

Properties of Experimental Panels Made from Mixture of Dolomite and Olivine with Calabrian pine Wood Particles. Part 1. Physical properties

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Abstract – The effects of two mineral adducts (dolomite and olivine) as proportion in Calabrian pine wood chip based-experimental panels which bonded with synthetic adhesive (UF) were evaluated. It appears both minerals (P: calabrian pine wood chip-, X: Dolomite- Y: Olivine in composite proportions) improve the water repellent efficiency (WRE) properties at certain extent. In all experimental procedure, WRE increased with dolomite and olivine proportions higher than >10% in board formulations. The highest WRE of -34.2% and -35.4% were found with PX5 and PY5 samples that made with 1:1 (w/w) dolomite/wood and olivine/wood formulations, respectively. Like water sorption, similar trend was also found for thickness swelling (TS) properties. The lower TS values were found to be in range of -11.2 to -56.4% for dolomite-, and -55.5 to -69.5% for olivine based panels, respectively. Only samples of PX1, which produced 1:9 dolomite/wood chips (w,w, %) proportions, show higher IB value than control (PX0: 0.87 MPa vs PX1: 0.99 MPa) while rest of dolomite- pine wood panels show lower IB values. In contrast to dolomite, olivine appear to be improving effects on IB properties at certain conditions. The highest IB value of 1.21 MPa was found with sample PY4, which produced 4:6 olivine/wood chips (w,w, %) proportions, indicate approximately 39.1% higher IB value than control. It is notable that olivine seems to more effective than dolomite at similar board preparation formulations in terms of lowering TS, WA and creating higher internal bond strength properties than dolomite based panels at similar experimental conditions. However, only sample of PY1, made with 1:9 (w, w,%) olivine/wood and proportions, show higher bending strength (13.27 MPa) and elasticity (2468.2 MPa than control (PY0:12.75 MPa vs 2245.2 MPa).

Keywords – Dolomite, olivine, particleboard, calabrian pine, strength properties, internal bond, modulus of rupture

Dolomit ve Olivin ile Kızılçam Odun Yonga Karışımından Üretilen Deneme Levhalarının Özellikleri. Bölüm 1. Fiziksel Özellikler

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Araştırma Makalesi

Öz – Çalışmada, kızılçam odun yongalarından sentetik tutkal (üre-formaldehit) kullanılarak hazırlanan deneme levhalarına, farklı oranlarda iki mineral madde ilavesinin etkisi araştırılmıştır (Kompozit içeriğinde; P: Çam odun yongası, X: Dolomite Y: Olivine olduğunu belirtir). Mineral ilavesinin, levhaların su itici özelliklerini belli derecede iyileştirdiği belirlenmiştir. Tüm deneme şartlarında, %10 ve daha fazla dolomit ve olivin ilave edilmiş levhaların su itici özellikleri yükselmiştir. En yüksek su itici özellik iyileştirme; 1:1 (ağırlık/ağırlık) dolomit/odun ve olivin/odun ile formüle edilmiş PX₅ ve PY₅ örneklerinde %34.2% ve %35.4 olarak elde edilmiştir. Su absorpsiyonuna benzer eğilim, örneklerin kalınlık artım özelliklerinde de gözlemlenmiştir. En yüksek kalınlığına artım iyileşmesi % -11.2 ile -56.4% dolomit ileve edilmiş levhalarda, %55.5 ile -69.5% olivin ilave edilmiş levhalarda bulunmuştur. Sadece 1:9 dolomit/odun yongası karışımından (ağırlık/ağırlık, %) üretilmiş PX₁ deneme numunesinde, kontrol örneklerinden (PX₀: 0.87 MPa; PX₁: 0.99 MPa) daha yüksek yüzeye dik çekme direncine (IB) ulaşılmış, diğer tüm levhaların yüzeye dik çekme dirençleri, kontrollerden daha düşük bulunmuştur. Dolomitten farklı olarak, olivin ilavesinin levhaların yüzeye dik çekme direncine bazı şartlarda olumlu katkı sağladığı anlaşılmıştır. Olivin ilaveli levhalarda, en yüksek yüzeye dik çekme direnci, 1.21 MPa olarak 4:6 olivin/odun yongası (ağırlık, ağırlık, %) oranıyla üretilmiş PY₄ örneğinde hesaplanmış ve bu değer, kontrol örneğinden yaklaşık %39.1 daha yüksek bulunmuştur. Olivin, dolomite göre benzer odun-mineral madde formülasyonlarında su içinde kalınlık artımı ve su absorpsiyonuna karşı daha etkili olduğu ve daha yüksek yüzeye dik çekme direnci sağladığı gözlemlenmiştir. Sadece 1:9 (ağırlık, ağırlık, %) olivin/odun karışımı ile üretilmiş PY₁ deneme levhası kontrol örneğinden daha yüksek eğilme direnci (13.27 MPa) ve elastiklik (2468.2 MPa) göstermiştir (PY₀:12.75 MPa ve 2245.2 MPa).

