



Research Article

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Enhancement of cured cement using environmental waste: particleboards incorporating nano slag

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Abstract: Industrial waste is constantly increasing, so measures must be taken to recycle it in more practical and environmentally friendly ways. The aim of this study was to investigate the suitability of sawdust wasted particles for the production of cement bonded particleboards, and to enhance their compatibility with cement by using physical pretreatment processes, addition of nano slag and accelerated carbonation curing. All tests were carried out with different manufacturing parameters: sawdust/cement: (20%, 30% and 40%), treated and untreated wood, CO₂ curing and the addition of nano slag. Experiments were performed to assess the mechanical properties of produced particleboards such as density (D), water absorption (WA), thickness swelling (TS), bending strength (BS), and microstructural properties of the cement particleboards. For treated sawdust, the outcomes demonstrated an enhancement in the mechanical and microstructural characteristics of produced cement bonded particleboards by using the accelerated CO₂ curing method for sawdust particleboards. According to Scanning Electron Microscopy (SEM) micrographs, part of the carbonate crystals was found to be diffused into the cell walls and cavities and were found to protrude from sawdust surfaces.

Keywords: wasted sawdust, carbonation, strength, nano slag, particleboards (CBPBs), Scanning Electron Microscopy (SEM), XRD, and ITZ

1 Introduction

Iraq produced annually huge amounts of wood wastes available in the form of agricultural and industrial residues. Estimates from 2005 on land used in Iraq indicated that 27% of lands are arable with permanent crops, and 73% are azalea, mountainous and aqueous [1]. If water is ignored, wood-cement particleboards contain 10 - 40% by weight of wood (45-80% by vol.) and 60 - 90% by weight of cementitious materials (20-55% by vol.) [2]. Almost all researches dealing with wood-cement composites contributed to justify the theory that wood inhibits the setting and hardening of cement. Sugars, tannins, and starches are among those compounds having an adverse effect on cement hydration [3].

Wood-cement composites combine the excellent properties of two very important construction materials from which they are made. They are well known for their good resistance to fire, water and decay attack. They have good strength and stiffness but they are still machinable, being easily worked with sawing, drilling, nailing, etc. They have high impact resistance and superior sound and heat insulation properties. They are environmentally friendly products because they do not contain and generate any hazardous materials during processing and final use. They are also, light in weight and low in cost [4]. Perhaps in future, cutting-edge engineering techniques [5–9] will contribute to the development of new features and applications for particleboards. Smaller amounts (0.6 - 0.8)% of Arenga Pinata fibers showed higher flexural toughness by 120% as compared to those of higher volumes [10]. Nadir Ayrimis *et al.* [11] indicated that the boards had higher bending strength properties when the cement/wood ratio was increased from 0.75 to 1.5. Alireza Ashori *et al.* [12] reported that an increase of the cement/wood ratios above 2.0 had reduced the bending stiffness of wood excelsior particleboards.

Sustainability has become an increasingly relevant topic since the rise of the industrial revolution in the ninetieth century. The accelerated hardening process with

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carbon dioxide revolutionized the manufacture of wood-cement composites [13]. The usage of carbonation as an accelerated curing method in cement based composites has been tolerated as a CO₂ sequestration technique that improve the quality of final product [14]. Accelerated curing with CO₂ of the cement matrix can be engaged to enhance the long term strength and durability of the composites with sawdust particles, since it makes the alkalinity of the matrix lower, and making it less hostile to the wood particles.

In wood-cement composites, the compatibility between wood and cement is a major concern. The “compatibility” refers to the degree by which the wood fragments can affect the setting time of cement after mixing with water. If the presence of wood does not disturb the hardening process of cement then the wood is considered compatible with cement [15]. Wood species are not compatible with cement because soluble carbohydrates and certain extracts in the wood inhibit the setting and hardening of cement [16]. Ordinarily, sugars, acids, hemicellulose, etc., appear to significantly affect cement hydration, while lignin appears to have minimal effect on cement hydration [17]. Three methods were adopted in the literature to improve the wood-cement compatibility, included injection of CO₂ gas (carbonation), pretreatment of wood, or counteracting the retarding effect of wood by accelerating the hydration process of cement [18].

