SII instrument was utilized for measuring changes in boards as a
98 function of increasing temperature.

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100 The natural weathering tests were conducted on 50x50x10 mm samples from panels were
101 exposed to outdoor for two months then color and surface hardness tests conducted for
102 determining property changes. The total color differences (ΔE) of the samples were
103 measured by X-Rite SP68 Spectrophotometer using CIE L*,a*,b* standards (1976). The
104 surface hardness properties of both control and weathered samples were measured with a
105 Shore D hardness tester, according to test method of ASTM D2240 standard [22]. The
106 board's code numbers (wood chips and rice straw ratios) with gypsum content at various
107 proportions are given in **Table 1**.

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Table 1. The proportions and code numbers of gypsum based boards

Board code	Wood (gr)	Rice straw (gr)	Wood (%)	Rice straw (%)	Gypsum (gr)
DE0	1000	0	100	0	800
DE1	900	100	90	10	800
DE2	800	200	80	20	800

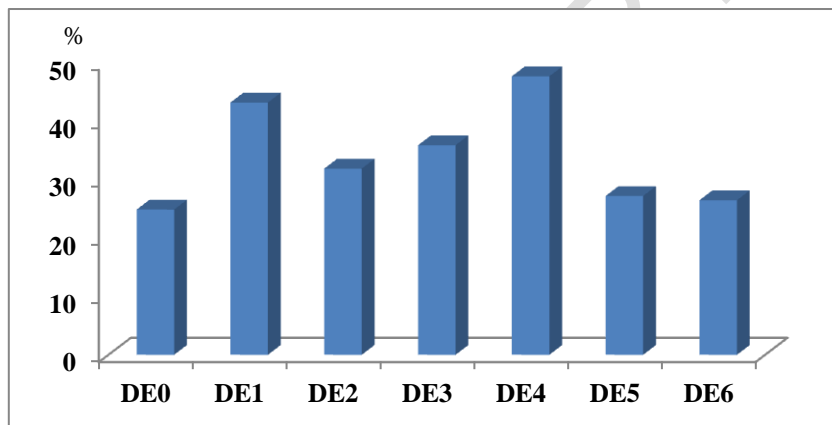
DE3	700	300	70	30	800
DE4	600	400	60	40	800
DE5	500	500	50	50	800
DE6	400	600	40	60	800

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3. RESULTS AND DISCUSSION

The thickness swelling properties of experimental boards in water are shown in Figure 1. It was realized that the lowest thickness swelling of 24.85% was observed in the panels produced from the mixture of wood/gypsum (DE0) (no rice straw added). However, the addition of rice straw to the mixture had adversely affects the thickness swelling properties. The highest thickness swelling value of 47.66% found at 60/40 wood/rice straw (w/w) mixture (DE4) condition. Interestingly, it was realized that the panels include rice straw was equal and/or higher than the wood chips (DE5 and DE6 boards), the thickness swelling values of 27.18% and 26.45% were found that only marginally similar to control sample (DE0), respectively.



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Figure 1. Thickness swelling properties of experimental boards in water.

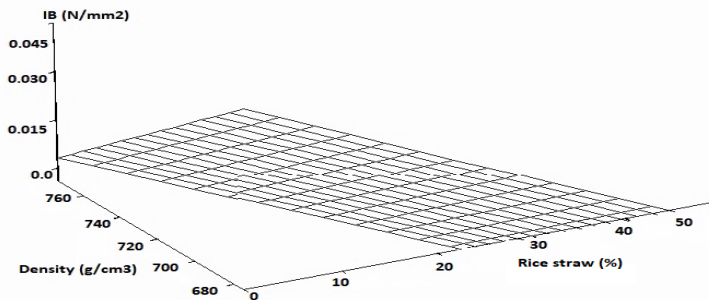
The comparative internal bond (IB) strength properties of experimental boards are shown in Table 2. The internal bond (IB) strength properties of test boards produced from wood/ rice straw-gypsum boards were lower than those of the board produced only with wood/gypsum mixture (DE0). That is, the addition of rice straw to the wood/gypsum mixture has a negative effect on the IB strengths in all conditions. The maximum IB strength was calculated as 0.06 N/mm² for control sample. In addition, the ANOVA general linear test results showed that the panels had no statistically difference IB values than each other at 95% confidence level.

