Strength Evaluation of CO_2-Cured Cellulose Date Palm Fiber Reinforced Cementitious Boards

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ABSTRACT

In recent years there has been an increasing demand to recycle wastes produced by the agricultural and industrial processing. The aim of this paper is to investigate the suitability of date palm (Phoenix dactylifera) as lignocellulosic materials for the production of wood-cement composite boards, in addition to enhance their compatibility with cement using physical pretreatment processes and accelerated carbonation curing.

Experiments were performed to assess the physical properties (as density, flexural strength, toughness and E-modulus), and micro structural properties (as determined by scanning electron microscopy) of the produced cement boards. The results show an improvement in the physical and microstructural properties of cellulosic fiber-cement composites by using accelerated CO_2 curing method. In addition, excessive carbonation rate associated with pure gas carbonation does not necessarily lead to high strength and even detrimental strength development was found, which was shown by cement paste.

Keywords: cellulose fiber, accelerated curing, flexural strength, microstructure, carbonation.

INTRODUCTION

The usage of waste fibers as reinforcement with the corporation of cementitious materials has great interest for civil engineering construction in terms of recycling. Besides, the accessibility of natural waste fibers encourages their promising usage to produce different building components through sustainable manners **[1]**. Iraq has relatively large quantities of lignocellulosic materials available in the form of agricultural and industrial residues since there are about 15 million trees of palmdate only **[2]**.

Cellulose fiber, as a reinforcing material that is renewable and very high in performance to cost ratio, improves the ductility, flexural and tensile strength, fracture toughness, and machinability of the cement matrix, mainly through arresting and deflecting microcracks and through the pullout action of wood fiber at fractures **[3]**. Synthetic fibers are expensive, high-energy consumers and often manufactured from nonrenewable resources. While, natural fibers have renewable resources and are accessible worldwide. Therefore, the use of concrete reinforced with natural fibers could be a method to enhance both concrete strength and sustainable production **[4]**.

Cellulose fiber cement boards (CFCBs) offer desirable durability, thermal insulation, life cycle economy and most important: strength and toughness characteristics. Typical applications of CFCBs are as floor and wall sheathing, exterior siding, sound insulation, roof shakes and roof decking **[5]**. More uses that are specialized include permanent formwork, pre-fabricated house

components, non-structural applications in both interior and exterior situations, sound barriers, and the construction of protective elements of fireproofing **[6]**.

The accelerated hardening process with carbon dioxide revolutionized the manufacture of wood-cement composites **[7]**. The utilization of CO_2 gas as an accelerated curing process in fresh concrete has been submitted as a CO_2 sequestration method that contribute a value-added product and carbon dioxide cured process **[8]**. [**Young, 9**] mentioned that, "the formation of C-S-H like gel as well as calcite ($CaCO_3$) that subsequently carbonated to silica gel and calcium carbonate in the first 3 minutes of carbonation of C_3S and C_2S".

Objectives

The ultimate goal of this study is to develop the industry manufacture of wood-cement composites in a way that the products can be made with higher productivity and better mechanical properties. The major objectives are:

1. Investigating the suitability of date palm (Phoenix dactylifera) as lignocellulosic materials for the production of wood-cement composite boards, in addition to enhance their compatibility with cement using physical pretreatment processes.
2. Studying the effect of accelerated hardening with carbon dioxide on the properties of wood-cement composites made from recycled palm date fibers.
3. Studying the effect of manufacturing parameters, such as gas concentrations, chamber duration and chamber temperature, on the development of carbonation reaction, strength and microstructure of wood-cement composite boards.

Materials and Methods

Ordinary Portland cement, commercially known (MASS), was used to produce the fiberboards. Tables 1 and 2 show the chemical composition and physical properties of cement used throughout this work respectively. Results indicate that the cement is conformed to Iraqi specification No. 5/1984. Natural sand with 2.36 mm maximum size brought from Al-Ekhaider region was used as a fine aggregate in this research. Table 3 shows the grading of sand used in this work. The grading of sand is conformed to Iraqi specification No.45/1984 (zone 1). The sulfate content, the bulk density, specific gravity and the absorption of the sand were 0.07 %, 1500 kg/m^3, 2.65, and 1.2 % respectively. Densified micro silica produced by BASF Company was used as pozzolanic admixture. The technical specifications and pozzolanic activity index are given in Table 4. The results show that silica fume used in this investigation conforms to the requirements of ASTM C-1240-05, ASTM C-618 specifications. The superplasticizer used in this work was based on a unique carboxylic with long lateral chains, which greatly improves cement dispersion. It is commercially known as GLENIUM 51. Table 5 shows the typical properties of it. This admixture is complying with type (F) according to ASTM C494-03.

