Original Research Article
Mineral Based Boards made from Lignocellulosic Wastes. Part 2. Chemical and Technological Properties

Abstract

The aim of this study was to investigate experimental panels produced from mixture of agriculture wastes with mineral adducts under synthetic adhesive bonding process. In this respect, the effects of the two different mineral loading as reinforced additive (olivine and dolomite) in lignocellulosic matrix (agricultural residue of tomato- and eggplant stalk chips) system were investigated. The highest heat conductivity value of λ: 0.461 W/mK and 0.449 W/mK was found on panels that made only from tomato stalk- and eggplant stalk chips (controls) while lowering effects were found when dolomite and olivine added to matrix at various proportions. But all heat conductivity value were found to be lower than standard value (λ: <0.065 W/mK). However, all the experimental panels show the burning pattern on the surfaces which char did not reach the 150 mm threshold limit, regardless of board formulations or experimental conditions. It could be proposed that proportions of olivine and dolomite as reinforcing element in lignocellulosic matrix have a lowering effects on flame spreading at certain extent. It is notable that mass loss (%) properties of samples were found to be quite different from the insulation values. For dolomite-based panels, the lowest mass loss values were found to be 8.83% and 9.97% for boards prepared from similar proportion (1:1, w/w, %) of dolomite-tomato stalk chips (XT_V) and dolomite-eggplant stalk chips (XE_V), respectively. For dolomite-based panels, the lowest mass loss of 6.79% and 9.11% were found to be boards produced from 30% tomato stalks chip and 70% olivine (YT_{III}), 60% eggplant stalk chip and 40% olivine in mixture proportions (YE_{IV}), respectively. It is realized that the panels produced with mineral additives show higher degradation temperature with less mass loses (%, w/w) than controls. These are clear evidence that the presence of dolomite and olivine with lignocellulosic matter make panels more durable against fire and significantly reduces the mass loss. The FTIR spectra show characteristic spectrum of lignocellulose structure concentrate in the range of 800–3500 cm^{-1} and the major peaks in that range had been identified.

Keywords: Boards, agricultural waste, olivin, dolomite, heat conductivity, insulation properties, FTIR.

1. INTRODUCTION

Biocomposite materials have usually made from lignocellulosic sources with using formaldhyde-based adhesives as bonding agent [1-3]. Because the deforestation concern has been emerged for wood supply from forestland, numerous studies have already been carried out on the feasibility of alternative non-wood sources for forest products industry [4,5], particularly on biocomposite [4-7] and paper manufacturing [8]. Thus, many agricultural wastes/residues and annual plants which naturally grown have reported to be suitable in chemical and physical properties, similar to wood in worldwide [9].

Some examples are, poppy (*papaver somniferum*) stalk [10], cotton carpel [11], peanut-shell flour [12], sunflower stalk [13], sugarcane waste [14], kenaf [15], vine pruning [16], coconut shell [17], and bagasse fiber/coconut shell particle hybrid [18]. It was proposed that mechanical and thermal stability of the composite manufacture from NaOH-treated coconut shell-reinforced cardanol enhanced due to a decrease in particle size with alkaline treatment [17]. In another study, the hybrid biocomposites prepared from bagasse fiber/coconut shell particle show acceptable strength properties [19]. Hence, there are many valuable literature reports on producing lignocellulose-based materials from non-wood sources with acceptable properties [10-19].

Although biocomposites generally utilized for construction and general purposes, there are also many other usage of those materials [3]. They could be useful both in interior (i.e. dividing rooms, coverings of walls, ceilings, doors), and exterior applications (i.e. homes, commercial units, prefabricated structures, architectural structures) depends on their matrix elements and adhesive types [20-21]. However, certain technological and mechanical advantages of those products over other materials have already been reported by researchers [5-16]. But it is important to note that special care should be taken during preparation particularly improving hydrophobic and insulation performances.

One of the solution for improving heat and fire resistance properties of those are using chemicals or substitute elements that higher resistance than lignocellulosic matters in network structure. In these regards, certain minerals which have higher heat resistance over lignocellulosics could be used in formulations which make material more resistant to fire [19, 23]. However, mineral added biocomposites are hybrid materials that combine lignocellulosic components within a synthetic resin. In the composite structure, lignocellulosics are main ingredients (matrix) and minerals are functional (reinforced) elements [3, 22, 23]. The inorganic additives may make a special element, but within limits. While the heat used to set synthetic adhesive does not affect minerals properly, rather effects on lignocellulosic substrates. But it is important to note that the effect of inorganic elements strongly depends on the size, shape, content, surface characteristics, aspect ratio, and dispersion in matrix [24]. Although the performance of traditional synthetic adhesive bonded biocomposites in combination with other reinforcing elements (i.e. fibers, chips, particles) has been well demonstrated in engineering applications, inorganic elements have a higher density than lignocellulosics and could be contributed to many desired properties [25].