Table 2. The IB strength properties of gypsum-based boards

Board code	Density (kg/m ³)	Internal bond (IB) (N/mm ²)	Difference from control (DE0) (%)
DE0	709.99	0.06	0.0
DE1	747.94	0.012	-80.0
DE2	717.94	0.014	-76.7
DE3	719.31	0.015	-75.0
DE4	705.89	0.013	-78.3
DE5	699.70	0.012	-80.0
DE6	697.48	0.011	-81.7

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The combine effects of panel density and rice straw additive level on IB properties shown in **Figure 2**. As seen, all rice straw addition negatively affects on IB properties some level. However, increasing panel's density had not much impact on IB of experimental boards.



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Figure 2. IB properties of experimental boards.

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The two important mechanical strength properties as a function of the mixture proportion with wood chip/rice straw is shown in Table 3. The bending strength values of the experimental panels (MOR) produced with various proportion additions to rice straw (D1 to D6) was found to be lower than those produced from only wood/gypsum mixture (DE0). This is obviously important in that the introduction of rice straw into the wood/gypsum mixture has the reducing effects on bending strength properties. However, the lowest MOR value was calculated as 0.97 N/mm² in the DE3 panel (70/30; wood chip/rice straw mixture).

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The elastic modulus characteristics (MOE) of the boards are also show similar trend. The highest MOE value of 553 N/mm² was calculated on the DE4 board, which was produced by adding 40% rice straw to the wood/gypsum mixture. It was clearly seen that the elastic modulus values of all boards produced by the addition of rice straw into wood/gypsum mixture were improved up to 60/40 wood/rice straw level (w/w), beyond this level the MOE values of experimental panels significantly reduced. In this sense, except a few conditions, the similar situations were also observed for Internal Bond strength (IB) and bending strength (MOR) properties. It could be summarize that the compatibility of rice straw with gypsum is low and should be useful only in controlled conditions or proportions.

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The Duncan's multiple range test results also showed that the rice straw and wood chips ratio had some statistically different MOR values that it was in the two for MOR while no statistically difference in MOE values.

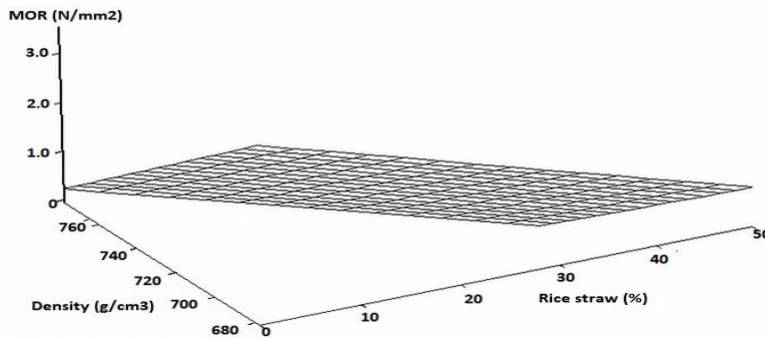
Table 3. The Bending strength and Modulus of Elasticity properties of experiemntal panels

Board Code	MOR (N/mm ²)	Difference from control (%)	MOE (N/mm ²)	Difference from control (%)
DE0	2.77 (B)	0.0	445	0.0
DE1	1.21 (A)	-56.3	113	-74.6
DE2	1.22 (A)	-55.9	192	-56.9
DE3	0.97 (A)	-64.5	443	-0.23
DE4	1.07 (A)	-61.4	553	24.3
DE5	1.16 (A)	-58.1	360	-19.1
DE6	2.30 (B)	-16.9	138	-68.9

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*Groups with the same letters in each column indicate that there is no statistical difference ($P < 0.05$) between the samples according to the Duncan's multiple range test.

The combine effects of both panels density and rice straw ratio impact on bending strength properties of experimental boards is shown in Figure 3. It could be seen that increasing rice straw ratio and panel's density has only marginally effects on panel's MOR values.



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Figure 3. The panels density and rice straw ratio effects on Bending strength properties of experimental boards.