Anahtar Kelimeler – Dolomit, olivin, yongalevha, kızılçam, direnç özellikleri, yüzeye dik çekme direnci, eğilme direnci

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1. Introduction

Because of higher weight-to-strength ratio, easy to process and cheaper than many synthetic sources, wood is still the leading raw material for making engineeringly design composite materials in worldwide. In particular, coniferous trees are usually preferred because their morphological characteristics are more suitable and adequate in terms of production of high strength of products (Bergland and Rowell, 2005; FPL, 2010; McKeever, 1997). However, a wide variety of production variables determine the physical and mechanical properties of those materials. Among them, wood/binder ratio, density, particle geometry, wood type and chemical composition have already reported to be primary importance (Yalcin, 2018; Vasiliev and Morozov, 2001). Typically, 90-92% of the weight of that products are lignocellulosic matter, while the rest consists of adhesive (6-8%) and other functional additives (1-2%) (Vasiliev and Morozov 2001; Bergland and Rowell 2005; Yalçın *et al.*, 2019; Sahin and Simsek 2021). It has well presented by various comprehensive studies that strength of those affected by the percentage of matter replacement as adducts, alone and/or with their fractions.

Mining industries generate million tons of waste annually while these materials occupy large parts of landfills and causing environmental pollutions worldwide. However, lack of space in dense population cities creates the problem of incipient landfills, disposal of wastes, and various pollutions like air, water, and soil. Therefore, finding a solution or utilization of those are an emerging issue for environmental and economical purposes. It has already proposed that olivine and dolomite are widely available pozzolanic minerals with the ability to improve some properties of materials (Yalcin, 2018; Yalçın *et al.*, 2019 and 2020). Chemical properties of dolomite [$\text{CaMg}(\text{CO}_3)_2$], a rock-forming mineral, is primarily composed of calcium oxide, silica, magnesium oxide, and small amounts of aluminum oxide and iron oxide (Yalcin, 2018). Olivine is also a rock-forming mineral consisting of magnesium iron silicate [$(\text{Mg}^{2+}, \text{Fe}^{2+})_2\text{SiO}_4$]. The relative density of dolomite and olivine are varying from 2.65 to 2.85 gr/cm^3 depending upon the source of the stones (Yalcin, 2018).

Recent studies on investigating the effectiveness of using olivine as an adduct have demonstrated improvement in fundamental soil properties such as compressive strength, permeability characteristics, and swelling properties (Emmanuel *et al.*, 2020). However, several researchers investigated the some properties of biocomposites consists of dolomite and olivine as mineral additive with natural sources (Yalçın *et al.*, 2019 and 2020; Özdemir, 2016; Özdemir *et al.*, 2016). It was reported that the addition of dolomite as filler to the structure of HDF boards that produced from beech-pine fibers, clearly effects some physical and mechanical properties of water absorption, thickness swelling, modulus of rupture (MOR), modulus of elasticity (MOE) and surface roughness lowered in some level while the fire resistance properties increased (Özdemir 2016; Özdemir *et al.*, 2016). It has reported that both olivine and dolomite as adduct in biocomposite formulations with mixture of eggplant- and tomato stalk chips could be feasible and improve some properties (Yalçın *et al.*, 2020). But limited studies have attempted to prepare bio-composites using olivine and dolomite by employing an experimental approach. However, the proper combinations for optimum improvement in the strength property of the lignocellulosics using olivine and dolomite prepared with mixture is not clear. In this regard, the current study aimed at applying in experimental approach the contents of olivine and dolomite mixed with calabrian pine to improve the strength property of composite materials.

