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Preparation of Cement Based Composites and Cellulosic Panels from Barley Straw for Thermal Insulation

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Abstract

Insulation materials are the indispensable elements for the conservation and economy of energy in buildings. This study investigates the thermal conductivity, compressive strength, fire behavior of cellulosic panels and cement based composites made from barley straw. The measured thermal conductivities were 0.071 W/mK for cellulosic panels and 0.11 W/mK for cement based composites on the average. According to the EN ISO 11925-2 ignitability test classification for cellulosic panels and straw-cement based composites were found as B-class. Most of the strength values of the composites were varied between 65 and 1270 kPa conform to ASTM and Turkish standards.

1. INTRODUCTION

Energy management in buildings, which were used for dwelling, education, business, public sector, etc., is becoming increasingly important. According to data for 2011, 34.47% of the Turkey's energy was consumed in homes; in the same year, industry was responsible for the 35.45% of Turkey's total annual energy consumption. A residential energy consumption survey in U.S. showed that space heating and cooling (space conditioning) accounted more than half of all residential energy consumption including space heating, air conditioning, and water heating appliances, electronics and lighting. Energy consumption in 1993 for space heating fell from 58% to 41.5% in 2009. Factors underpinning this trend have increased the adoption of more efficient equipment, better insulation, more efficient windows and population shifts to warmer climates [1].

Since the world energy resources are being consumed very quickly, the need for good insulating materials has become a critical/important issue. There are some methods to reduce the amount of heat loss from buildings. Applying heat insulation to roofs and walls can save 77% of the energy consumed by buildings [2]. Therefore, people involved in construction industry are searching new strategies for well-insulated buildings [3].

Nowadays, public health and environment protection concerns accelerate researches into natural sourced products. Wood and lignocellulosic materials belong to the natural biocomposites of plant origin, containing cellulose, hemicelluloses, lignin and other compounds. Their chemical structures and compositions depend on the plant nature such as: tree, annual, biannual and perennial plants rich in cellulose (bast plants like flax, hemp, kenaf, jute, roselle (karkadeh), and others like sisal, grass-like Miscantus, grain straw, reed, bagasse, bamboo, etc.) [4]. In order to recycle these natural resources, many people have succeeded in developing new lignocellulosic materials, which have been used in a great number of applications including textiles, geo-textiles, furnishings and composites [3, 5-8].

Straw (with 150 kg/m^3 density and 0.058 W/mK thermal conductivity) has been used as insulation material alone in historical buildings for filling the spaces in partition walls and it was mixed with clay and lime for producing adobe bricks (1200-1300 kg/m^3, 0.40 W/mK) in Anatolia [9]. Straw panels with a density of 320 kg/m^3 and a thermal conductivity of 0.0714 W/mK were used in Europe in this early century [10].

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Uncut straw was pressed at 470 K to 300-370 kg/m^3 in the 1930s in Sweden. The thermal conductivity was 0.0833 W/mK [11]. Many researchers, in particular Belhadj et.al. [12], have examined the production of lightweight and heat-insulating concrete using barley straw.

Fire resistance of insulation materials is an important property for the construction industry. Using different types of additive was investigated for enhancement of the fire resistance. Boric acid, ammonium phosphates and borates, ammonium sulphate and chloride, zinc chloride and borate, phosphoric acid, dicyanodiamide, sodium borate and antimony oxide are the most commonly used inorganic fire retardant additives for cellulosic materials. Organobromine compounds are also used as organic fire retardants [13]. Kurt et al. [14] used ammonium sulphate, borax, boric acid and zinc chloride to impregnate wood material (oriental beech) for the fire resistance. Metallurgical slags, fly ash and silica fume are the waste materials and evaluation of them like secondary building materials is gaining importance in terms of environmental pollution. Cree et al. [15] summarized the research dealing with effects of adding industrial waste such as fly ash, blast furnace slag, silica fume and biomass/volcanic ashes to concrete. Also, many researchers have recently become interested in the possibility of increasing the fire resistance of concrete with these materials. The replacement of cement with slag or fly ash has been found to be useful for the fire resistance of concrete [16].

