Manufacturing of sustainable cellulose date palm fiber reinforced cementitious boards in Iraq

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Abstract

The present work investigates the suitability of utilizing date palm residues in manufacturing wood-based cementitious boards. It also concerns other environment issues like trying to consumption the pollutant carbon dioxides in boards manufacturing process as an accelerated curing method. Two categories of date palm cellulose fiber cement boards were produced and evaluated, (8% and 5% cellulose fiber content by weight). Comparisons were made between the flexural strengths, stiffness and toughness of the produced boards which fabricated with conventional and different concentrations of CO_2 curing (i.e. 0%, 30%, and 100%). This paper is an attempt to fabricate sustainable products- preferably environmentally friendly- that incorporate agriculture waste in Iraq. Analysis results yielded that higher concentration (100%) have significant effects on the performance of the produced boards, particularly in lower fiber/matrix ratio (5%). Lower CO_2 concentration; however, were generally comparable to those obtained at 0% concentration (conventional curing). SEM images confirm the matrix densification effect due to CO_2 curing.

Keywords: sustainability; accelerated CO_2 curing; cellulose fibres; cement composites; flexural strength; date palm.

تصنيع الواح سمنتية مستدامة معززة بالياف نخيل التمر السيليلوزية في العراق

الخلاصة

تحرت هذه الدراسة عن امكانية الاستفادة من مخلفات نخيل التمور في صناعة الواح سمنتية- خشبية مركبة. كما ركزت على شؤون بيئية اخرى مثل محاولة استهلاك الملوث ثاني اوكسيد الكاربون في تسريع معالجة الالواح السمنتية اثناء عملية التصنيع. تم انتاج وتقييم نوعين من الالواح المعززة بالياف النخيل السيليلوزية (8% و 5% محتوى الالياف وزنا). قورنت نتائج فحص الانثناء، الصلابة، و المتانة للالواح المصنعة والمعالجة تقليديا مع

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الالواح المعالجة بغاز ثاني اوكسيد الكاربون وبتراكيز مختلفة (0%، 30%، و 100%). تشكل هذه الدراسة محاولة لصناعة الواح سمنتية مستدامة صديقة للبيئة تستفيد من المخلفات الزراعية المتوفرة في العراق. بينت نتائج التحليل ان التركيز العالي 100% قد حسن بشكل واضح اداء الالواح المنتجة وخاصة لنسبة الالياف المنخفضة 5%. كما ان التركيز المنخفض لغاز ثاني اوكسيد الكاربون لم يؤدي الى تاثير يذكر على الاداء. بينت نتائج تحليل الاشعة السينية ان المعالجة بغاز ثاني اوكسيد الكاربون قد ادت الى زيادة محتوى كاربونات الكالسيوم فيما اكدت صور المجهر الماسح الالكتروني الزيادة في كثافة المادة نتيجة هذه المعالجة.

INTRODUCTION

For a long time, Iraq is best known firstly for producing and exporting oil; and secondly for farming and agriculture production. Agriculture residues consist more than 50% of the farming products however, limited efforts were undertaken to utilize these residues in the industry for economical or environmental purposes. Wood fibers are considered important due to their availability in these farming wastes. Cementitious materials are known to be week in tension strengths, and the presence of fibers may help to enhance their post cracking behavior including toughness and cracking resistance [1-5].

According to formal reports from the Iraqi Ministries, (i.e. Ministries of Agriculture and Planning), the number of date palm trees currently exceeds 16 million and may reached higher number in the near future [6,7]. A large quantity of this date palm population sheds huge quantity of plant biomass annually from seasonal pruning as an essentially agricultural practice [8] or simply due to the end of their life and death. El-Juhany [9] mentioned that annually about 35-kg average of palm residues are obtained per tree. In developed countries a large quantity of these residues is utilized in the industry such as wood-based cement composites and light or medium weight cellulose fiber cementitious boards. In Iraq and perhaps the whole Middle East, however, they are burnt. Such large amount of date palms in Iraq make the use of its residues as a new source for manufacturing of building materials purposes a promising investigation.
Furthermore, the availability of this kind of vegetable fibers promote more investigations to be used as an alternative reinforcing fibers to asbestos especially in the present of health restrictions.

Manufacturing process of wood-based cement board may includes heat curing combined with pressing steps of the fresh mixes in the board molds. Such long time procedure may lead to increase initial costs and reduce production rates. However, these processing steps are considered essential to prevent swelling back to the original thickness after pressure release [10].