2. Material and Methods

Calabrian pine (*Pinus brutia*) wood chips (approx. 2.0 cm) and urea-formaldehyde adhesive were supplied from a commercially operate a particleboard plant, in Isparta province, Türkiye. The minerals of olivine and dolomite materials were collected from Isparta-Aksu mining sites in Türkiye. The wood chips dried in the oven at 105 °C (± 3) until they reached 3.0% moisture content. The 65% urea-formaldehyde adhesive and 20% ammonium chloride hardener were used in the production of the experimental boards. The glue and hardener ratio were constant in all conditions, at 10% and 1% (w/w), relative ratio, respectively. Metal mold plates with the dimensions of 40 cm x 40 cm x 10 mm was used to made experimental panels. The specially prepared board paste was pressed for 5-10 minutes under 3.0-5.0 N/mm^2 at 130-160 °C with laboratory type electrically heated hot-press. The experimental panels were kept between metal plates after the end of pressing process

and then climatized. The target densities of the manufactured boards were $0.9 \text{ gr/cm}^{-3} (\pm 0.1)$; a total of 24 boards (two from each condition) were made. The panels were conditioned at $23 \text{ }^\circ\text{C}$ and 65% relative humidity and samples were cut to determine the IB (Internal Bond), MOE and MOR (Modulus of Elasticity and Rupture), in accordance with TS EN 310 (1999), TS EN 319 (1999) and TS EN 317 (1999) standards, respectively. The small samples were soaked in distilled water for 24 hours. Water absorption and water repellent efficiency were calculated according to Eq.1 and Eq. 2 given below.

$$\text{Water absorption, WA (\%): } [(W2 - W1) / W1] * 100 \quad (1)$$

$$\text{Water repellent efficiency, WRE (\%): } [(Wu - Wt) / Wu] * 100 \quad (2)$$

Where, $W1$: initial weight of the sample before water soaking, $W2$: weight of the sample after water soaking, Wu : water absorption of control sample, Wt : water absorption of mineral adducts containing sample.

A general linear model procedure (ANOVA) was employed for data to interpret interaction of the panels manufactured. Duncan test was used to make comparison among board types for each property tested if the ANOVA found significant.

Many combinations were tested, some code number and abbreviations were established throughout the study given in Figures and Tables. These are; P: Calabrian pine wood chips in composite proportions, X: Dolomite in composite proportions, Y: Olivine in composite proportions, X-/Y- 0, 1, 2, 3, 4, 5: Dolomite and olivine fractions (w, w, %) of 0-, 10-, 20-, 30-, 40- and 50%, respectively.

3. Results and Discussion

The water sorption (WA) behavior of a lignocellulosic material is closely related to equilibrium moisture content (EMC) (Cai et al., 2017). It has proposed that water sorption is one of a pivotal marker of the matrix system as it reflects chemical and structural properties of composite system (Paës et al., 2019). However, in a hydrophobic/hydrophobic mixture system, it could be revealed how each proportions (mineral and lignocellulosic adducts) influence sorption properties. The water absorption properties of experimental boards are comparatively given in Table 1. The only sample of PX_1 show higher sorption value than control (PX_0 : 83.40% vs PX_1 : 87.46%), which could be negligible at the time scale of our experiments. All other experimental boards show -4.8 to -35.4% lower sorption properties for dolomite-based experimental boards and -2.9 to -38.1% lower sorption properties for olivine-based experimental boards. The experimental results reveal a previously unknown role of minerals of dolomite and olivine in water-sorption resistance, which is by means of sealing of hygroscopic wood particles. It could be hypothesized that olivine and dolomite addition could be inherited with the reduction in water sorption while those could be improved the barrier properties of polymer matrix because of creation of tortuous paths for penetrant water molecules (Karumuri et al., 2017).