Some minerals, including cement, gypsum, magnesia have been investigated for their utilization in various type material manufacturing [26]. It has been proposed that some reinforced elements (mineral additives) are capable of forming stable structures with strength improvement properties after reacting with the wood in the presence of water [25-28]. Among alternative mineral-lignocellulosic formulations, olivine and dolomite may prove to be the promising candidates with some improving properties [27-28]. However, applications of those over conventional biocomposite process may have some advantage in improving certain properties, including resistances of biotic- (fungi and insects) and abiotic (UV radiations, moisture, heat) factors.

There is limited research on the properties of boards which made from mixture of agricultural wastes with olivine and dolomite as mineral adducts under synthetic adhesive bonding process. A systematic investigation has been carried out with two different agricultural waste (eggplant stalk- and tomato stalk chips) substrates to determine the effects of olivine and dolomite as mineral adducts onto experimental boards formulations and the chosen methods. The first part of this study, "*Mineral Based Boards made from Lignocellulosic Wastes: Part 1-Physical and Mechanical Properties*" was published [24]. In the second part of this study, we seek to demonstrate clear effects on selected technological properties of olivine and dolomite mixed with eggplant-and tomato stalk particles to produce an alternative mineral reinforced biocomposite product.

2. MATERIALS AND METHODS

The agricultural wastes of eggplant- and tomato stalk chips were used for particleboard production obtained from the greenhouses of Antalya province, Turkiye. These are collected and separated from soils and other substances after the main production in greenhouses. The olivine and dolomite used in the experiments were supplied from a mining company, operated in Aksu, Antalya. The olivine $(Mg^{2+}, Fe^{2+})_2SiO_4$ and dolomite $[CaMg (CO_3)_2]$ had similar specific gravity of 2.8-3.0 g/cm^3. These minerals were crushed and grinded in size of 3-10 mm, prior to mixing with lignocellulosic substrates to form composite paste. The urea-formaldyhde adhesive was supplied from a commercially operated a particleboard plant and utilized as received. The synthetic adhesive was used in this study was constant (10%) at all experimental conditions. A detailed information on sample preparation could be found first parts of this study [27]. A total of 48 boards (two for each condition) were made. The experimental procedure for manufacturing experimental particle boards as follows:

- Press temperature (°C): 150-175
- Pressing time: 5.0 min.
- Press pressure (N/mm^2): 2.5-3.0 Mpa
- Board dimensions (mm): 400x400x10 mm.
- Target density (gr/cm^{-3}): 1.0 (± 0.1).

The standard test method for thermal conductivity by hot wire (Platinum resistance thermometer technique) was used to determine thermal insulation behavior of composites according to ASTM C-1113-09 by QTM 500 device [29]. The reaction to fire tests was determined according to the ISO 11925-2 and DIN 4102-1 [30]. The test samples were cut according to standard of 90x250 mm pieces and placed on the test apparatus at vertical position. Single-source small flame simulation on the edge of the bottom of samples applied at 45^o slope and distance of 20 mm from the middle of samples was initiated. The total test duration was 60 s. at the end of test, whether the specimen ignition occurs or not 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. For determining mass burning rate, the test samples were cut as standard dimensions of 100x100x10 mm pieces and placed on the test apparatus at vertical position. The boards were flamed at approx. 800 ^oC with the distance of 30-50 mm from the heater surface in duration of 5.0 min. At the end of test, the boards were weighted and mass loss calculated based on weight differences. Thermogravimetric analysis (TGA), Perkin Elmer SII instrument was utilized in order to determine the thermal degradation change. For surface chemical analyses, FTIR spectrophotometer (Shimadzu, IR Prestige-21) was used to evaluate the chemical groups present in surfaces.

While many combinations were tested, some code number and abbreviations were established throughout the study given in Figures and Tables. These are; **X:** Dolomite in composite formulations, **Y:** Olivine in composite formulations, **E:** Eggplant stalk chip in composite formulations (Type 1 boards), **T:** Tomato stalk chip in composite formulations (Type 2 boards), X**-/Y-,** $_{\mathrm{I,\,II,\,III,\,IV,\,V}}$**:** Dolomite and olivine proportions (w/w, %) of 10-,20-,30-,40- and 50%, respectively.