The carbonation of wood based cementitious products, offers excellent opportunities to achieve good wood-cement compatibility. The hardening of cement is so quick that those inhibitory ingredients in wood less affect the hydration of cement [19]. Treatment of wood with hot water and/or chemicals before mixing is another option. In fact, this has become the most often used method to mitigate the deleterious effects of wood on the hydration of cement [20]. Using of some supplementary cementitious materials is also considered as a good option to enhance the wood-cement compatibility. Ping Duan *et al.* [21] found that the addition of silica fume or rice husk ash can improve wood-cement compatibility, with higher strength for the composites. Arkamitra Kar *et al.* [22] reported higher physical and mechanical features of particleboards with the addition of fly ash.

According to the above literature, the major parameter related to the wood-cement composite is the compatibility. So, in this paper, with the aim of developing eco-friendly sawdust-cement composites that could exhibit lower weight, cost with higher productivity, we analyze the influence of the curing conditions in CO₂ gas and the addition of nano slag on the hydration degree of Portland cement composites incorporate different percentages of saw-

dust. Besides, the addition of nano materials was not discussed extensively in the wood-cement composite. According to Givi *et al.* [23], the presence of nano particles can promote the hydration process of cement as its acting as a nucleation sites as well as the nano particles can increase the strength of the composite by filling the cement pores. Thus, the nucleation effect of nano slag together with its pozzolanic activity may hinder the incompatibility between wood and cement. Furthermore, most of the previous studies discussed the strength property and as obvious it was increased with the increasing of CO₂ concentration and curing time. However, this could be a problem in lower productivity. Also, there is a lack of information about the microstructure of wood cement composites as well as linking the strength results with its characteristics like thickness swelling. Therefore, the main objective of this study is to produce sawdust-cement particleboards using cheap and sustainable ways. The other important objective, the emissions of carbon dioxide gas to the atmosphere were reduced and the compatibility between sawdust and cement was enhanced.

2 Materials and methods

2.1 Materials characteristics

The basic raw materials used consisted of type I cement, micro silica powder, natural fine aggregate, tap and distilled water, super plasticizer, nano slag in addition to sawdust particles. Ordinary Portland cement, conforming to I.Q.S 5/1984, was used to produce the particleboards. The chemical analysis of micro silica (produced by BASF Company) and nano slag is displayed in Table 1. Micro silica is used as pozzolanic admixture associated with 135% accelerated pozzolanic strength activity index. The results showed that micro silica and nano slag used in this study conform to the requirements of ASTM C-1240-05 and ASTM C-618 specifications. A high quality third generation of High Range Water Reducer (Superplasticizer) with a density of 1.095 g/cm³ was used in the mixtures (Sika ViscoCrete 5930). According to ASTM C494-03, this superplasticizer is classified as type G.

Sawdust particles were brought from local wood factory in Iraq. A hummer mill used to make the particles, and then they were screened to a final 4 mm mesh as shown in Figure 1. Table 3 shows the physical properties of sawdust particles. Physical particles pretreatment (hornification) aimed to remove extractives that inhibit cement reaction. Heating up the sawdust in water saturated with 20%

Table 1: Chemical composition of micro silica and nano slag*.

Constituent	Micro Silica (%)	Nano Slag (%)	Limits of ASTM C-618/05
CaO	1.27	1.16	
SiO ₂	89.36	67.95	≥ 70%
Al ₂ O ₃	0.81	20.75	
Fe ₂ O ₃	0.52	3.50	
SO ₃	1.04	0.01	≤ 5
NaOH+KOH	1.33	0.50	
Loss on Ignition	4.7	---	≤ 6%
Fineness	≥ 15000 cm ² /gm	= 63 nm*	

* The average particle size according to manufacture using AFM test.

Table 2: Sawdust and sand particle grading.

Type	Sieve size (mm)						
	10	4.75	2.36	1.18	0.60	0.30	0.15
Sand	100	92	80	54	42	13	5
Sawdust	100	79	38	13	5	4	1

* Tests were made by the Concrete Laboratory in Karbala Technical Institute.

Table 3: Physical properties of sawdust particles.

Property	Test according to ASTM D 4442 – 03 and ASTM D 2395 – 07a
Dry density (kg/m ³)	627
Moisture content (%)	62
Specific gravity Water	0.65
absorption (%)	189

* Tests were made by the Concrete Laboratory in Karbala Technical Institute.