Compatibility between vegetable fibers and Portland cement matrix

Implementation of vegetable particles and fibers in cement based composites are growing in importance. Several aspects should be taken into account to facilitate producing functionally successful and durable composites. The main drawback in the utilization of these vegetable/cellulose fibers is their possible degradation in the Portland cement matrix due to its high alkalinity (pH ranged between 12 to13). The hydroxide ions resulted from the hydration reactions between cement particles and water may penetrate into the fiber lumen leading to the creating of ettringite and monosulphate inside the fiber and then negatively influencing cellulose fibers strength [11, 12]. Setting of cement is another property influences greatly by adding woods to cementitious matrix. It is well known that hemicellulose inhibits the setting in cement. Sandermann et al [13] found starches; sugars, tannins, and certain phenol have an inhibitory effect. Wood contains abundance quantity of carbohydrates and phenolic compounds which have detrimental influence on the set and strength of wood based composites. The water soluble materials in wood have the greatest inhibitory effect [13, 14]. Wood species, logging season, and sampling location within the tree are also another factors influence the hydration behavior of cement matrix. Hardwoods (i.e palm fibers) are generally having lower effects on the cement hydration process than softwoods. Other researchers (15, 16), mentioned that spring cutting wood delayed hydration progress of cement particles probably due to the presence of water-soluble extractives in large quantity compared with other seasons.

Cement hydration is a complex process due to the various chemical and physical changes in the resulted hardening composite and the several possible factors affected it. Adding vegetables fibers make this process more complicated. Such incompatibility and set inhibitory effects can be overcome by partial or complete removal of extractable form wood fibers before ingredients are mixing and composites manufacturing, which may help improve the mechanical properties and long term serviceability of the final cement based composites. Accordingly, one or more of the following measures can be taken to overcome this incompatibility problem:

1- Storage the raw materials for 3 to 4 months in storage yards to reduce the concentration of free sugar and other carbohydrates (17, 18).
2- Increase cement hydration speed by using accelerated agents such as calcium chloride, aluminum sulfate and sodium silicate (19, 20).
3- Immersion of wood in hot water before mixing (21, 18, 22).
4- Adding pozolanic materials such as silica fume and fly ash (23, 24).
5- Accelerated hardening of wood-based cement composites, for example by carbon dioxide curing (25) or injection (26).

The aim of this study is to develop an efficient approach to processing cellulose fiber-reinforced cement composites, which makes value-added use of carbon dioxide and/ or agriculture waste materials. The performance characteristics were evaluated through flexural testing of composites and different processing aspects were implemented.

Experimental program

Materials and manufacturing procedures

In this study, date palm cellulose fiber was used, (**Fig. 1),** with an average length of 4.0 mm. Ordinary Portland cement conforms to IQS 5/1984, was used in the mixtures of this investigation; its physical properties and chemical composition are shown in **Table 1**. The matrix mix proportions and fiber mass fraction used are shown in **Table 2**. Silica sand brought from western desert in Iraq was used in this study. It consists of 98% of SiO_2 and has a one uniformed sieve analysis (i.e. passing 1.18 mm and return on 0.3 mm). The manufacturing process of a cementitious thin-sheet reinforced with cellulose fiber was similar to that used by Soroushian et al [27, 28]. It involved mixing of the constituents in a mortar mixer, and placing the blend into a 300 mm by 152.5 mm (12 in. by 6 in.) rectangular wooden mould (made of plywood). Cellulose fiber/cement weight ratio of 0.05 or 0.08, and water/ cement weight ratio of 0.27 were used to produce 10 mm thick boards. The mould was first painted with oil to prevent any possible adhesion with hardened matrix, the mix was then spread in the mould and carefully leveled with appropriate tool, and was then covered by nylon sheet to keep it in moist condition. After 32 hrs, the wooden mould was removed and specimens were now ready for curing. **Fig. 2** shows the cement-bonded cellulose fiberboard (CBCB) processing system for CO_2 curing. Different concentrations of CO_2 gas in air, as seen in **Fig. 2**, were produced by using two gas cylinders (one CO_2 and the other air). Each one was connected to a flow meter which controlled the gas flow level and thus the CO_2 concentration.

Table (1) Properties of cement and silica fume.

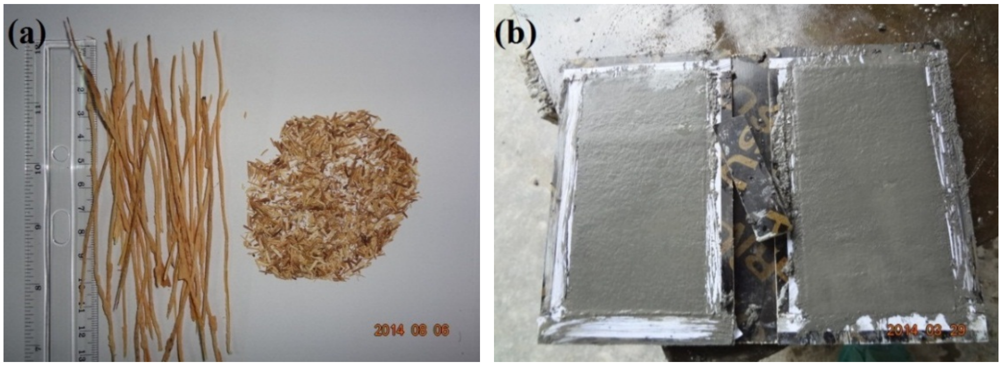
Composition	Cement (%)	Silica fume (%)
Chemical compositions		
SiO_2	21.7	90.65
Al_2O_3	4.61	0.02
Fe_2O_3	3.35	0.01
CaO	61.89	1.22
MgO	3.05	0.01
SO_3	2.4	0.24
Lime saturation factor	0.87	-
Insoluble material	0.6	-
Loss on ignition	2.16	2.86
Physical properties		
Bulk density(kg/ m^3)	1180	500
Specific surface (cm^2/g)	391	20000
Compressive strength of Mortar: 3-days (MPa) 7-days (MPa)	23 37	