In this study cement based composites containing barley straw, cement, pumice/furnace slag were produced as building materials, which have low thermal conductivity values. For the production of cellulosic panels available as thermal insulation material, barley straw was treated with 3, 4 and 6 mole/L HCl solutions, including $ZnCl_2$ at different temperatures to decompose straw structure, release lignin, for easier molding and fire resistance of cellulosic panels. An experimental full factorial design method was used for the evaluation of parameters effects on the thermal conductivity.

2. MATERIAL AND METHOD

Chopped barley straw, which is grown in Pasinler – Erzurum (east of Turkey), was used and processed using two different methods in this study:

1. Cement, cement + pumice or cement + furnace slag were mixed with the straw at different ratios and molded for the production of cement based composites. Table 1 lists the chemical compositions and apparent densities of these materials.

2. HCl solutions (3, 4 and 6 mole/L) were prepared and 0.05 kg $ZnCl_2$ was added per L of these solutions. The straw was treated with these solutions at different temperatures for the production of cellulosic panels. Then the straw was washed with water and filtered. The waste solution was stored for later use.

Table 1. *Chemical compositions of used materials*

Material(%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Others	Apparent density kg/m^3
Cement	19.02	3.27	3.11	63.7	3.12	7.78	955
Furnace slag	45.62	31.90	1.95	2.25	16.80	1.48	1060
Pumice	70	14	2.5	0.9	0.6	12	515

Thermal conductivity was determined using a thermal conductivity meter with a needle probe. Three different points were used to determine an average thermal conductivity for samples and two measurements were achieved at one point.

Fire behaviors of composites and panels were determined by a method which was described in EN ISO 11925-2 standard [17]. The test took place inside a test chamber where the test specimen with a thickness 40 mm was mounted vertically. The test specimen was subjected to the edge and surface exposure from a gas flame. During the test, time of ignition, burning droplets and whether the flames reached the top marking of the test specimen within a prescribed time period were recorded. Classes of reaction to fire performance for composites and panels were determined according to the commission decision of implementing Council Directive 89/106/EEC [18] as regards with the classification of the reaction to the fire performance of construction products.

Determination of compressive strength was performed by Schimadzu Ag-Is 100kN. Among the prepared mixes, rectangular specimens of cross sectional area of 40 mm x 60 mm were tested for their compressive strength using a compressive testing machine with constant 1mm/minute rate of loading. Compressive strength was calculated by dividing the maximum compressive load on the specimen by the initial cross-sectional area as follows:

$$\sigma = P / A \tag{1}$$

Where: σ = compressive strength (kPa), P = maximum load (kN) and A = cross sectional area (m^2).

2.1. Preparation of straw – cement based composites

Compositions of composites containing straw, cement, pumice, furnace slag are shown in Table 2.

Table 2. *Components and codes of cement based composites*

Components (%)	Composite code														
	S_1	S_2	S_3	SP_1	SP_2	SP_3	SP_4	SP_5	SP_6	SF_1	SF_2	SF_3	SF_4	SF_5	SF_6
Straw	22	26	32	32	32	25	25	22	22	32	32	25	25	22	22
Cement	54	50	44	32	21	42	33	44	33	32	21	42	33	44	33
Water	24	24	24	26	26	25	25	22	22	26	26	25	25	22	22
Pumice	-	-	-	10	21	8	17	11	22	-	-	-	-	-	-
Furnace slag	-	-	-	-	-	-	-	-	-	10	21	8	17	11	22

Dry straw cannot adhere to the cement mix. To create strong and durable composites, straw was treated with water using different methods before adding the mixture of composites:

Straw was kept waiting in water at room temperature for 24 hours and used for the mixture of composite after filtration.

Straw was added into water at room temperature and heated to the boiling point. Boiled for 5 minutes and filtrated before adding to the mixture.