Ca(OH)₂ for 60 minutes will ensure the removal of poisoning extractives [24]. The particle grading of sawdust and sand used in this study is presented in Table 2. Natural sand with 4.75 mm maximum size was used as a fine aggregate in this research as a quartz based sand that is complying with I.Q.S No.45/84, zone 3.

2.2 Experimental procedure

In terms of the Japanese Industrial Standard for Particleboards (JIS A 5908: 2003), the following equations are used to calculate the raw materials needed to make a



(a)



(b)

Figure 1: (a) Hammer mill for sawdust manufacture and (b) Sawdust pretreatment.

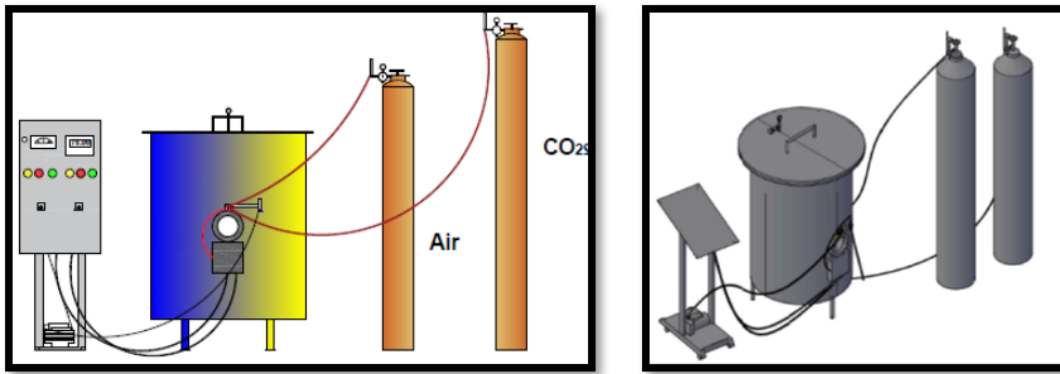


Figure 2: CO₂-curing experimental set-up.

board:

$$G = \frac{DV\delta}{(1 + B + R)} \quad (1)$$

$$P = GB(1 + M) \quad (2)$$

where:

G = mass of cementitious materials used to make a particleboard;

P = mass of sawdust used to make a particleboard;

D = target panel density ($D=1400 \text{ kg/m}^3$);

V = target panel volume (m^3);

δ = loss coefficient of raw materials during production ($\delta = 1.1$);

B = sawdust to cement ratio by mass (%);

M = moisture content of sawdust particles (%);

R = water cement ratio (%).

In this study, cement bonded particleboards made from treated and untreated sawdust particles with a density of 1400 kg/m^3 were employed and tested at 28-day following the ASTM C1185-12. In light of past investigations, the experimental factors are: sawdust/cement: (20%, 30% and 40%), treated and untreated wood, CO₂ curing and the addition of nano slag with constant water to cement ratio of 30%, 1% SP by weight of cement, 0.2% addition of nano slag by weight of cement and 20% replacement of microsilia by weight of cement. All the specimens, which have dimensions of (305*152*20) mm, were kept inside their molds underneath wet burlap covered with a plastic sheet for 24 hrs. Then, the particleboards were carefully removed from the molds, dried at $105 \pm 2 \text{ }^\circ\text{C}$ for 30 min. and cured for the specified technique. This drying process will ensure the availability of empty paths for CO₂ to take place. As it can be seen in Figure 2, the samples in the curing chamber was treated with CO₂ gas at 6.9 MPa (1000 psi) at 55 °C for 2 hrs soaking time. In the specimen preparation of this section and the following ones, 20-minutes vacuum were applied before CO₂ injection, and the CO₂ flow

rate was 10 L/min. To ensure whether the accelerated carbonation took place or not, the textural study of the fractured surface for all specimens was performed on a (SEM Model: TESCAN-VEGA/USA) with tungsten source and detector X-Flasb 5030, which operates at a voltage of 1–20 kV with a range of between 10 and 80,000 magnification, at a work distance from 1 to 20 mm [25]. In addition, concrete samples from different mixtures were analyzed using X-ray diffractometer (XRD) analysis to characterize their reaction products. XRD is an attractive analytical technique for concrete research because of the speed and simplicity at which testing is conducted. Additionally, this technique is nondestructive and requires only a few grams of material for analysis, thus it is a powerful tool used in studying crystallographic structure. The horizontal scale of a typical XRD patterns gives the crystal lattice spacing, and the vertical scale gives the intensity of the diffracted ray.