Cellulosic fibers were derived from Iraqi date palm trees by dry mechanical processing method (Plate 1). A hummer mill used to make the fibers, and screened the resulting fibers to a final 4 mm mesh. The moisture content and density of the investigated fibers were measured according to the ASTM D-4442-03 and ASTM D 2395 – 07a standards as shown in Table 6. Physical fiber pretreatment (hornification) aimed to remove extractives that inhibit cement curing. The hornification of the fibers was carried out according to procedures outlined by **[Ali, 10]**.

Mix Proportions and Casting of Boards

Many trial mixes were carried out in the building materials laboratory to find a suitable mix that having the desirable properties in the fresh state to satisfy the ASTM C-208-01 and ASTM C-1186-08 requirements of cellulose fiber-cement boards in the hardened state. All samples with nominal thickness of 12 mm were obtained from the dry process. The semi-automatic dry process is the most employed in the fiber cement industry worldwide. The water and superplasticizer contents

were chosen in order to achieve a reasonable fresh mix workability characteristics represented by a flow (ASTM C-1437) of 65-75 % at 1 min after mixing. All the specimens, which have dimensions of (305*152*12) mm, were kept inside their molds underneath wet burlap covered with a plastic sheet for 24 hrs as shown in Plate 2. They were then demolded, dried at 60 ^{o}C for 30 min. and cured for the specified time and temperature following that procedure outlined by **[Alanbari, 11]**. The proportions of concrete mixes are summarized in Table 7.

Program of Work

The work consisted of two series of Cellulose Fiber Cement Boards (CFCBs) mixes, namely: control and CO_2 cured boards, to investigate the effect of several factors. In order to evaluate the strength of CFCBs, different factors were performed: CO_2 concentration, chamber duration and chamber temperature. One day after completion of processing, the fiberboards were carefully removed from the molds, dried at 60 ^{o}C for 30 min and then subjected to CO_2 curing. The drying process will ensure the availability of empty paths for CO_2 to take place. The CO_2 curing chamber process was performed at different ambient temperatures (25 ^{o}C, 50 ^{o}C and 75 ^{o}C) and CO_2 concentrations (0%-25%-50%-100%) for two duration times, 90 and 180 minutes. A comparison has to be made for the CO_2 cured specimens with control specimens.

The carbonation curing apparatus was used to subject samples to a low pressure of carbon dioxide gas and oxygen gas. Major components of the set-up included compressed gas tanks, pressure vessel, thermocouples, data acquisition, and vacuum with pressure and heat transducers. The samples in the curing chamber were treated with CO_2 gas at 6.9 MPa (1000 psi) at different temperatures for 90 and 180 minutes, respectively. In the specimen preparation of this section and the following ones, 20-minutes vacuum were applied before CO_2 injection, and the CO_2 flow rate was 10 L/min that mean about 25 min. is needed to reach 100 % concentration.

Mechanical Properties and Strength Tests

Flexural Strength, Toughness and Modulus of Elasticity

Flexural tests are performed at 28-day according to ASTM C1185-12 and ASTM C1186-12. Board dimensions measured (305 mm) in length and (152 mm) in width with 12 mm thickness. The specimens were simply supported over a span of (255 mm) and loaded at a displacement rate of 0.5 mm/min (which were conducted in a displacement-controlled mode). A computer controlled data acquisition system is used to record the test data. The load-deflection curves are characterized by flexural strength, toughness (total area underneath the load deflection curve), and modulus of elasticity. Each value of the flexural strength was the average of the test results for three specimens.

Dry Density

Density was determined at 28-day in accordance to ASTM C 1185-08 (2012) with reference to ASTM C 20-07 using the water displacement method. Each specimen was weighed under water after being immersed for 48 hours. The saturated weight in air was then measured and the dry mass was obtained by drying each specimen to constant weight in an oven at 194 ± 4°F (90 ± 2°C).

Scanning Electron Microscopy (SEM) Observations

The textural study for the fractured surface of the samples 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 12 mm. Four SEM micrographs were obtained for each composite treatment and just the typical images of the observed microstructure were used in this manuscript. Conducting this test requires to prepare a sample of test with the suitable dimensions (1*1*1) cm. This test was carried out by Nanotechnology and Advanced Materials Research Center/University of Technology.