So, for the first treatment 15, for the second treatment 15 and in total 30 different composite mixtures were prepared. The mixtures were put into steel molds (11x 6x4cm), which were lubricated by synthetic oil and were compressed. The surface was leveled and compacted by applying approximately 10 kN pressure. The slabs were stripped after 24 hours and cured with water for 7 days. At the end of the seventh day, slabs were dried in a stove at 105ºC until they reached a constant weight. Their densities changed in the range between 690 – 1150 kg/m^3. Thereafter, thermal conductivities and compressive strengths of these cements based composites were measured.

2.2. Preparation of cellulosic panels

In this part of the study, the experimental full factorial design method was used to observe the main and interaction effects of four parameters (factors) that were chosen for the preparation of straw panels. Factors and their levels can be seen in Table 3. There are four parameters, whereas there are two or three levels. In the industrial experimental design, the factorial experimental design and orthogonal central composite design methods are widely used to obtain empirical linear models relating to process responses to process factors, with a minimum effort of experimentation and with the highest level of statistical confidence. Using this method, modeling becomes possible and requires only a minimum number of experiments. Furthermore, the analysis performed on the results is easily recognized and experimental errors are minimized [19-23].

Table 3. *Designed experimental factors and their levels for cellulosic panels*

Factors	Code	Level 1	Level 2	Level 3
Solution concentration (mole/L)	X_1	3	4	6
Solution temperature (°C)	X_2	60	70	80
Stove temperature (°C)	X_3	80	120	-
Waiting period in stove (hours)	X_4	2	4	-

The first step was heating the solutions. When the temperature of the solutions reached to the specified value, the straw was added to solutions and kept at a constant temperature throughout the determined experiment time (15-10-5 minutes for the solutions 3 - 4 - 6 mole/L respectively). Thereafter, the straw was filtered and washed with water to remove HCl and $ZnCl_2$. It was filtered again and mixed with adhesive and then molded. The surface was leveled and compacted by hand. The slabs were placed in an oven bag and kept in the oven within a specified time at the specified temperature. Next, samples were taken out of the bags and dried in an oven until they reached a constant weight. Finally, the thermal conductivities of the cellulosic panels were measured. Their densities were changed in a range between 230-300 kg/m^3.

3. RESULTS AND DISCUSSION

3.1. Thermal conductivity

3.1.1 Straw-cement based composites

The thermal conductivities of straw-cement based and two different conventional composites can be seen in Table 4.

Table 4. *Thermal conductivity values of straw-cement based composites.*

Composite code	Thermal conductivity (W/mK)	
	Including straw boiled in water	Including straw soaked in cold water
S_1	0.125	0.125
S_2	0.122	0.128
S_3	0.098	0.098
SP_1	0.077	0.081
SP_2	0.072	0.08
SP_3	0.106	0.123
SP_4	0.108	0.100
SP_5	0.135	0.138
SP_6	0.130	0.122
SF_1	0.081	0.078
SF_2	0.088	0.08
SF_3	0.126	0.130
SF_4	0.118	0.116
SF_5	0.132	0.124
SF_6	0.130	0.127

The lowest thermal conductivities were measured for S3, SP1, SP2, SF1 and SF2 composites within the range 0.072 to 0.098 W/mK , which had maximum ratio of straw. When the ratio of straw increased in composites, thermal conductivities decreased due to the low thermal conductivity of straw (0.058 W/mK). Similarly, the highest thermal conductivities were measured for S1, S2, SP5, SP6, SF5, SF6 composites within the range 0.125 to 0.135 W/mK , which had minimum ratio of straw. Belhadj et.al. [24] used barley straws and wood shavings for the production of sand concrete based composites. They found the thermal conductivity of composites which including barley straw as 1.35 W/mK with 1900 kg/m^3 dry density. Ashour et. al. [25] used wheat straw, barley straw, wood shavings, soil and sand to produce natural plaster materials. They showed that the thermal conductivity of all materials decreased with the increase in straw fibre and sand content. For this study, two linear models have been found between straw percentages and thermal conductivities of samples which one group was produced from boiled straw and the other group was produced from straw soaked in cold water. Figure 1 shows these equations and R-squared values.