Table 1

Water Sorption (WA, %) properties of experimental panels

Board Code	WA (24 h)	Differences (%) from control
Dolomite-based panels		
PX_0	83.40 ^b	-
PX_1	84.76 ^b	1.6
PX_2	63.25 ^a	-4.8
PX_3	64.06 ^a	-23.2
PX_4	61.71 ^a	-26.1
PX_5	53.91 ^a	-35.4
Olivine-based panels		
PY_0	83.40 ^c	-
PY_1	81.01 ^{bc}	-2.9
PY_2	70.39 ^b	-15.6
PY_3	53.61 ^a	-35.7
PY_4	56.02 ^a	-32.8
PY_5	51.63 ^a	-38.1

* Values sharing the same capital letter (s) within a column are not statistically different at the 0.05 level of confidence.

The marked effect of mineral adducts on water repellent efficiency (WRE, %) of experimental boards are clearly evident in Figure 1. It could be seen that similar trend was found with dolomite and olivine which increasing mineral adducts in board formulations (>10%) very effective for creating resistance against water. The highest WRE of -34.2% and -35.4% were found with PX₅ and PY₅ samples that made with 1:1 (w/w) dolomite/wood and olivine/wood proportions, respectively. The wood particles have a high tendency to hold water due to capillary forces (surface tension) as well as undergo volumetric expansion (swelling), an effect known as imbibition (Karumuri et al., 2017). However, the WRE properties appear to well correlated with adduct proportions, where the polymer relaxation and diffusion may conduct together. These comparisons between the board formulations and the measured results reveal the WRE of experimental panels can be predicted quite well by adducts proportions.

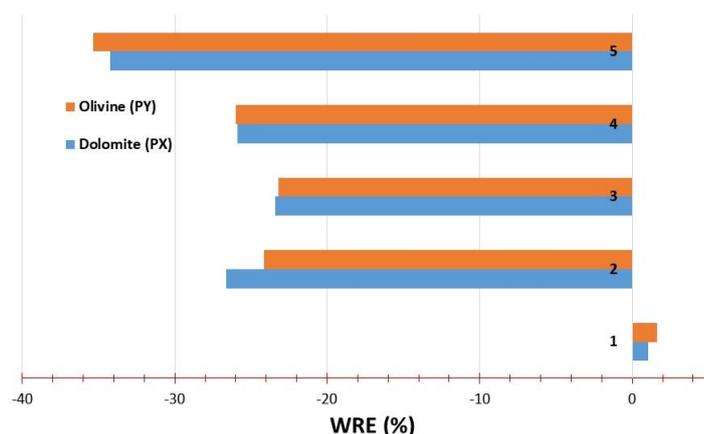


Figure 1. Water Repellent Efficiency (%) of experimental boards

Table 2 shows the thickness swelling properties in water (24 h) for experimental panels. Similar trend was also found that only sample of PX₁ show higher TS value than control (PX₀: 26.01% vs PX₁: 29.53%) while rest of samples show lower TS values than control, regardless of mineral type and experimental conditions. It was calculated to be -11.2 to -56.4% lower TS values for dolomite-based panels and -55.5 to -69.5% for olivine-based panels, respectively. However, the lowest swelling value of 11.33% was found with sample PX₅ for dolomite-based panels and 7.93% was found with sample PY₅ for olivine-based panels, respectively. It is important to note that increasing dolomite and olivine in boards formulations have lowering effects on TS values while it seems to olivine more effective than dolomite at similar formulations. This could be attributed to the less interaction between wood and water.