Results and Discussion

Flexural Strength

Fig. ١ shows a typical load-deflection curves that obtained with different CO_2 concentrations. It could be seen that, the behavior is elastic at the beginning of the loading; up to the first crack. Furthermore, this figure shows that the CFCB behaves as an elastic-degrading plastic material with significant loss of strength following the achievement of peak load.

After that, either the initiated crack had an unstable growth leading to separation of the body into parts for control mix, or to a macro-crack with a deflection around 0.55 mm, when the fibers could stop the crack growth for CO_2 cured boards.

Further, it is obvious that the flexural performance enhanced with the increasing of CO_2 concentrations. The toughness indices derived from results under bending test were (200-350) N.mm, and the results indicate that the CO_2 concentrations have a significant influence on the toughness of the fiberboards. A medium concentration (50 %) of CO_2 observed comparable to that obtained at 100 % CO_2 on flexural performance. An economic criterion encourages the use of lower CO_2 concentrations since the difference between them was negligible.

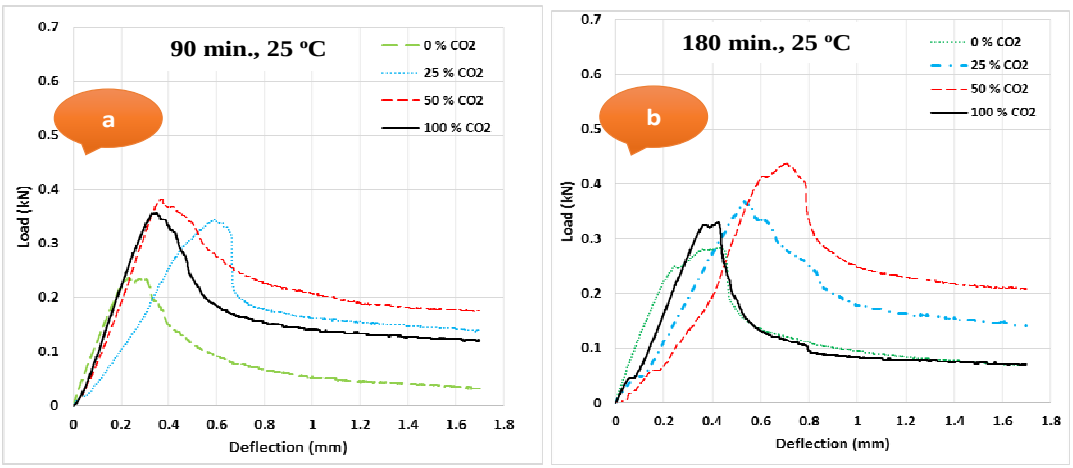


Figure 1: Load-deflection curves for varying CO_2 concentration at chamber temperature of 25 ^oC for (a) 90 min of CO_2 curing and (b) 180 min of CO_2 curing.

Figure 2 shows the values for flexural strength results of all date palm mixes at 28-day. With respect to boards without CO_2 curing, there is an increase in the flexural strength for all CO_2 cured boards. In other words, samples cured by CO_2 gas were stronger than those made conventionally after 28-day. In addition, comparing to control mix (0 % CO_2), it was found that the increase in flexural strengths were between (42%-59%), (19%-72%) and (33%-45%) for 25 ^oC, 50 ^oC and 75 ^oC respectively at 90 min. The rate of increase in flexural strengths were (30%-55%), (40%-100%) and (14%-24%) for 25 ^oC, 50 ^oC and 75 ^oC respectively at 180 min. The beneficial effect of CO_2 injection on the accelerated hardening of wood-cement composites was clearly established. This trend is similar to that found by [**Beltran, 12]**.