Table (2).The Composition of Cellulose Fiber Reinforced Cement Composite

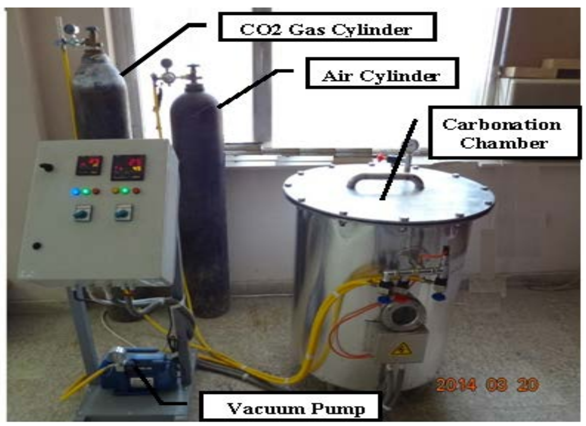
Fiber Type	Softwood date palm fiber
Fiber Mass Fraction (%)	5 or 8
Sand-Binder ratio (by weight)	0.75
Superplasticizer (% by weight of cement)	1%
Silica Fume-Cement ratio (by weight)	0.75

*According to previous studies [27, 28]

After the completion of processing and then wooden mould removal, curing was started firstly by a pre-curing oven-drying for young sheet prior to CO_2 curing for a half hour duration. This step is essential to lower moisture contain of board to the point where CO_2 penetration and reaction would be facilitated [29]. Typical appearance of the resulting cellulose fiber cement boards is shown in **Fig. 3**. The set-up of carbonation system is capable of applying any combination of CO_2, air and vacuum on the board. Three different carbon dioxide (CO_2) gas concentrations: 0%, 30%, or 100%, were used for duration of 2 days inside the chamber for each board.



Figure(1). Appearance of cellulose date palm fibers (a) and produced boards (b).



Figure(2). Processing system incorporating CO_2 curing.

Figure(3). Typical appearance of cement-bonded cellulose fiberboard (CBCB).

Specimens and test procedures

Flexural tests were performed according to the ASTM C 1185-12 [30]. A minimum of three replicated specimens were tested for each condition for all mix designs considered. The flexural test samples have a clear span of 254 mm (10 in.), a width of 152.4 mm, and a thickness 10 mm. **Fig. 4** shows the one point flexural test set-up used for cellulose fiber reinforced cement composites. A displacement rate of 0.5 mm/ min was used in flexure tests (which were conducted in a displacement-controlled mode). A computer-controlled data acquisition system was used to record the test data. The load–deflection curves were characterized by flexural strength, toughness (total area underneath the load–deflection curve), and initial stiffness (defined here as the stiffness obtained through linear regression analysis of the load–deflection points for loads below 15% of maximum load). The flexural performance was evaluated in wet condition.

Figure(4). Set-up of flexural test of the cement-bonded cellulose fiber-board (CBCB).

In this study, a full factorial experimental design was implemented, to investigate the effects of using CO_2-curing combined with two fiber/matrix ratios, on the flexural performance of the produced fiberboard composites.

Test results and discussion

Flexural performance behavior

Figures. 5 and 6 present typical flexural load–deflection curves of fiberboards subjected to different concentrations of CO_2-curing. The flexural strength, toughness and stiffness test results are presented in **Tables 3 to 5** and **Figs. 7a, b, and c,** respectively.