3 Results and discussion

3.1 Effect of mechanical and microstructural properties

Analysis on the data gathered, by following the test methods outlined previously, provided some insightful information into the effects of different variables on the physical, mechanical, and microstructural properties of cement bonded particleboards. The results of this study, shown in Figures 3, 4, 5 and 6, demonstrated that there is a considerable difference exists between treated and untreated sawdust particleboards. The treated sawdust particleboards appeared to have higher bending strength and density than untreated particles. In addition, all treated sawdust particleboards have lower moisture absorption and thickness swelling than those of treated wood. For comparison,

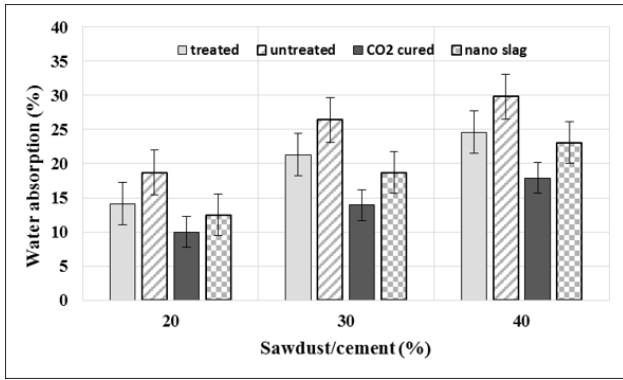


Figure 3: Water absorption of cement-bonded particleboard prior to and after different accelerated effects.

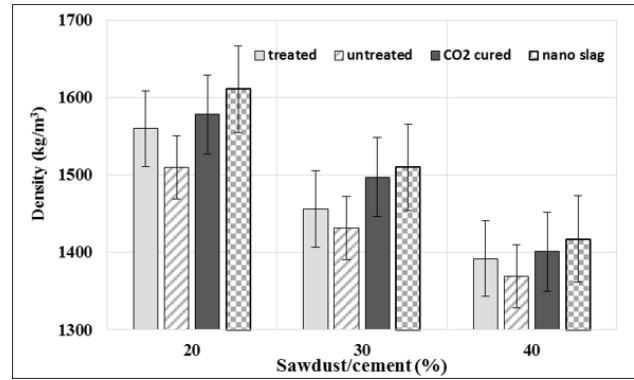


Figure 5: Density of cement-bonded particleboard prior to and after different accelerated effects.

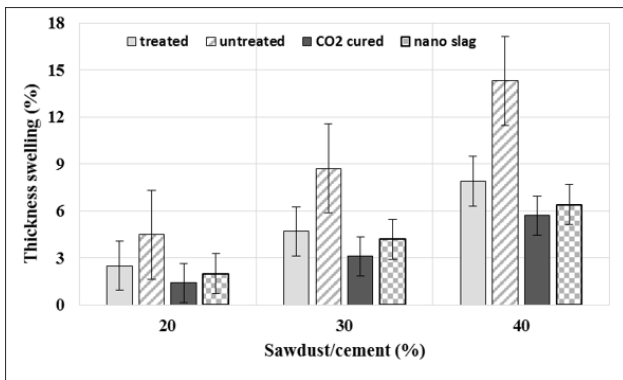


Figure 4: Thickness swelling of cement-bonded particleboard prior to and after different accelerated effects.

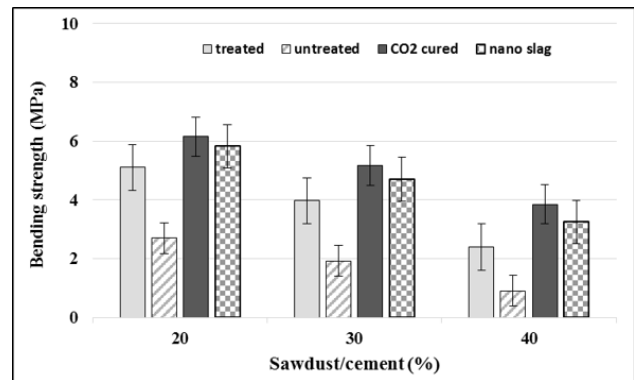


Figure 6: Bending strength of cement-bonded particleboard prior to and after different accelerated effects.

there is an increase in the bending strength and density for all CO₂-cured boards associated with lower moisture absorption and thickness swelling with respect to particleboards without CO₂ curing. In other words, samples cured by CO₂ gas were stronger than those made conventionally after 28-day due to lower spring back action by sawdust to boards after the completion of the water submerged.