3. RESULTS AND DISCUSSIONS

The heat conduction coefficient values of the experimental boards are comparatively given in Table 1. It has found that panels produced in mixed proportion of olivine and dolomite with eggplant- and tomato stalk chips in matrix system were lower heat conduction properties than controls, regardless of experimental conditions. It is clear evidence for improving insulation properties with using both mineral additives in matrix. However, the highest heat conductivity values of λ: 0.461 W/mK were found with control samples which made from 100% tomato- and eggplant stalk chips (E_0 and T_0) while the lowest value of λ: 0.338 W/Mk was found panel produced with 10% dolomite and 90% (w/w, gr) tomato stalk chip (XT_I) in experimental panel structure (Type 2 boards). But it is important to note that all findings are lower than standard value that the required (λ: <0.065 W/mK) for a material to rated as an insulation material, according to ISO and CEN standards [23].

Like panels made from mixture of tomato stalk chip-olivine and dolomite (XT and YT–based boards), similar results were also found with panels prepared from mixture of eggplant stalk chip and olivine/dolomite (Type 1 boards). However, the highest heat conduction coefficient value was observed in the control sample (E_0) with λ: 0.449 W/mK while the lowest value was measured in panel made from 1:1 (w/w, %) olivine-eggplant stalk chips (YE_V) with value of λ: 0.303 W/mK.

Table 1. Heat conduction (λ, W/mK) properties of experimental panels

Boards	**X**	**Y**
	Type 1 experimental boards	
E_0	0.449	0.449
XE_I-YE_I	0.359	0.333
XE_{II}-YE_{II}	0.412	0.362
XE_{III}-YE_{III}	0.403	0.348
XE_{IV}-YE_{IV}	0.417	0.316
XE_V-YE_V	0.406	0.303
	Type 2 experimental boards	
T_0	0.461	0.461
XT_I-YT_I	0.338	0.392
XT_{II}-YT_{II}	0.409	0.423
XT_{III}-YT_{III}	0.471	0.413
XT_{IV}-YT_{IV}	0.408	0.361
XT_V-YT_V	0.469	0.428
Standard (ASTM C1113/C1113M-09)	If <0.065, *thermal insulation material*	

In order to determine combustion properties of panels, a single flame source combustion test (Fig.1A) and combustion behaviors (char spreading) (Fig. 1B) visual results are presented. It could be seen that the burning pattern on the surface of all samples produced with proportion mixture of olivine to eggplant stalk- and tomato stalk chips did not reach the 15 cm threshold limit (Type 1 and 2 boards), regardless of board formulations or experimental conditions (Fig. 1A). It can be suggested that adding dolomite and olivine to the mixture improves the combustion condition on the surface of the material to a certain extent (Fig.1B). Similar behavior characteristics of samples which produced with the addition of dolomite to eggplant stalk- and tomato stalk chips were also observed. It is seen that the burning pattern on the surface of all dolomite added samples did not reach the 15 cm threshold limit (Fig. 1A). It can be proposed that proportions of olivine and dolomite as reinforcing element in lignocellulosic matrix has an improving effects on flame at certain extent.