Table 2

The Thickness swelling (24 h, %) properties of experimental panels

Board Code	TS (24 h)	Differences (%) from control
Dolomite-based panels		
AX ₀	26.01 ^a	-
AX ₁	29.53 ^a	13.5
AX ₂	23.09 ^a	-11.2
AX ₃	16.83 ^a	-35.3
AX ₄	13.43 ^a	-48.4
AX ₅	11.33 ^a	-56.4
Olivine-based panels		
AY ₀	26.01 ^c	-
AY ₁	24.57 ^c	-5.5
AY ₂	18.47 ^{bc}	-28.9
AY ₃	18.40 ^{bc}	-29.3
AY ₄	11.56 ^{ab}	-55.6
AY ₅	7.93 ^a	-69.5

* Values sharing the same capital letter (s) within a column are not statistically different at the 0.05 level of confidence.

Thickness swelling of composite materials are very sensitive to matrix structures and this must be taken into account for the measurements. However, 2h and 24h TS values (%) have shown similar trend while it appears

a positive relationship with both adducts for experimental boards (Fig.2). These results could be expected considering lowering WA (Table 1), WRE (Fig.1) and TS (Table 2 and Fig.2) which is well correlated with adduct concentrations. Moreover, at certain conditions, it was reported by sahin et al., (2022) that both dolomite- and olivine adducts to biomass in matrix system could be lowering effects on water sorption and thickness swelling of boards (Sahin et al., 2022). The result found in this study is also in good agreement with the results reported on dolomite- and olivine- based biocomposites (Yalcin, 2018).

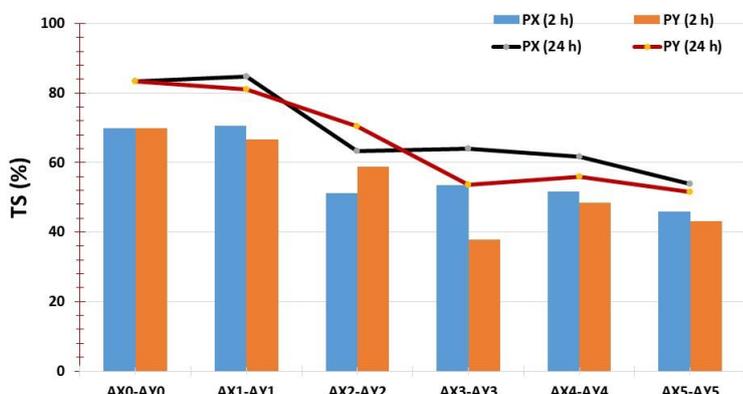


Figure 2. Thickness swelling (%) of experimental boards

Table 3 show internal bond (IB) properties of experimental panels, comparatively. However, only samples of PX₁ in dolomite-Calabrian pine panels show higher IB value than control (PX₀: 0.87 MPa vs PX₁: 0.99 MPa) while rest of dolomit-calabrian pine wood panels show lower IB values. The lowest IB value of 0.45 MPa was found with sample of PX₅ which prepared with 1:1 dolomite/calabrian pine wood chip proportions, followed by PX₄ (0.49 MPa), PX₃ (0.51 MPa) and PX₂ (0.76 MPa). These are approximately -48.2-, -43.5-, -41.4-, and -12.6% lower than control sample, respectively. It is clear increasing dolomite (> 10%) in panel formulations lowering effects on IB properties of panels.

In contrast to dolomite, olivine seems to improve on IB properties at certain conditions. The samples of PY₁ and PY₅ show -6.9-, and -4.6% lower IB values while samples of PY₁, PY₃ and PY₄ show 10.3-, 25.3- and 39.1% higher IB values than control. It is notable that olivine-based panels show higher internal bond strength values than dolomite-based panels at similar experimental conditions.

Table 3

The internal bond (IB) strength properties of experimental panels

Board Code	IB (MPa)	Differences (%) from control
Dolomite based panels		
PX ₀	0.87 ^b	-
PX ₁	0.99 ^b	13.8
PX ₂	0.76 ^b	-12.6
PX ₃	0.51 ^a	-41.4
PX ₄	0.49 ^a	-43.5
PX ₅	0.45 ^a	-48.2
Olivine based panels		
PY ₀	0.87 ^{ab}	-
PY ₁	0.96 ^{ab}	10.3
PY ₂	0.81 ^a	-6.9
PY ₃	1.09 ^{bc}	25.3
PY ₄	1.21 ^c	39.1
PY ₅	0.83 ^{ab}	-4.6

* Values sharing the same capital letter (s) within a column are not statistically different at the 0.05 level of confidence.