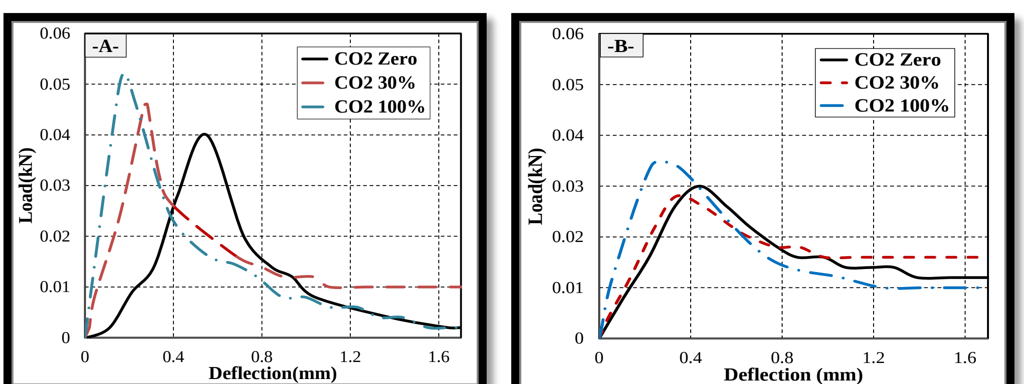
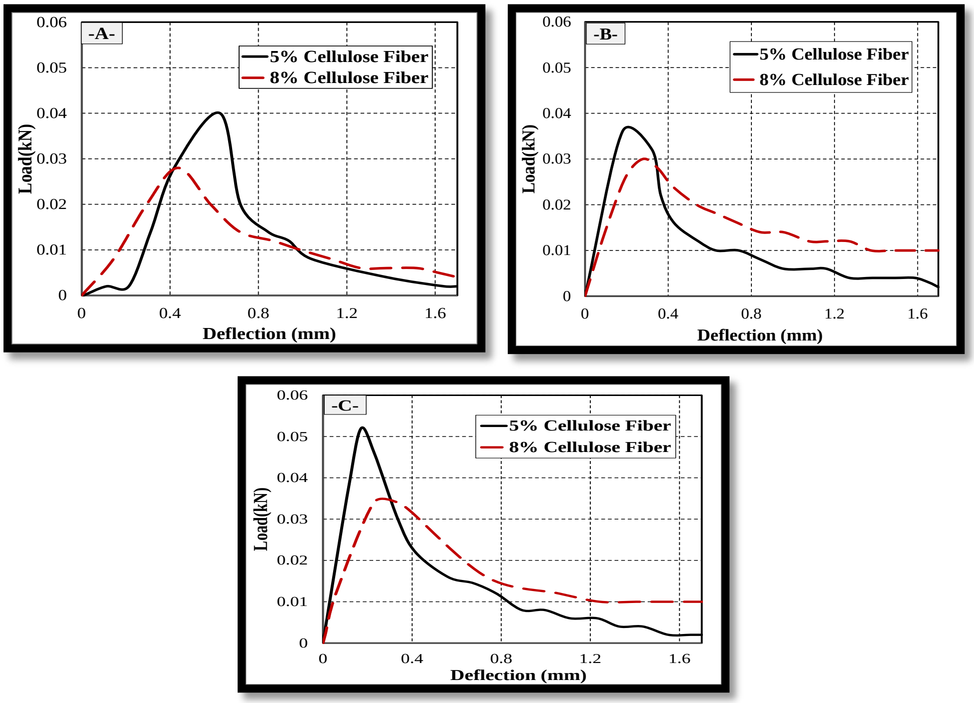


Figure (5).Typical Load deflection curve characteristics of cement-bonded cellulose fiberboard; (A) Cellulose fiber/matrix=0.05, (B) Cellulose fiber/matrix=0.08.



Figure(6).Effects of various CO_2 concentrations on the L-D curve characteristics of cement-bonded cellulose fiberboard; (A) 0% CO_2 concentration, (B) 30% CO_2 concentration, (C) 100% CO_2 concentration.

Table (3).Flexural performance of cellulose fiber reinforced cementations boards subjected to 0% CO_2-curing concentrations (28-day).

Mix No.	Flexural Strength (N)	Flexural Toughness (N-mm)	Initial Stiffness (N/mm)
Mix No. 1 (5%)	45	19.71	137.40
	40	20.05	132.50
	40	20	133.40
Mean	41.66	19.92	134.43
SD	2.88	0.18	2.60
Mix No. 2 (8%)	28	16.08	80
	30	15.69	73.30
	26	13.9	75
Mean	28	15.22	76.1
SD	2	1.166	3.48

In general, the flexural performance of CO_2 cured cement bonded cellulose fiberboard versus control specimen was improved for lower cellulose fiber content. A higher concentration of CO_2, 100%, is observed to yield better flexural performance characteristics compared to those obtained with 30% CO_2 concentration. The effect of high concentration seems to have the same effect on both cellulose fiber ratios 5% and

8%. Furthermore, all tested specimens behaved elastically up until the peak flexural strength (P_{max}). Beyond the P_{max} the initiated cracking exhibited instable growth leading to separation of the board into two parts. It is also noted that for both fiber/cement ratios, the recorded deflection associated with P_{max} continuous to increase while P_{max} decreases, when the concentration of CO_2-curing decreases. The post peak part of the load deflection curve drops down sharply in the case of higher values of P_{max} achieved by using 100% concentration of CO_2-curing, while for 30% and 0% concentrations it decreases slowly in a sequential order. **Fig. 5a** provides a good example for this explanation.

Table (4): Flexural performance of cellulose fiber reinforced cementations boards subjected to 30% CO_2-curing concentrations (28-day).

Mix No.	Flexural Strength (N)	Flexural Toughness (N-mm)	Initial Stiffness (N/mm)
Mix No. 1 (5%)	30	17.72	155.3
	46	17.02	200
	36	16.06	198
Mean	37.33	16.94	184.43
SD	8.08	0.83	25.25
Mix No. 2 (8%)	22	17.87	133.3
	30	12.55	72.7
	28	12.35	87.3
Mean	26.66	14.26	97.76
SD	4.16	3.13	31.62

Table (5).Flexural performance of cellulose fiber reinforced cementations boards subjected to 100% CO_2-curing concentrations (28-day).