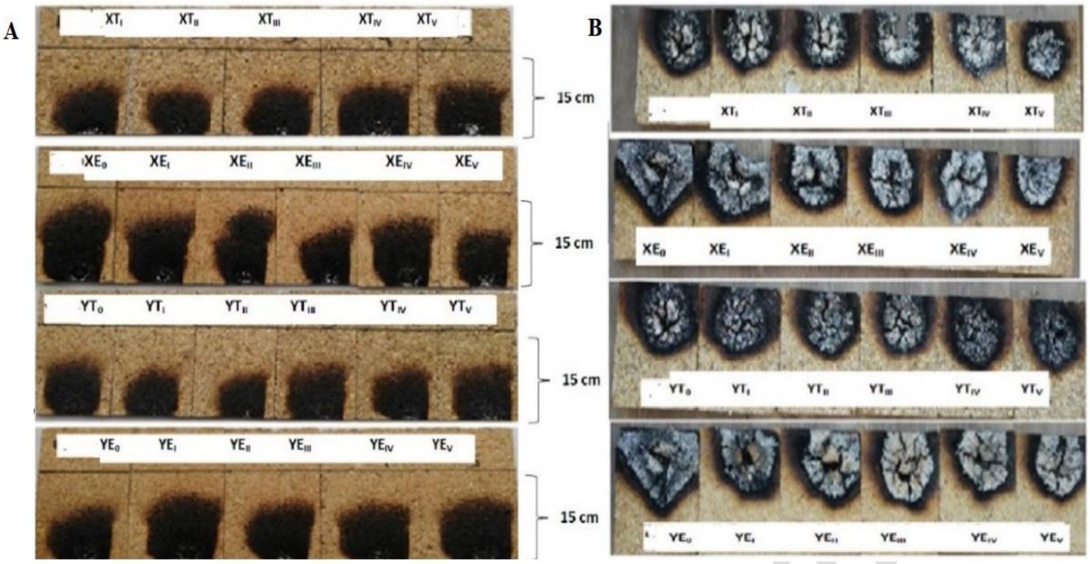

Figure 1. The fire behaviors on the experimental panels which exposed to a single flame source (**A:** Flame spread according to TS EN-ISO 11925-2, **B:** Combustion behaviors of panels).

For further evaluating combustion behaviors of panels and determining effects of mineral additives (olivine and dolomite) to lignocellulosic matrix in terms of heat insulation properties, a single flame combustion test was conducted and 120-second intervals measured results are comparatively shown in Table 2. However, the lowest heat permission values on the back side surfaces of samples was observed in sample of T_0 (prepared from 100% tomato stalk chips) as 101.7 ^{o}C, and in sample of E_0 (prepared from 100% eggplant stalk chips) as 97.6 ^{o}C, respectively. This is surprising considering typically higher heat-resistant for minerals added to experimental panels, rather than lignocellulosic matter. This is also contrasts to finding in heat conduction coefficient values (Table 1). But when Table 2 is carefully examined, it could be realizable that the mass loss (%) properties of samples were quite different from the insulation values (mass loss % of samples measured after 300 s. combustion). Surprisingly, the lowest mass loss values were found to be 8.83% and 9.97% for boards prepared from similar proportion (1:1, w/w, %) of dolomite-tomato stalk chips (XT_V) and dolomite-eggplant stalk chips (XE_V), respectively. This is another evidence that the presence of dolomite make panels more durable against fire and significantly reduces the mass loss.

However, similar results have also been observed with olivine added experimental panels. The lowest mass loss of 6.79% was found with sample of YT_{III} which produced from 30% tomato stalk chips and 70% olivine in mixture proportions. It was found to be 9.11% with sample of YE_{IV} which 40% olivine and 60% eggplant stalk chips in mixture proportions. It appears that olivine has also an improving effects which significantly reduces the mass loss of experimental panels.

Table 2. Thermal insulation and total mass loss (%) properties of experimental panels

Samples	**0s**	**120s**	**240s**	**300s**	**Mass loss (%)**
Type 1 experimental boards					
E_0	18.1	50.7	84.4	97.6	24.46
XE_I	18.4	65.9	101.3	145.0	24.87
XE_{II}	18.9	51.3	88.9	120.8	14.01
XE_{III}	18.6	58.8	103.1	125.9	12.06

XE$_{IV}$	18.6	55.6	99.4	129.0	11.07
XE$_{V}$	18.7	57.7	99.4	139.5	9.97
YE$_{I}$	21.9	37.2	61.5	98.4	13.68
YE$_{II}$	21.3	47.6	66.4	107.3	14.04
YE$_{III}$	20.0	60.7	90.0	110.0	10.49
YE$_{IV}$	20.7	62.3	112.7	111.9	9.11
YE$_{V}$	20.5	56.9	122.4	220.5	11.55
Type 2 experimental boards					
T$_{0}$	20.1	53.1	84.0	101.7	12.76
XT$_{I}$	20.4	64.9	99.3	117.3	12.44
XT$_{II}$	20.3	54.7	70.7	124.1	11.87
XT$_{III}$	20.6	69.4	121.1	179.7	12.43
XT$_{IV}$	20.0	59.6	92.4	183.0	11.75
XT$_{V}$	22.9	71.0	113.8	175.3	8.83
YT$_{I}$	18.4	62.2	95.4	109.6	11.36
YT$_{II}$	18.3	60.1	87.8	109.4	11.86
YT$_{III}$	16.7	54.4	74.8	105.7	6.79
YT$_{IV}$	19.8	78.3	144.0	164.8	10.35
YT$_{V}$	21.3	64.4	115.2	159.0	7.53