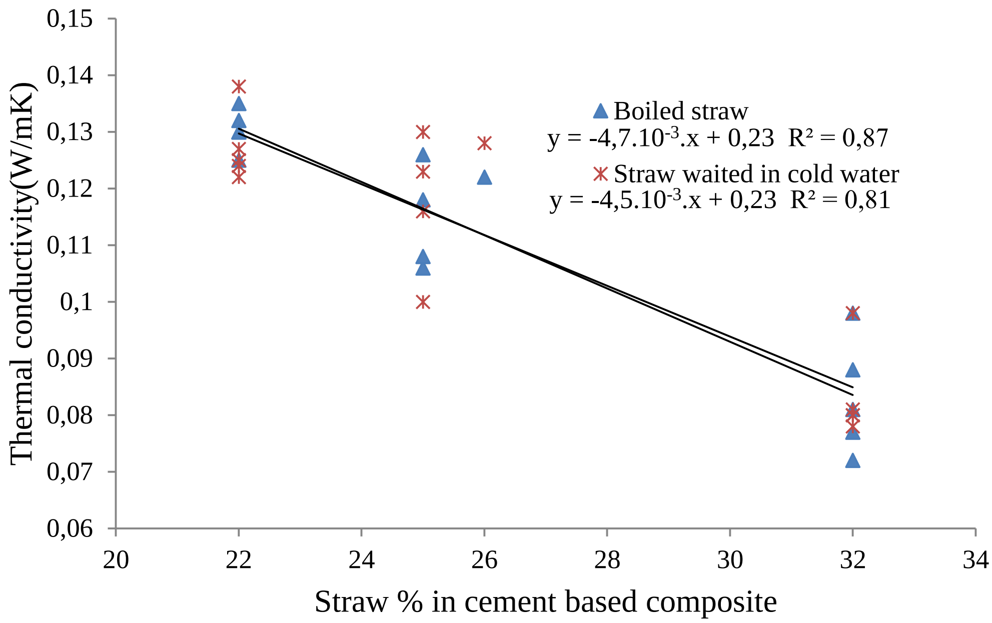


Figure 1. *Linear models for cement based composites*

When the ratio of straw implies a minimum value, the addition of pumice or furnace slag to the mixtures increased their thermal conductivities (S_1, SP_5, SP_6, SF_5, SF_6). Nevertheless, when the ratio of straw is at a maximum, the addition of pumice or furnace slag to the mixtures decreased the thermal conductivities (S_3, SP_1, SP_2, SF_1, SF_2). In this case, we can consider the amount of cement in the composites. The minimum thermal conductivity values belong to the composites having the maximum ratio of $\frac{(\text{straw} + \text{pumice or furnace slag})}{\text{cement}}$ and also the maximum thermal conductivity values belong to the composites having the minimum ratio of $\frac{(\text{straw} + \text{pumice or furnace slag})}{\text{cement}}$. This second situation may have originated from adherence forces of cement that was adhered to the straw closely and blocked air spaces. Keeping the straw in water or boiling did not significantly affect the thermal conductivity values. So, for economic production, soak into water is preferable.

3.1.2. Cellulosic panels

The full factorial experimental design was used in this study to observe the main and interaction effects of the factors (concentration of HCl solutions (X1), solution temperature (X2), stove temperature (X3), and waiting period in stove (X4) on the thermal conductivity values of panels. Figure 2 shows the main effects of the factors. The effect of a factor is defined as the change in response, produced by a change in the level of the factor. This is frequently called a main effect because it refers to the primary factors of interest in the experiment. The main effects plots are graphs of the marginal response averages at the levels of the factors [19].