Mix No.	**Flexural Strength (N)**	**Flexural Toughness (N-mm)**	**Initial Stiffness (N/mm)**
Mix No. 1 (5%)	60	41.47	226.3
	60	36.89	200
	80	23.02	227
Mean	66.66	33.79	217.76
SD	20.81	9.60	113.74
Mix No. 2 (8%)	32	4.93	147.4
	22	9.02	102
	34	15.43	129.4
Mean	29.33	9.79	126.26
SD	6.42	5.29	22.86

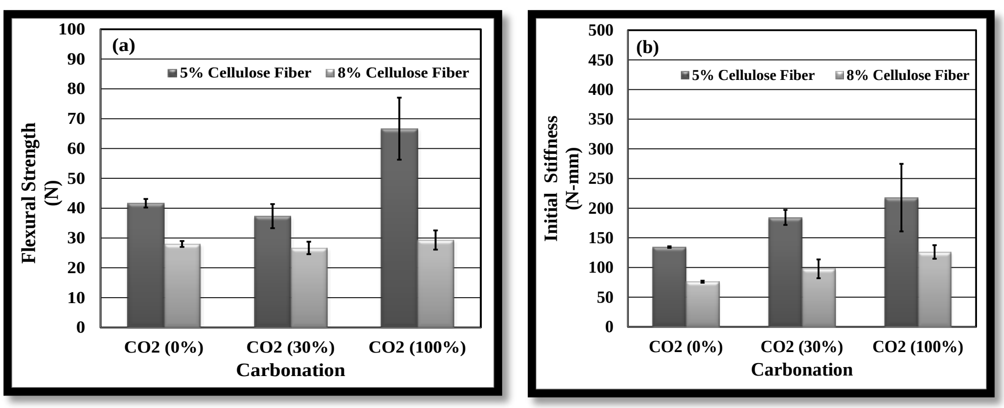
Table 6 shows the percentage differences in the flexural properties of the CO_2-cured composites versus those of the control boards (i.e. without CO_2-curing). CO_2-curing seems to have yielded better matrix and boards qualities. The improvements were more pronounced in lower fiber/matrix ratio. Any improvements in the flexural properties (i.e. flexural strength, toughness, and stiffness) will depend on whether fibers bridging the cracks are able to support the load previously carried by the matrix and whether the fibers break or pull out of the matrix [31]. Hannant [32] mentioned that improving the bond between the fiber and the matrix (as a result of CO_2 curing, particularly in the 5% cellulose fiber ratio used in this study) leads to an improvement in the contact area and frictional force at the interface. The strain in the composite at a given stress depends on the length of debonded fibers and, hence, a greater bond leads to raising the peak flexural force P_{max} and some fibers are expected to broken rather than pulled out only. This behavior probably interprets the enhancement in flexural properties associated with 100% CO_2 curing.

Table (6). Percentage difference of flexural performance of CO_2-cured boards versus control (0% CO_2).

	5% fiber content ratio		8% fiber content ratio	
	30 % CO_2	100 % CO_2	30 % CO_2	100 % CO_2
Flexural strength, %	-10.4	+60	-4.7	+4
Flexural toughness, %	-14.9	+72.2	-6	-35.6
Initial stiffness, %	+37.2	+62	+28.4	+65.1

In the case of initial stiffness **(Fig. 7b)**, CO_2 concentration factor had relatively significant effect on stiffness. The effect was more pronounced in lower cellulose fiber ratio. From practical point of view, the combined effects of higher CO_2 curing concentration and lower cellulose fiber seem to be of major practical significance, especially when higher stiffness and uncracked section are the main concern of the designer.

In the case of toughness **(Fig. 7c)**, 100% CO_2 concentration combined with lower cellulose fiber ratio have a definite improvement effect. Other effects and interactions between CO_2 curing concentration and cellulose fiber ratio in relation to toughness seem to be of minor practical significance.



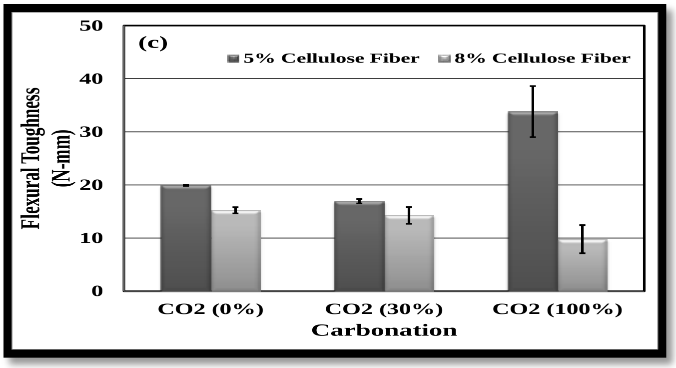


Figure (7). Effects of various CO_2 concentrations on the flexural performance of cement-bonded cellulose fiberboard, (a) Flexural strength, (b) initial stiffness, and (c) flexural toughness.