Figures 3 and 4 represent the relationship between moisture absorption, thickness swelling and sawdust to cement ratio at various treatment conditions. After 24 hours of soaking in water, it is obvious that an increase in the sawdust /cement ratio, the moisture absorption and thickness swelling of all boards increased up to 66% and 210% respectively. This is due to increased amounts of porous wood particles in the board. Moreover, treatment of sawdust, carbonation and the addition of nano slag all have beneficial effect on reducing the moisture absorption and thickness swelling up to 20%, 43% and 24% respectively as compared to untreated sawdust particleboards. However, the filling effect of nano slag besides its pozzolanic reaction, and the removal of extractable substances could be the main reasons for these improvements. This trend

is comparable to that found by Hassan *et al.* [26] which they recorded an improvement in the physical and microstructural properties of cellulosic fiber-cement composites by using accelerated CO₂ curing method. They concluded that CaCO₃ is precipitated in the pore structure of the matrix, filling the voids and thereby blocking the intake of water due to the decrease of pore size. Petrification of fibers and densification interface zones caused tendencies toward increased strength and embrittlement.

The particleboards with higher density did not bring about higher values of bending strength as shown in Figures 5 and 6 respectively. This could be attributed to the fact that improving the bond between the sawdust particles and the matrix (because of CO₂ curing) resulted in an improvement in the interfacial transition zone as the SEM micrographs proved later, and the petrification of sawdust particles due to carbonation curing. This would make the ITZ thicker and stronger and, thus increasing the bonding forces between matrix and sawdust particles. As Figures 5 and 6 show, the boards made from treated particles had higher density and bending strength values than those made from untreated particles. Moreover, an increase in

Table 4: Parameters sensitivity analysis as compared to untreated specimens.

Mix	BS			TS			D			WA		
	treated	NS	CO ₂	treated	NS	CO ₂	treated	NS	CO ₂	treated	NS	CO ₂
M-20	+84	+158	+223	-44	-55	-67	-3	-0.6	-2	-39	-36	-33
M-30	+100	+171	+292	-46	-40	-64	-5	-2	-3	-33	-30	-28
M-40	+162	+225	+318	-47	-41	-60	-6	-4	-5	-31	-26	-25

+: increment,

-: reduction

the sawdust/cement from 20% to 30% and 40% decreases the density and bending strength up to (6%, 11%), and (17%, 48%) respectively. While, the rates of increase in density and bending strength after treatment of sawdust, carbonation curing and addition of nano slag were of (4%, 5% and 7%), (100%, 134% and 126%) respectively. This estimation is supported by V. D. Pizzol *et al.* findings [17] who found that the accelerated carbonation curing decreased the apparent porosity and increased the bulk density of the hybrid fiber-cement composite (fiber cement boards reinforced with cellulose pulp and synthetic fibers).

Regarding all parameters considered in this study which presented in Table 4, it can be concluded that the sawdust pretreatment, carbonation curing of boards and the addition of nano slag were beneficial for improving the compatibility of the sawdust particles with cement and thus enhance the final product strength, reduce the water absorption and thickness swelling.

3.2 Microstructure of cement particleboards

Scanning Electron Microscopy (SEM) method was employed to characterize the microstructure of the studied boards. Figure 7 views SEM micrographs of sawdust particleboards fractured surface prior to and after different accelerated effects. Five representative images for each sample were undertaken and only one SEM micrograph was chosen from them. It seems clear that the surface of sawdust particle is covered with Ca(OH)₂ (arrow 1/a). Also, it is obvious that after sawdust pretreatment, there is a good adhesion force between sawdust particles and cement paste so cracks appear far away from ITZ (arrow 2/a). Meanwhile, the cross section of sawdust particle used here, is composed of empty black holes (arrows 3/b), so the fracture crack at failure initiate within ITZ and through sawdust particles. However, these black holes are empty for non-carbonated samples, and completely filled with CaCO₃ for carbonated boards. (Arrow 4/c) shows plate like of CaCO₃ formed in the fiber pores, on and around the particle surface after CO₂ curing. Therefore, the carbon-