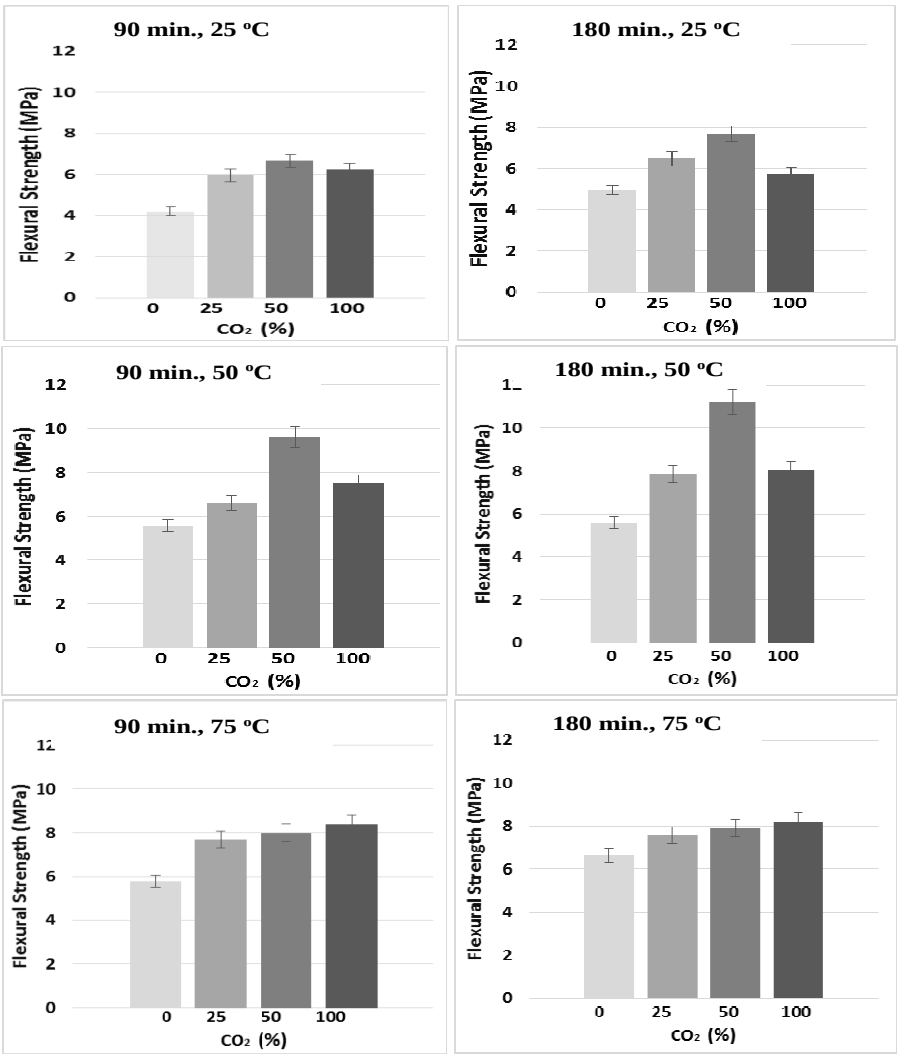


Figure 2: Flexural strength of date palm-cement boards at different (CO_2 concentrations, chamber duration and chamber temperature), mean and S.D.

The CO_2 content in the solidified material increases with increasing temperature (up to 50 ^oC), possibly reflecting the leaching of Ca^{2+} ions from the fibers. Above 50 ^oC, the CO_2 content decrease with further increase in reaction temperature, which may be due to the decreased solubility of CO_2 in water at elevated temperatures. This result suggests that the carbonation process may be controlled by the solubility of CO_2 and the amount of dissolved Ca^{2+} ion in the water. Calcite ($CaCO_3$) products a layer coverage and associated loss of exposed board surface area and pore network and it was identified as the main factor limiting the rate, depth and extent of carbonation.

Toughness

Toughness is a measure of the absorbed energy per unit area of material and is usually defined as the area under the load-deflection curve. The toughness of wood-cement composites after CO_2 curing was illustrated in Figure 3. Similar to the flexural strength development, the toughness of the carbonated samples increased after the beginning of CO_2 exposure. The strength gain effect was significant for samples subjected to 50 % of CO_2 concentration, but a sharp increase was observed after the samples were cured by 25 % and 100 % of CO_2 concentrations. Moreover, an increase in the CO_2 concentration from (25% to 100%) increases the flexural toughness by (104%-152%), (36%-44%) and (44%-25%) for 25 ^{O}C, 50 ^{O}C and 75 ^{O}C respectively at 90 min. The rate of increase in flexural toughness were between (53%-81%), (1%-48%) and (47%-31%) for 25 ^{O}C, 50 ^{O}C and 75 ^{O}C respectively at 180 min. The flexural toughness kept increasing until 50 % of CO_2 curing. After that, there was no significant difference in toughness with the further increase of CO_2 concentration.

The examination of toughness properties represented by Figure 3, shows that for each term (90 or 180 min.) the flexural toughness, increases with an increase in the CO_2 concentration incorporated in the mix, with the maximum toughness, being obtained with the 50 % for both curing periods. In addition, there was no significant difference in toughness with the further increase of CO_2 concentration more than 50 %.

Further, as compared to control fiberboard, the palmdate fiberboard yielded higher flexural toughness by (152%-84%) at 50 % CO_2 concentration for 90 and 180 min respectively. It is also a fact that improving the bond between the fiber and the matrix (because of CO_2 curing) resulted in an improvement in the interfacial transition zone. The strain in the composite at a given stress depends on the length of debonded fibers and, hence, a greater bond leads to smaller failure strain and fibers are broken rather than pulled out. This behavior probably interprets the reduction in flexural toughness associated with increasing CO_2 curing **[5]**.