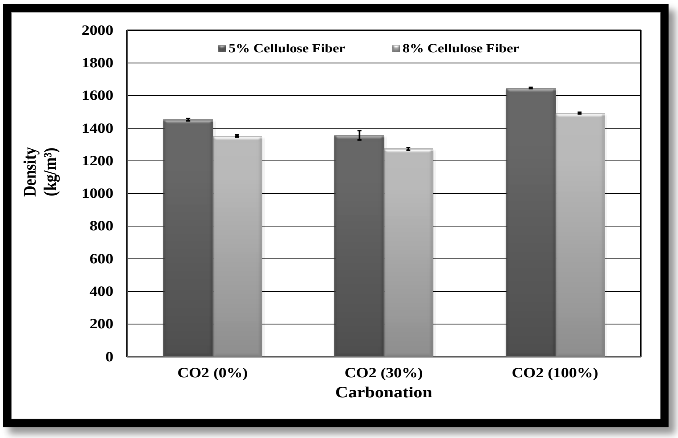
Block analysis of variance of the flexural test results (see **Table 7**), at 95% level of confidence, suggested that: cellulose fiber/matrix ratio (A), CO_2-curing concentration (B), and the interactive between the two factors (A×B), had statistically significant effects on the flexural strength of cement-bonded cellulose fiberboard. Cellulose fiber/matrix ratio (A) seems to have significant effects also on the stiffness and toughness strengths, while the effect of CO_2-curing concentration (B), seems to be fluctuated on the stiffness and toughness strength results.

Table (7). Analysis of variance of the flexural test results for CO_2-cured, cellulose fiber reinforced cementations boards.

	Flexural Strength		Flexural Toughness		Initial Stiffness	
	F-Ratio	P- Value	F-Ratio	P- Value	F-Ratio	P- Value
A	42.305	0.000*	22.32	0.000*	69.44	0.000*
B	9.73	0.003*	2.72	0.106	16.62	0.000*
A×B	7.12	0.009*	9.429	0.003*	1.196	0.336

*: Statistically significant difference at 95% level of confidence
A: Cellulose fiber/matrix ratio; B: CO_2-curing concentration

Fig. 8 and **Table 8** show measured values of bulk density for cement-bonded cellulose fiberboard subjected to 0%, 30%, and 100% of CO_2-curing. Specimens subjected to 0% and 30% CO_2-curing are observed to provide similar densities. 100% concentration of CO_2-curing however, resulted in 13.34% and 10.38% increase of bulk densities for cellulose fiber ratios 5% and 8% respectively. The reason behind this is the increment in $CaCO_3$ in the resulted composite matrix which is denser than $Ca(OH)_2$, C-S-H, and other hydration products [33]. Higher CO_2-curing concentration seems to have significance effect to increase specimens densities due to the densification effects of carbon dioxide and its chemical reactions with the hydration product calcium hydroxide $Ca(OH)_2$ filling existing pores with new solids and products leading to reduce porosities and increase bulk densities.



Figure(8). Bulk density of cement-bonded cellulose fiber-board (CBCB) subjected to different CO_2-curing concentrations.

X-Ray diffraction

Fig. 9 shows the X-ray patterns of cement-bonded cellulose fiber-board of CO_2 cured composites after 28 day of curing. **Fig. 9a** and **b** reveals CO_2-cured specimens had higher $CaCO_3$ contents and lower $Ca(OH)_2$ contents. Composites with different cellulose fiber ratio performed similarly. This behavior is probably due to conversion of $Ca(OH)_2$ to $CaCO_3$ throughout the CO_2-curing process [28]. The results are consistent with observation of Maail et al [34], who observed that the application of CO_2-curing could promote the reaction of carbon dioxide to produce calcium carbonate ($CaCO_3$), resulting in more strength to the final composites.

Table (8). Bulk densities mean values of cement-bonded cellulose fiber-board (CBCB) subjected to different CO_2-curing concentrations.

CO2-concentration	Cellulose Fiber 5%		Mean (SD)	Cellulose Fiber 8%		Mean (SD)
0%	1442.34	1462.9	1452.6 (14.53)	1361.25	1343.76	1352.5 (12.36)
30%	1316.65	1397.48	1357.1 (57.15)	1261.72	1285.30	1273.5 (16.67)
100%	1652.05	1640.81	1646.4 (7.94)	1499.91	1485.83	1492.9 (9.95)