ated cement matrix is denser than control mix that can enhance the interfacial transition zone between cementitious matrix and particles, and certainly promote the bending strength of the end composite. In addition, from these micrographs it can be noticed that the cement matrix is denser and compact as shown in (arrow 5/d) after nano slag addition, improving the contact between sawdust and cement matrix, and possibly favoring the better adhesion between them, so reducing the thickness of ITZ and increasing of board density. The results were in accordance with prior findings conducted by Hosseinpourpia *et al.* [27]. They found that the presence of nano silica particles led to an improvement of the cement paste microstructure, as well as to proper coupling between the fiber and hydration products. Therefore, the bending strength of the mixture with silica nano-particles was higher than that of other pastes. It seems clear that due to the smooth, dense surfaces of the nano slag particles, the cement absorbed little if any water during initial mixing. Their results indicate a better particle dispersion and higher fluidity of the pastes and mortars. Pores in concrete that normally contain calcium hydroxide are, in part, filled with calcium silicate hydrates resulting from the hydration of the slag cement. This improve the packing of the solid materials by occupying some of the spaces between the cement grains in the same way as cement occupies some of the spaces between the fine-aggregate particles.

The results of the X-ray diffraction spectra presented in Figure 8 include data from four representative mixtures from the testing matrix untreated, treated, carbonated and with nano slag. According to Figure 8, a major peak hump (CaCO₃ and CSH) was observed in the diffraction pattern between 2θ values of 13°, 25° and 30° for all the investigated mixes. Meanwhile, a minor peaks (Ca(OH)₂ and quartz Qz) were noticed in the diffraction pattern between 2θ values of 34°, 43° and 52°. This could be due to the presence of amorphous glassy materials after increasing the activity of cement (for treated sawdust) or for the carbonated crystals (due to carbonation curing) or, because of the pozzolanic activity (associated with nano slag ad-

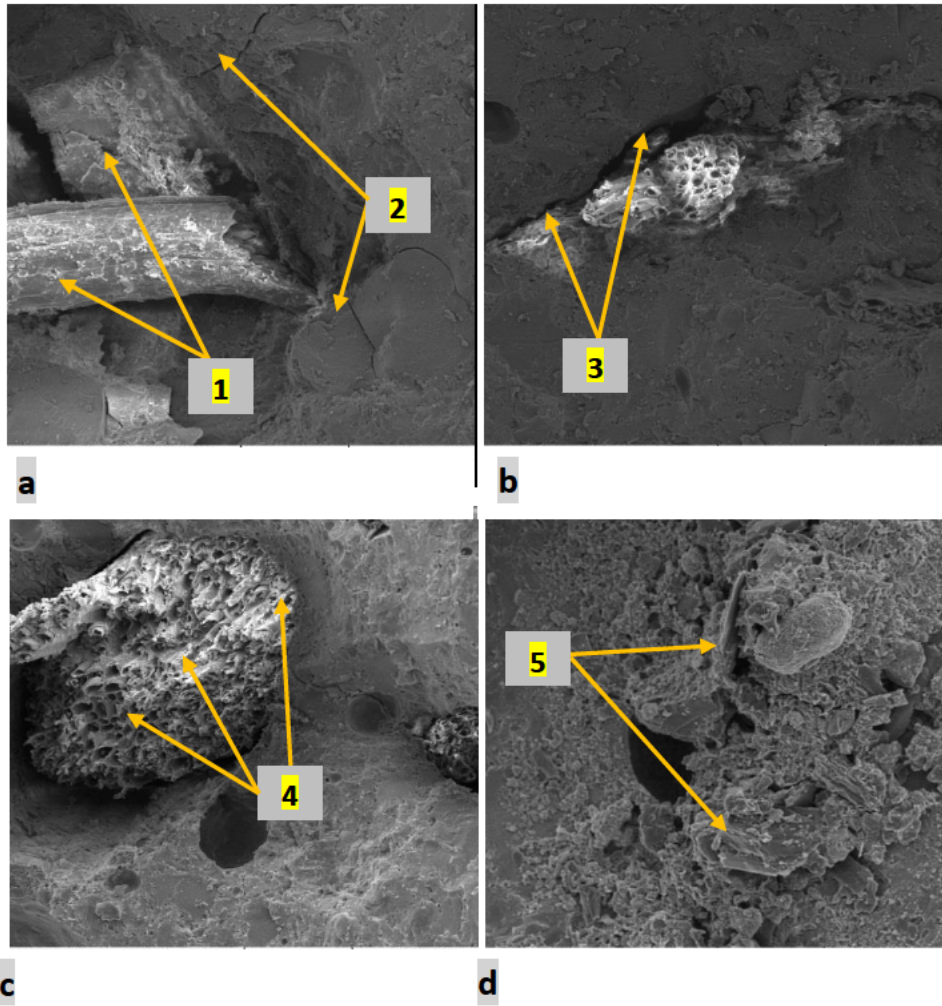


Figure 7: SEM micrographs of fractured surface of: (a) treated, (b) untreated, (c) CO₂ cured and (d) nano slag addition.