Figure 2 depicts the effect of dolomite and olivine on the thermal degradation (TGA) of experimental boards. When Figure 2 (A-D) carefully reviewed, it was realized that olivine and dolomite mixed both type boards (Type 1 and 2) looks marginally similar plot shapes. However, control panels (T$_0$ and E$_0$) show a very high mass loss (%), especially at initially and this continues up to final temperature level. It appears to both dolomite and olivine containing boards looks resistance against thermal degradation at similar temperatures. It is presumed that this is probably due to the typical high heat resistance properties of minerals in panel structure. Because panels produced from only tomota stalk- and eggplant stalk chips typically contains polysaccharides, lignin and some extractive substances that easily degraded with heat rather than dolomite and olivine [31].

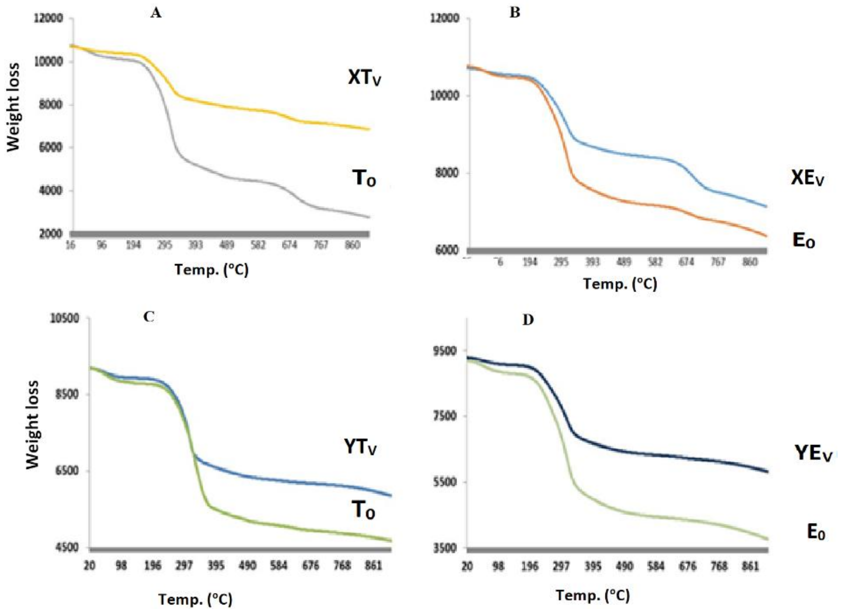

Figure 2. TGA analysis of experimental panels

The TGA curves were divided at three different temperature level to evaluate thermal degradation properties of experimental panels. These are; starting temperature (Tb), first maximum temperature (Tm), and final temperature (Ts) and measured mass losses (%) are given in Table 3. For boards produced with only tomato stalk chips (T_0) and eggplant stalk chips (E_0), the initial degradation temperature (Tb) was determined as 185 ˚C and 178 °C, the maximum temperature (Tm) 315 °C and 312 °C, the final temperature (Ts) 411 °C and 383 °C, and final mass loses were calculated to be 58.63% and 57.87%, respectively. On the other hand, the Type 1 boards (XT_V and YT_V) show 13 °C to 19 °C higher initial (Tb) degradation temperature while 3 °C to 53 °C lower final (Ts) degradation temperature levels than control. However, although Type 2 boards (XE_V and YE_V) show marginally similar initial (Tb) and first maximum degradation (Tm) temperatures, but they had 7 °C to 63 °C higher final (Ts) degradation temperature levels than control. It appears that the degradation temperatures of panels produced with mineral additives show some variables. But it is clear that both olivine and dolomite effects preserving mass loss of samples when used in composite network structure. The data presented in Table 3 evidenced that conclusion.