The comparative surface hardness values (Shore D) of the experimental panels are given in **Table 3**. The highest Shore D hardness value of 44 was observed on the DE0 type board while the lowest value of 22 found for DE5 board produced from the mixture of 50:50 wood/rice straw. In general, it is understood that the hardness values of the boards produced with the increase in rice straw or the decrease in the wood ratio (at fixed the gypsum ratio) are affected negatively on hardness properties of experimental boards. However, an interesting situation was encountered that the hardness values of the boards kept under external atmospheric conditions were found to decrease less than the DE0 sample. The lowest hardness value reduction in the boards that were kept under external atmospheric conditions was calculated as 3.2% in DE3 and 4.3% in DE1 type boards, respectively. The increase in the ratio of rice straw in the mixture adversely affects the hardness values, but it is important to ensure that the hardness properties of the panels against external atmospheric conditions remain at a lower trend than control sample (DE0).

However, according to Duncan's multiple range test result, rice straw and wood chips ratio had some statistically different hardness values that it was in the six statistically different groups for experimental panels.

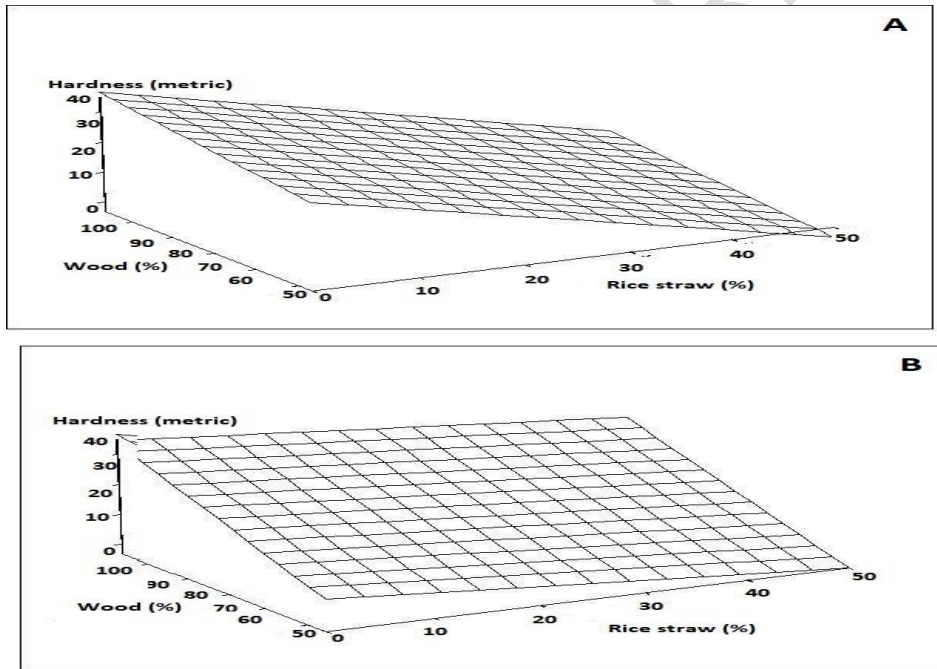
Table 4. Surface hardness properties oof boards.

Board code	Hardness (metric)	Difference from control (DE0) (%)	After weathering hardness (metric)	Changes (%)
DE0	44 (D)	0,0	33	-25
DE1	45 (E)	2.2	43	-4.4
DE2	32 (BC)	-27.2	27	-15.6
DE3	31 (CD)	-29.9	30	-3.2
DE4	23 (A)	-47.8	21	-8.7
DE5	22 (A)	-50.0	20	-9.1
DE6	26 (AB)	-40.9	25	-3.8

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*Groups with the same letters in each column indicate that there is no statistical difference ($P < 0.05$) between the samples according to the Duncan's multiple range test.

The effects of wood and rice straw ratio on both control and weathered experimental panels are shown in Figure 4. It was realized that the similar trend was found for both panels that increasing rice stalk ratio or (decreasing wood ratio) negatively effects on experimental panels.