The bending strength properties of experimental panels are comparatively given in Table 4. For dolomite-based panels, all experimental panels show -12.1% to -61.6% lower bending strength properties than control (PX₀: 12.75 MPa). However, the lowest MOR value of 4.89 MPa was found with sample of PX₅ which prepared with equal proportions of wood chips and dolomite (1:1, w/w). It is clear that increasing dolomite in board proportions lowering effects on bending strength of panels. For olivine-based panels, only sample of PY₁ which

produced 10% olivine and 90% wood chips (w/w) proportions, show higher MOR value (13.27 MPa) than control. Similar to dolomite-based panels, it appears to increasing olivine (> 10%) in board proportions have negative impact on MOR properties of boards. The lowest MOR value of 5.16 MPa was found with sample of PY₅, followed by 6.71 MPa with sample of PY₄, 8.71 MPa with sample of PY₃, and 9.63 MPa with sample of PY₂. These values are about -59.5-, -47.4-, -31.5-, and -24.5% lower than control sample, respectively.

When the Tables 3 and 4 carefully overviewed, the experimental findings confirm the lowering of the strength with two mineral adduct fractions to Calabrian pine wood chips in matrix structure. However, the strength development of the panels could be possible with formation of matrix together. The lowering of the strength may also be due to the less interlocking force constituted by a spatial network of the distributed hydrophilic wood and hydrophobic mineral adducts in the matrix, which negative impact on bonding and frictions, thus, preventing the displacement of the matrix system (Vasiliev and Morozov 2001). It is hypothesized that wood chip inclusion may also lead to the reduction of potential failure plane, reduction in ductility, cohesion, energy absorption capacity, and impact resistance of the composite.

Table 4

The bending strength (MOR) properties of experimental panels

Board Code	MOR (MPa)	Differences (%) from control
Dolomite based panels		
PX ₀	12.75 ^e	-
PX ₁	11.21 ^d	-12.1
PX ₂	10.56 ^d	-17.2
PX ₃	8.18 ^c	-35.8
PX ₄	6.41 ^b	-49.7
PX ₅	4.89 ^a	-61.6
Olivine based panels		
PY ₀	12.75 ^d	-
PY ₁	13.27 ^d	4.1
PY ₂	9.63 ^c	-24.5
PY ₃	8.71 ^c	-31.7
PY ₄	6.71 ^b	-47.4
PY ₅	5.16 ^a	-59.5

* Values sharing the same capital letter (s) within a column are not statistically different at the 0.05 level of confidence.

The elasticity properties (MOE) of experimental panels are comparatively given in Table 5. Only sample of PY₁ show 9.7% higher elasticity (2462.8 MPa) than control (PX₀; 2245.2 MPa) while rest of samples show lower MOE values than control, regardless of preparation conditions and type of mineral in formulations. The lowest MOE's of 731.8 MPa and 585.8 MPa were found with samples of PX₅ in dolomite based-, and PY₅ in olivine-based experimental panels, respectively. It appear that increasing dolomite and olivine (> 10%) in formulations negative impact on elasticity properties of panels.

The elasticity properties in the material upon the addition of olivine and dolomite can be hypothesized as follows; the mineral adducts could be attributed to the interface between wood chips, such interaction results in agglomeration and flocculation of the particles, making them coarser, brittle, and less plastic, hence facilitating frictional resistance of the matrix.

All the three measured strength values for olivine and dolomite formulated experimental panels are plotted in Figure 3. For IB properties (Fig. 3A), olivine had improving effects on PY₁, PY₃ and PY₄ panels. The highest IB values of 1.21 MPa observed with PY₄ panel which is the highest among all panels. However, dolomite-based panels usually had a lower IB values in all experimental conditions compared to olivine--based panels. It is notable that both MOR and MOE values (Fig. 3B and C) have show similar plot shape which both adducts have negative impact on the MOR and MOE properties of experimental boards.