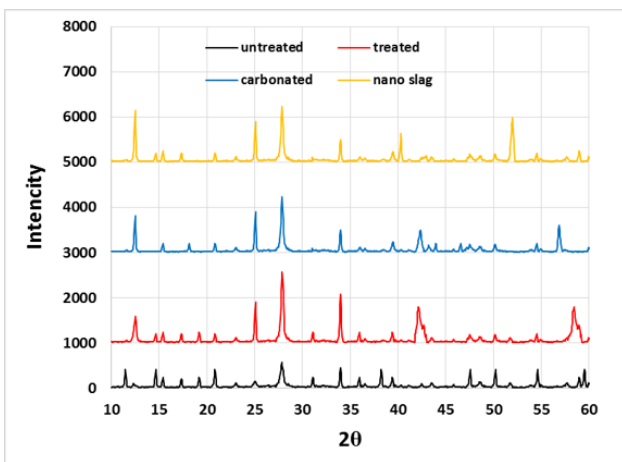


Figure 8: X-ray diffractions for: (a: untreated), (b: treated), (c: carbonated) and (d: with nano slag).

dition). The crystallinity for particleboards was found to be lower in the untreated samples than in the samples of treated, carbonated and with nano slag addition. These humps proved the creation of aluminosilicate hydrate gel C-A-S-H that has been described as the main reaction product of hydration process in the diffraction patterns of concrete. However, the results are compatible with those of obtained by Ping Duan *et al.* [21] who studied the ITZ and pore structure of concrete incorporated mineral admixtures (silica fume, metakaolin and slag). It was found in that study that the addition of these materials led to densify the ITZ, optimized the pore structure and the pore size distribution became more reasonable. Moreover, the micro filling and the pozzolanic reaction of silica fume, metakaolin and slag enhanced the microstructure. Additionally, as a result of the pozzolanic reaction, calcium aluminosilicate hydrates and calcium silicate hydrates were produced.

4 Conclusions

Based on the analysis of data compiled throughout this study, these conclusions are summarized as follows:

- (a) Treated sawdust particleboards had higher values of bending strength, density and lower water absorption and thickness swelling than those of untreated, which demonstrates that treated sawdust is more compatible with Portland cement. The rates of increase in density and bending strength after treatment of sawdust, carbonation curing and addition of nano slag were of (4%, 5% and 7%), (100%, 134% and 126%) respectively.
- (b) Carbonation curing seems to have yielded better matrix and particleboards qualities.
- (c) Part of the carbonate crystals was found diffused into the sawdust cell walls and cavities and were found to protrude from surfaces.
- (d) Petrification of sawdust and densification of interfacial transition zone enhanced the final strength. Petrified sawdust with higher bonding tend to rupture rather than pullout at fracture surfaces.
- (e) Increasing the sawdust to cement ratio from 20% to 30% and 40% decreases the density and bending strength up to (6%, 11%), and (17%, 48%) respectively of produced particleboards, meanwhile, it make the moisture absorption and thickness swelling more pronounced.
- (f) The addition of nano slag all have beneficial effect on physical, mechanical and microstructural performance of particleboards. However, higher density did not necessarily associated with higher values of bending strength.
- (g) The X-ray diffraction spectra proved that the carbonation led to increase CaCO_3 contents during the curing process. The CSH peaks in XRD spectra becomes more evident after sawdust treatment. Carbonation crystals appeared after carbonation curing. Stable CSH and Qz are the higher peaks after nano slag addition.
- (h) SEM images have shown that the petrification of sawdust particles due to carbonation curing would make the ITZ thicker and stronger and, thus increasing the bonding forces between matrix and sawdust particles.
- (i) The crystallinity for particleboards was found to be lower in the untreated samples than in samples of treated, carbonated and with nano slag addition.

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