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Figure 4. Hardness properties of boards (A: Control samples, B: Weathered samples)

The comparative surface optical (color) properties of boards are shown in Table 5. In general, it was proposed in literature that total color difference (ΔE) of samples are more useful for explain materials surface color changes rather than other CIE L^*, a^*, b^* properties [23]. The highest total color difference value of 8.15 was observed in the control sample (DE0) while the lowest in DE6 sample (0.63). It was also found that the highest whiteness color value of -35.48 (in metric) and the corresponding increase in the yellowness color value of 7.03 (in metric) were found in the DE0 board as well.

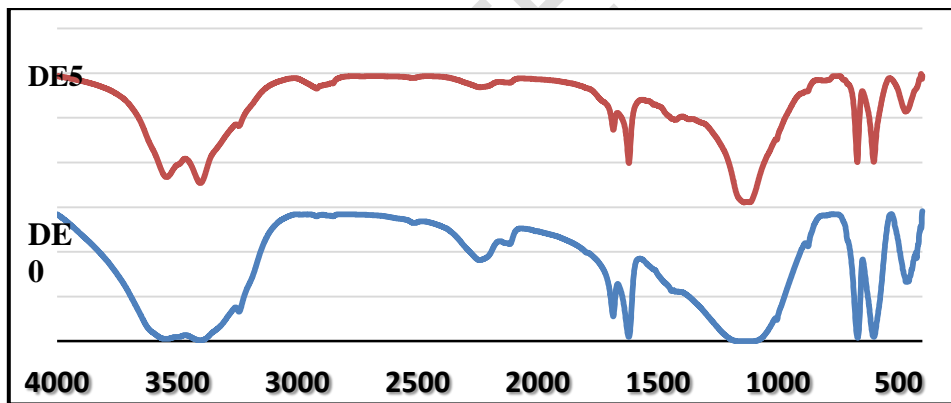
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Table 5. The surface color properties of experimental panels.

Board codes	ΔL	Δa	Δb	ΔE	CIE Whiteness	E313 Yellowness
DE0	-7,19	1,55	3,51	8,15	-35,48	7,03
DE1	-2,78	0,48	0,3	2,84	-8,09	1,01
DE2	1,26	-0,35	1,17	1,75	-3,48	1,56
DE3	-1,12	-0,16	0,8	1,39	-6,9	1,48
DE4	0,17	1,81	-1,46	2,33	8,79	-2,42
DE5	0,98	-0,44	1,27	1,67	-5,13	1,85
DE6	0,47	-0,39	-0,15	0,63	1,91	-0,38

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The comparative Fourier transform infrared spectroscopy (FTIR) analysis of DE0 and DE5 boards are shown in Figure 5. Generally, bands in the range of 1500-1610 cm^{-1} are considered as a characteristic peak for lignin components and composed of C=O and COO-symmetric tension vibrations in aromatic rings of lignin structure [14, 24]. However, the bands in the range of 1360-1380 cm^{-1} were generally showing C-H degradation in polysaccharides. The change in the bands at 1230-1270 cm^{-1} was reported to explain the vibration in the guayacil ring with CO groups in lignin and hemicellulose. Although some chemical groups could be modified under the water/gypsum environment that might be deteriorate to some extent (alkaline environment). In this sense, some bands are modified to some extent but all these groups observed on the surface of boards.

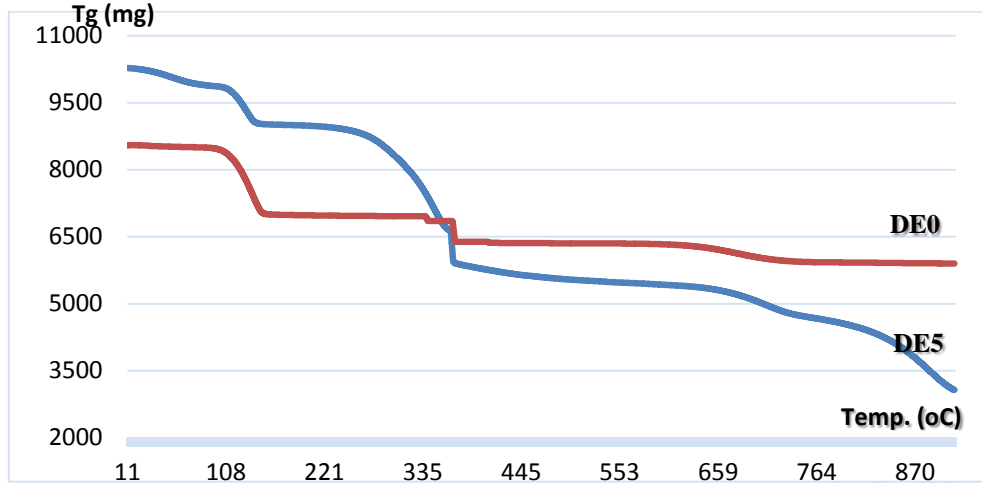


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Figure 5. The FTIR spectra of boards