Table 5

The Elasticity (MOE) properties of experimental panels

Board Code	MOE (MPa)	Differences (%) from control
Dolomite based panels		
PX ₀	2245.2 ^b	-
PX ₁	1760.1 ^a	-21.6
PX ₂	1429.9 ^a	-36.3
PX ₃	1099.8 ^a	-51.1
PX ₄	1025.6 ^a	-54.3
PX ₅	731.8 ^a	-67.4
Olivine based panels		
PY ₀	2245.2 ^c	-
PY ₁	2462.8 ^b	9.7
PY ₂	1271.2 ^{ab}	-43.4
PY ₃	1026.9 ^{ab}	-54.3
PY ₄	926.6 ^{ab}	-58.7
PY ₅	585.8 ^a	-73.9

* Values sharing the same capital letter (s) within a column are not statistically different at the 0.05 level of confidence.

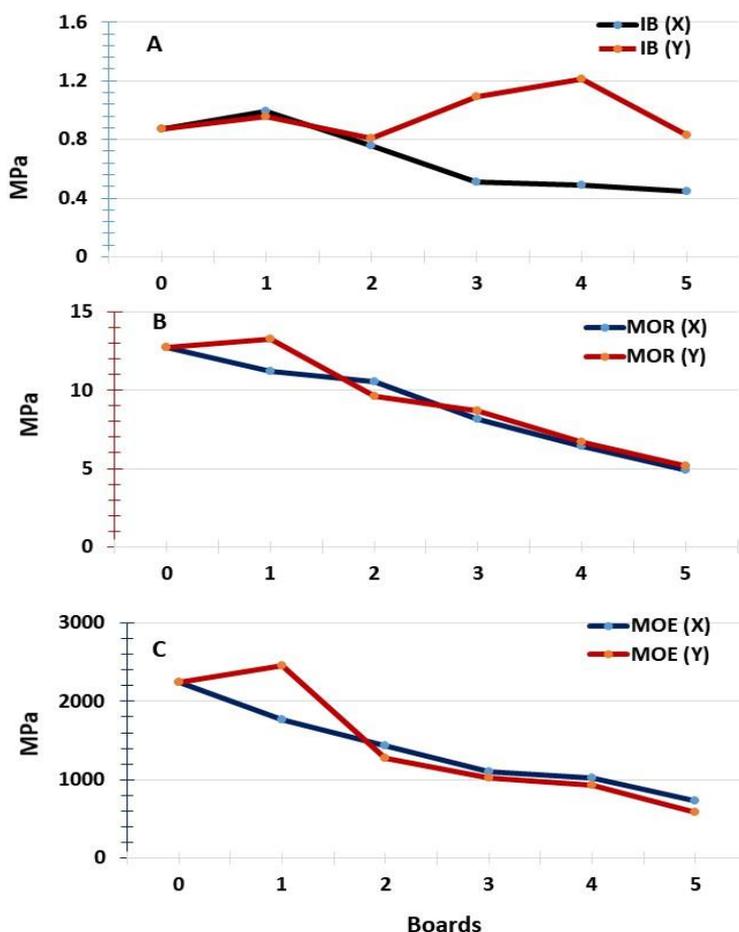


Figure 3. Strength properties of experimental boards

4. Conclusion

Natural mineral matters are receiving increased attention due to environmental protection and property development issues. However, there is a potential to utilize certain minerals renewable sources together in composite manufacturing due to their low cost, non-abrasive natures, and the abundant availability in

worldwide. The experimental findings obtained from this study could be divided into two parts. The first part considers the water interactions of composite panels and the second evaluates the effects two mineral adducts on strength properties. The strength development of composite materials are very complex and include some phenomenal results. However, these property changes are measurable evidence of impart hydrophobic properties. Moreover, the results found for strength properties of experimental panels may be useful for similar studies while it is possible to improve some strength properties of experimental panels at certain conditions. There are numerous literature reports on wood-adducts interaction effects on composite properties. Further study is needed to study for mineral-wood interactions in matrix system.

Conflict of interest

The authors declare that they have received no funds and there is no conflict of interest.

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