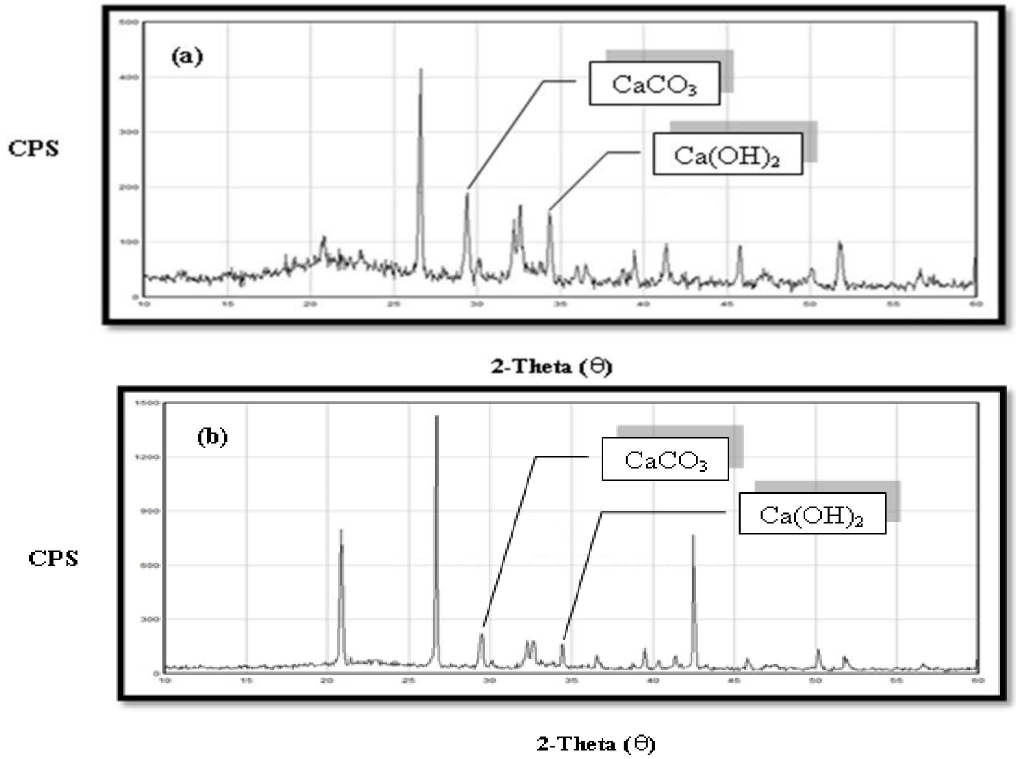
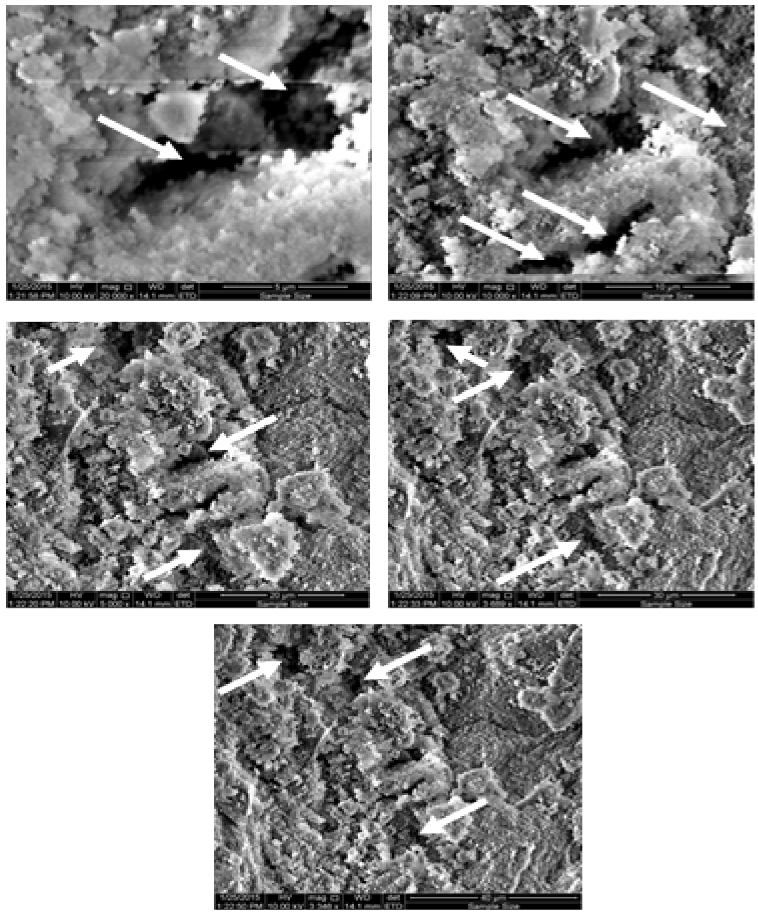