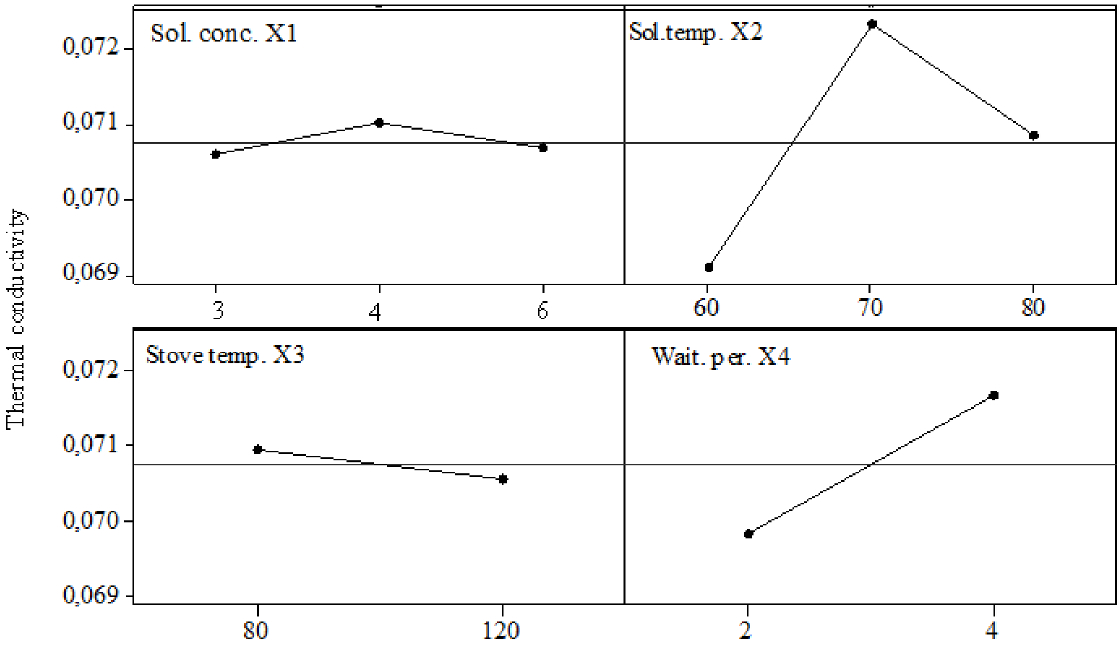


Figure 2. *Main effects plot of factors*

A statistical analysis of variance (ANOVA) table was performed using the Minitab software [26] and prepared from the values of Table 5. ANOVA determines the significance and percentage of contribution for each parameter. Usually the larger F-ratio and percentage of contribution values show a greater effect on the thermal conductivity. The optimal combination of process parameters and their levels can be predicted using ANOVA analyses. ANOVA presented in Table 6, showed that the main effect of solution temperature (X2), waiting period in stove (X4) and interaction effects of solution concentration-solution temperature (X1X2), solution concentration-stove temperature (X1X3), solution temperature- stove temperature (X2X3), solution temperature-waiting period in stove (X2X4), solution concentration-solution temperature-stove temperature (X1X2X3) and solution concentration-solution temperature- waiting period in stove (X1X2X4) were the significant model terms that influenced the thermal conductivity of panels. The greater percentage contribution values of these main and interaction terms in ANOVA table supported these results.

Table 5. *Thermal conductivity values of straw panels*

Sol. conc. X_1	Sol. temp. X_2	Stove temp. X_3	Waiting per. X_4	Thermal conductivity(W/mK)
3	60	80	2	0.0645
3	60	80	4	0.0660
3	60	120	2	0.0700
3	60	120	4	0.0705
3	70	80	2	0.0780
3	70	80	4	0.0740
3	70	120	2	0.0680