The TGA (Thermal Gravimetric Analyzer) analysis based on temperature-time variables at range of 25-900 $^{\circ}\text{C}$ is shown in **Figure 6.** The TGA thermographs has divided four different regions as; heating zone (Tb); (the water assumed to remove) up to 120 $^{\circ}\text{C}$; cell wall degradation zone (Tm1); above 120 $^{\circ}\text{C}$ to 370 $^{\circ}\text{C}$; completely break down zone (Tm2); cell wall organic constituents completely break down up to 700 $^{\circ}\text{C}$; non-organics degraded zone (Ts) up to 900 $^{\circ}\text{C}$, on-organics have degraded and char residues obtained. In this approach, the mass loss of in that zones were found to be 2.5%, 27.7%, 49% and 55% for DE5 sample, respectively.

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259 **Figure 6.** The TGA micrographs of experimental panels

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261 According to the TS EN-ISO 11925-2 standard, the flame spreading characteristic of the
262 boards was conducted. However, the flame spreading pattern of all test boards produced by
263 adding wood/rice straw-gypsum based boards show that the flame did not reach the
264 threshold limit of 150 mm. This is a well indication of boards that could be classified as class
265 A1 (non-flammable material).

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267 For determining heat insulation properties, the temperature values passed to the back
268 surfaces were measured in accordance with DIN 4102 for 30 seconds intervals and for a
269 total of 300 seconds. The measured values were shown in Table 6. It has been observed
270 that the addition of rice straw and wood particles to the gypsum structure has a positive
271 effect on the heat transfer properties. It was measured as 39.1 °C in the DE6 panel. When
272 Table 6 is carefully examined, the addition of rice straw has improving effects on heat
273 insulation properties of boards. However, the lowest mass loss was also measured as 2.01%
274 on the DE6 boards that produced with the highest rice straw ratio (60:40, rice straw/wood
275 chips by weight).

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276 **Table 6.** The heat insulation properties of boards

	0 (sec.)	60 (sec.)	120 (sec.)	180 (sec.)	240 (sec.)	300 (sec.)	Total Mass loss (%)
DE0	20.2	20.2	24.3	41.9	58.8	66.0	4.15
DE1	20.0	22.7	32.4	45.1	52.0	62.9	3.90
DE2	12.9	15.2	16.3	20.5	30.4	40.9	3.04
DE3	14.2	15.3	23.1	35.0	45.3	56.1	6.21
DE4	10.8	12.9	14.6	19.3	28.7	40.3	2.66
DE5	13.8	14.4	16.0	25.6	32.5	45.3	2.65
DE6	11.8	12.1	15.4	25.1	34.6	39.1	2.01

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278 4. CONCLUSION

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280 In this study, the fundamental knowledge and approaches for producing gypsum based boards
281 from wood/rice straw was investigated. Thus could help researchers to this area gain the
282 understanding and to make meaningful contributions to this field of study. However, it is clear

283 that in order to be an composite panel has efficient mechanical and technological properties,
284 there must be a hydrophobic substances added to mixture for improvement thickness swelling
285 properties in water. Moreover, although the addition of rice straw to the wood/gypsum mixture
286 adversely affected the strength properties negatively, with using some longer fibers may
287 improve the strengths of panels. On the other hand, rice straw in wood chip/gypsum mixture
288 helps to improve heat resistance (insulation) properties some level. It was also realized that
289 the addition of rice straw to wood/gypsum mixture effects on extending hardening time. It is
290 probably gypsum-rice straw interaction that could not effectively wet and penetrate the rice
291 straw surface due to the hydrophobic wax and inorganic silica on the outer surface of rice
292 straw.

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