Table 3. Thermal degradation (TGA) and mass loss properties of boards

Samples	Tb (°C)	Mass loss (%)	Tm (°C)	Mass loss (%)	Ts (°C)	Mass loss (%)
Type 1 experimental boards						
T_0	185	8.05	315	41.69	411	58.63
XT_V	204	6.88	315	34.72	414	52.90
YT_V	198	6.72	311	39.66	358	55.18
Type 2 experimental boards						
E_0	178	5.85	312	42.0	383	57.87
XE_V	178	3.55	312	25.3	445	37.73
YE_V	178	3.73	312	27.71	390	37.67

Comparative FTIR spectra of some selected sample's prepared with mixture of dolomite-eggplant- and tomato stalks chips are shown in Figure 3A. However, the characteristic spectrum of lignocellulose structure concentrate in the range of 800–2000 cm^{-1} and the major peaks in this range had been identified (E_0 and T_0). All spectra exhibit multi-modal absorption in the 600-1000 cm^{-1} region due to –OH groups. The out-of-plane C-H vibration was assigned at 800 cm^{-1}. However, the spectra of dolomite contained samples (XE_V and XT_V) exhibit the more absorption in the 800-1600 cm^{-1} region relative to the controls, where a more complex OH vibration is dominant. However, the less intense CH_2-CH_2- vibrations (1400-1800 cm^{-1}) and C-C and C-O-C peak areas indicates some modification. This modification can be seen in stretching 1000-1700 cm^{-1} region (Fig. 3A).

The FTIR spectra of panels prepared with mixture of olivine-eggplant- and tomato stalk chips are shown in Figure 3B. The characteristic spectrum of lignocellulosics could also be visible in the range of 500–2000 cm^{-1} and in 2900-3500 cm^{-1} (E_0 and T_0). There is no clear different spectra determined with dolomite filled eggplant- and tomato mixed panels (YE_V and YT_V). However, more less similar intense CH_2-CH_2- vibrations (1400-1800 cm^{-1}) and C-C and C-O-C peak areas could be visible. In the spectra, the band at 900-1150 cm^{-1} is attributed to C-C out of the plane stretching, C-C-O stretching at 1060 cm^{-1}; C-O-C symmetric stretching at 1150 cm^{-1} (Fig. 3B).

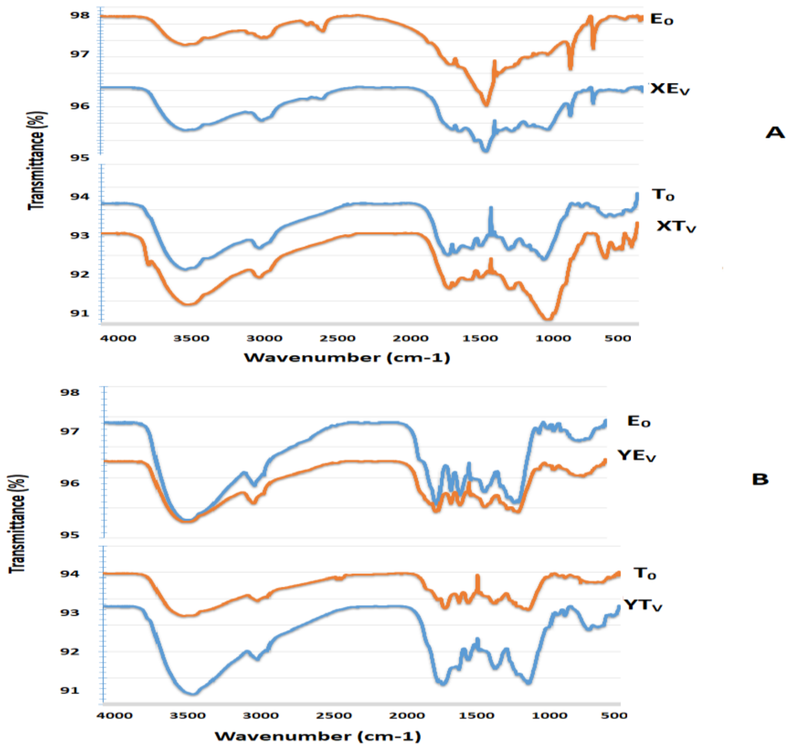

Figure 3. FTIR spectra of experimental panels (A: Dolomite added panels, B: Olivine added panels).

4. CONCLUSION

There is a great potential for the use of agricultural wastes to make mineral based composites. These panels have been studied for various purposes. However, the use of these panels are highly dependent on physical and mechanical properties, and all the other underlying factors which determine public acceptance of a product. In this study, selected physical properties were evaluated with tomato stalk- and eggplant stalk chips mixing with olivine and dolomite at certain proportions in presence of synthetic resin as bonding agent. The experimental results clearly indicates that the fire resistances of panels are improved some level, while causing a lower mass loss and heat conduction coefficient which is a sign of a chemical change on the structure of the boards. In general, the results of this study on the bio- composite materials are compatible with the findings in the literature.

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