3	70	120	4	0.0685
3	80	80	2	0.0705
3	80	80	4	0.0790
3	80	120	2	0.0675
3	80	120	4	0.0705
4	60	80	2	0.0645
4	60	80	4	0.0745
4	60	120	2	0.0690
4	60	120	4	0.0770
4	70	80	2	0.0730
4	70	80	4	0.0660
4	70	120	2	0.0730
4	70	120	4	0.0705
4	80	80	2	0.0760
4	80	80	4	0.0730
4	80	120	2	0.0690
4	80	120	4	0.0665
6	60	80	2	0.0690
6	60	80	4	0.0685
6	60	120	2	0.0620
6	60	120	4	0.0735
6	70	80	2	0.0705
6	70	80	4	0.0710
6	70	120	2	0.0775
6	70	120	4	0.0780
6	80	80	2	0.0695
6	80	80	4	0.0695
6	80	120	2	0.0655
6	80	120	4	0.0735

Table 6. *Analysis of variance for thermal conductivity values of straw panels*

Source	Degrees of freedom	Sum of squares	Mean squares	F-ratio	Percentage of contribution
X_1 (Sol. conc.)	2	0.0000012	0.0000006	0.07	0.19
X_2 (Sol. temp.)	2	0.0000635	0.0000318	3.67	10.12
X_3 (Stove temp.)	1	0.0000014	0.0000014	0.16	0.22
X_4 (Wait. per.)	1	0.0000303	0.0000303	3.5	4.82

$X_1 X_2$	4	0.0000658	0.0000165	1.9	10.48
$X_1 X_3$	2	0.0000351	0.0000175	2.03	5.60
$X_1 X_4$	2	0.0000122	0.0000061	0.7	1.93
$X_2 X_3$	2	0.0000702	0.0000061	4.06	11.19
$X_2 X_4$	2	0.0000782	0.0000351	4.52	12.46
X_3X_4	1	0.0000122	0.0000391	1.42	1.96
$X_1 X_2X_3$	4	0.0001244	0.0000123	3.6	19.85
$X_1 X_2X_4$	4	0.0000742	0.0000311	2.14	11.80
$X_1 X_3X_4$	2	0.0000222	0.0000185	1.28	3.53
$X_2 X_3X_4$	2	0.0000020	0.0000111	0.12	0.33
Error	4	0.0000346	0.0000010		5.51
Total	35	0.0006272	0.0000086		100

S = 0.00294038   R-Sq = 94.49%

When the main effect figure (Figure 2) is examined to determine the optimal conditions, it can be seen that the lowest thermal conductivity was obtained for 3 M solution concentration, at 60ºC solution temperature and 120ºC stove temperature during a two hour waiting period in a stove. Nada et al. [27] decomposed crystalline structure of cellulosic materials by metal chloride solutions. In this study increasing solution concentration and temperature led to decomposing of straw and releasing lignin. It is known that lignin is a natural wood binder. In these conditions air spaces were blocked so the porosity was decreased in the composites and thermal conductivity values were increased. The significant effects of the solution temperature (X2) and waiting period in stove (X4) parameters were also observed with their main effect plot lines having the highest inclination in Figure 2. An interaction plot matrix is presented in Figure 3 to verify these findings. The four independent factors are listed along the diagonal. Graphically, an interaction is identified by comparing the inclination of the lines. Non parallel lines indicate interaction; the greater the lines depart from being parallel, greater the degree of interaction. It can be seen in the graph that there are significant interactions between solution concentration-solution temperatures (X1X2), solution concentration-stove temperature (X1X3), solution temperature- stove temperature (X2X3) and solution temperature-waiting period in stove (X2X4).