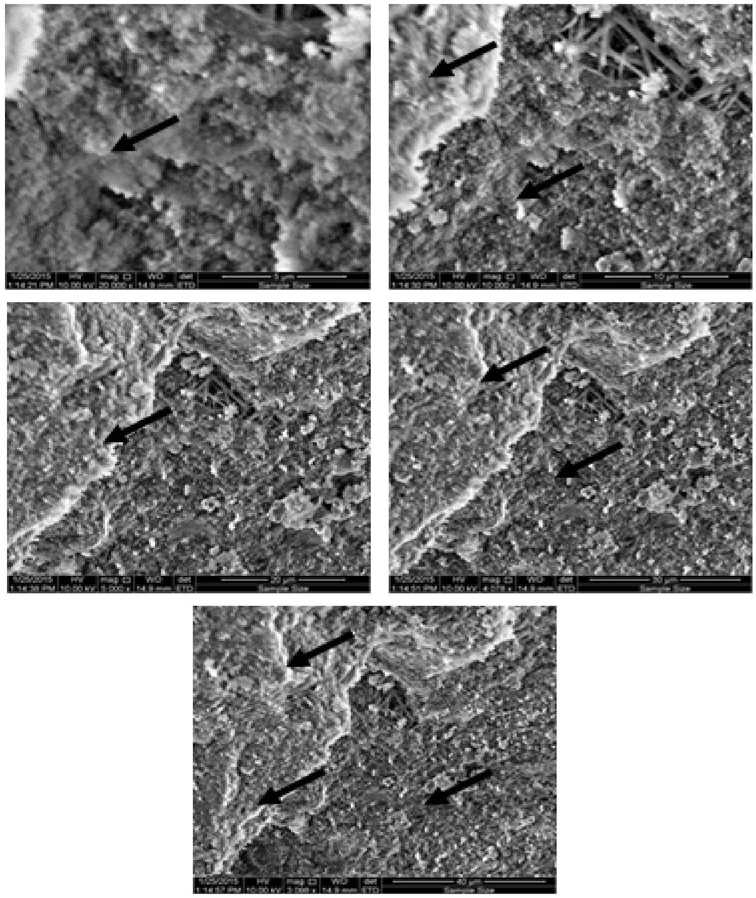
Figure (9). X-ray patterns after CO2 curing 30% concentration for cement-bonded cellulose fiber-board; (a) cellulose fiber ratio 5%, and (b) cellulose fiber

Fracture surface observations

Figs. 10 and **11** depict the SEM images of the fractured surface of the cellulose palm fiber reinforced cementitious composites. Samples taken from the lower tension fracture zone of tested boards under flexural load. The SEM micrographs used here are typical images of the microstructure observed from around overall twenty images for each composite treatment. The analysis of these micrographs allows the observation of the cement phases developed after the exposition to accelerated carbonation, and their impact on the interface between fibers and the cement matrix.



Figure(10). SEM micrographs of the fractured surface of non-carbonated and 5% fiber/matrix ratio of fiber-cement composites, white arrows indicate porous areas.



Figure(11). SEM micrographs of the fractured surface of 100% CO_2 cured and 5% fiber/matrix ratio of fiber-cement composites, black arrows indicates denser areas.

In an image such as **Fig. 10**, pores area (indicated by the white arrows) appears to occupy higher percentage in non-carbonated specimens compared to the tested specimens subjected to CO_2 curing. Differently from the non-carbonated composites, the microstructure in the accelerated carbonated composites, (**Fig. 11**), is compact and formed by layered structures (black arrow), probably related to the $CaCO_3$ phases. These results agree with the lower content of carbon dioxide $Ca(OH)_2$ and higher content of calcium carbonate ($CaCO_3$), observed in the X-ray diffraction (XRD) of carbonated composites (**Fig. 9**).

The observed high percentage of pores area in the non-carbonated specimens is also an indicative of a lack of contact between cellulose fibers and matrix. As a result, during a bending test, the cellulose fibers would be easy pulled out from the cement matrix when compared with carbonated specimens.

Summary and Conclusions

An experimental study was conducted to evaluate the effects of CO2 curing on the mechanical properties of cellulose date palm fiber-reinforced cementitious boards, and to develop an efficient processing approaches which makes value-added use of carbon dioxide and/ or date palms residues. Two cellulose palm fiber/matrix ratios were evaluated: 5% and 8%. The performance characteristics were evaluated through flexural testing of composites and different processing aspects were implemented. The results indicate that:

- All processing variables (CO_2 curing concentrations and cellulose fiber/matrix ratio) had statistically significant effects on the end product at 95% level of confidence, on flexural performance.
- The CO_2-cured cellulose date palm fiber reinforced cementitious composite boards generally have higher $CaCO_3$ and lower $Ca(OH)_2$ contents. Higher $CaCO_3$ contents usually correlate with higher flexural strength and stiffness.
- The SEM micrographs show that the CO_2 curing increases matrix densities and reduces the pore volume in both fabricated boards with 5% and 8% fiber/matrix ratios.
- Analysis of variance of flexural performance results yielded the preferred processing conditions of cellulose fiber cement boards. Lower fiber/matrix ratio and higher CO_2 curing concentration were chosen as the preferred conditions.
- From a practical point of view, the interaction effects of cellulose date palm fiber/cement ratio with CO_2-curing on flexural strength are relatively high.
- Higher CO_2 curing concentration densified the matrix structure and the fiber matrix interfaces were enhanced. Similar effect was notified at both cellulose fiber/matrix ratios used.
- This manufacturing procedure might benefits the construction industry by offering sustainable building products to be used wildly and helping consume CO_2 emitted.

This study demonstrates the positive impact of accelerated CO_2 curing and fiber/matrix content, on the flexural performance and matrix microstructure characteristics of cement-bonded date palm cellulose fiberboards composite.

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