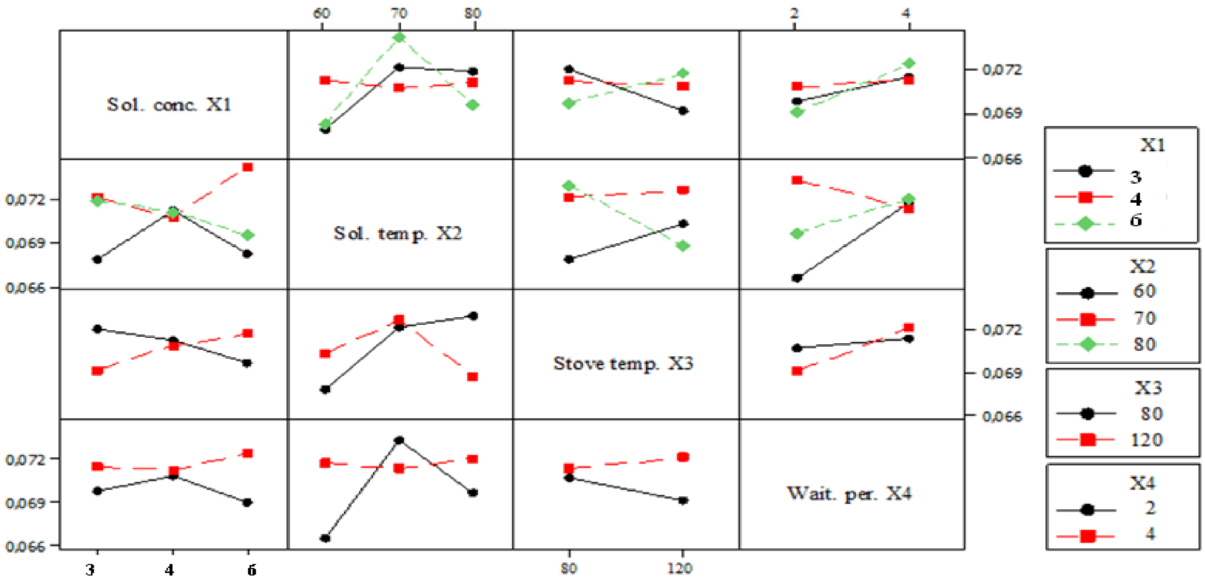


Figure 3. *Interaction effects plot of factors*

3.2. Compressive strength

3.2.1. Straw-cement based composites

Compressive strength values of some samples are presented in Table 7. In order to show the effect of straw and other additives on compressive strength, some cement based composite samples were tested. Results are grouped as A, B and C for evaluation. While group B contained only straw in addition to cement, groups A and C included puzzolonic materials as well. In A and C group composites, reducing the cement and increasing the pumice or furnace slag amount increased the compressive strength. Similarly, compressive strengths of B group composites increased with the respect to the decreasing amount of cement and increasing amount of straw. Pre-treatment of straw with cold water or boiling water did not affect the composites strengths. It was showed that the straw was effective for improving the ductile behavior of adobe, and straw fibre could be used as reinforced material [28-29]. Binici et al. [30] considered that the minimum compressive strength values required for traditional brick should be between 500-1000 kPa according to Turkish and ASTM standards. It is seen that most of these composites conform to standards.

Table 7. *Compressive strength values of cement based composite samples.*

group	composite code	straw%	cement%	water%	pumice%	furnace slag%	compressive strength(kPa)
A	S_1	22	54	24	-	-	65
	SP_5	22	44	22	11	-	840
	SP_6	22	33	22	22	-	1270
	SF_6	22	33	22	-	22	380
B	S_1	22	54	24	-	-	65
	S_2	26	50	24	-	-	605
	S_3	32	44	24	-	-	923
C	SP_5	22	44	22	11	-	840
	SP_6	22	33	22	22	-	1270
	SP_1	32	32	26	10	-	310
	SP_2	32	21	26	21	-	1260

3.2.2. Cellulosic panels

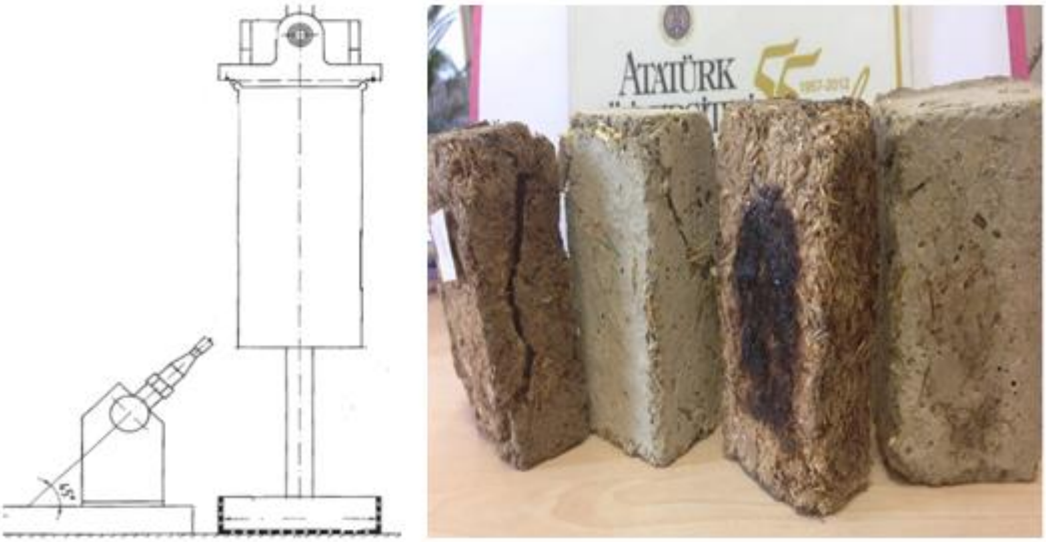
Compressive strength of straw panels calculated by averaging was a value of 144 ± 30 kPa. There is no certain standard for compressive strength of panels including straw.

3.3. Fire performance

European Union (EU) countries have adopted the Euroclass system which is based on the response to the fire performance of building products. The classification is based on observations whether the flame spread (Fs) reaches 150 mm within a given time period and whether the filter paper below the specimen ignites due to flaming debris. According to the ignitability test EN ISO 11925-2, the classification of cellulosic panels and straw-cement based composites is showed in Table 8. Figure 4.a shows the fire performance specification system and Figure 4.b shows the samples exposed to fire and compressive strength. It has been seen that the flame was only partially effective on the surface and could not penetrate into the samples. After compressive strength tests many samples have not broken into pieces, they have preserved their structural integrity.

Table 8. *Fire performance of panels and composites.*

Class	Test Method	Classification criteria	Additional classification
B	EN ISO 11925-2 Exposure = 30 s	Fs≤150 mm within 60 s	No flaming particles



(a) Fire performance specification (b) Samples that exposed to fire and compressive strength

Figure 4. *Fire and compressive strength performance of panels and composites*

4. CONCLUSION

The production of thermally efficient materials for residential building construction is of importance in most areas of the world. Production of cement based composites and cellulosic panels for thermal insulation presented in this study. Cement, cement + pumice, cement + furnace slag were mixed with the barley straw at different ratios in order to produce cement based composites. The straw was treated with 3, 4 and 6 mole/L HCl solutions including $ZnCl_2$ at different temperatures for the statistically planned production of cellulosic panels. The experimental full factorial design method was used for this section of the study. The composites with the biggest ratios (straw + pumice or furnace slag)/cement and the panels that were produced at minimum solution concentration and minimum solution temperature have minimum thermal conductivity values. The measured thermal conductivities (approximately λ = 0.071 W/ mK for cellulosic panels and λ = 0.11 W/mK for cement based composites) are higher than those of conventional insulation materials such as organic foamy materials, glass or mineral fibres (λ = 0.02-0.04 W/ mK) which are widely used as building insulation materials. Nevertheless, in an experimental study the results showed that the organic foamy materials, polyethylene foam and polyurethane foam did not meet the requirements of the low fire hazard material and were unsuitable for proofing buildings [31]. The panels and composites produced in this study have good fire resistance. In addition, they create a healthier living space in contrast to the organic foamy materials containing CFCs and HCFCs.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors

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