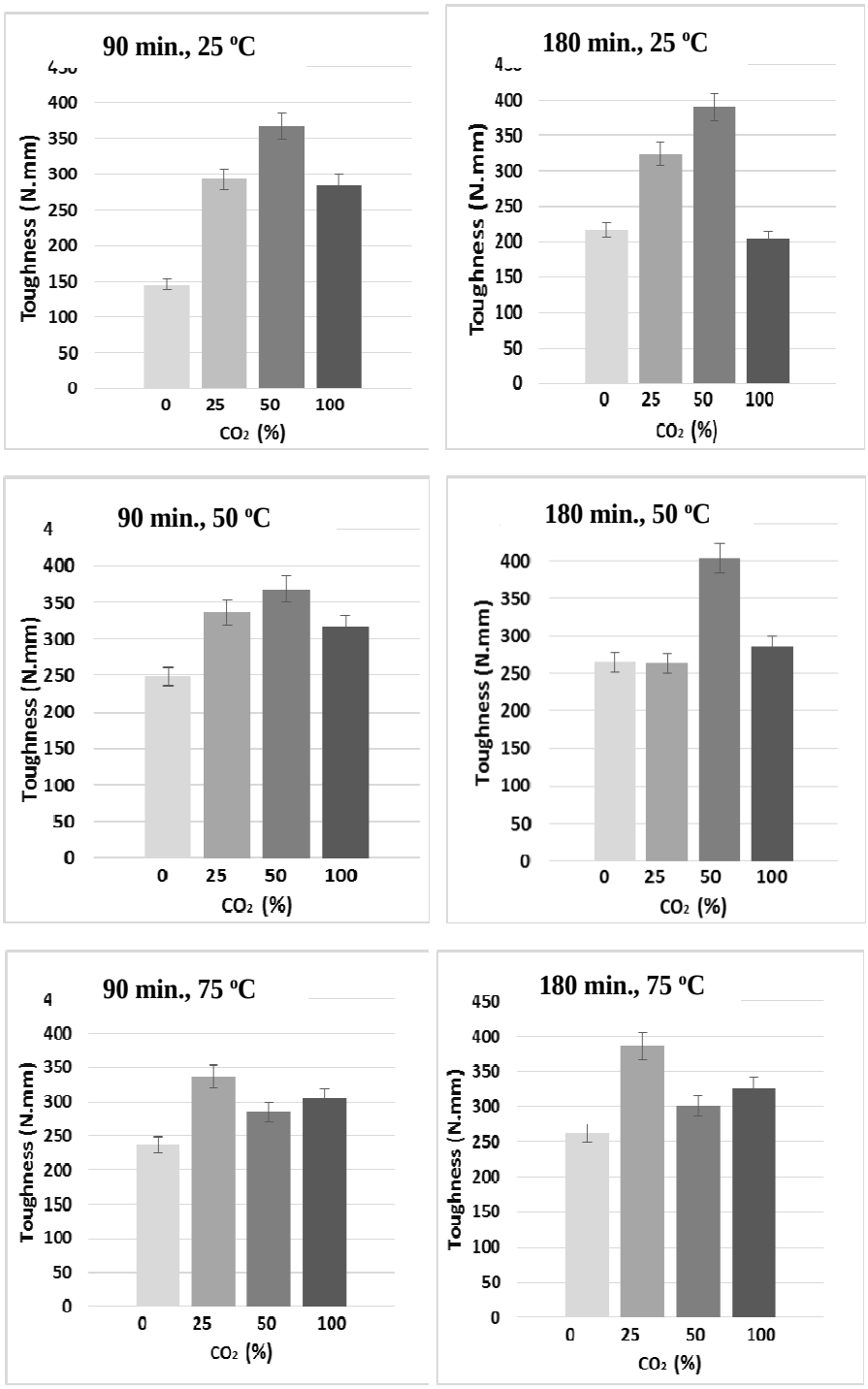


Figure 3: The effect of CO_2 concentrations, chamber duration and chamber temperature on flexural toughness of date palm-cement boards, mean and S.D.

E-Modulus

Initial modulus or initial stiffness is defined as the stiffness obtained through linear regression analysis of the load–deflection points for loads below 15% of maximum load **[13]**. The modulus of elasticity for all of the investigated cellulose fiberboards is shown in Figure 4.

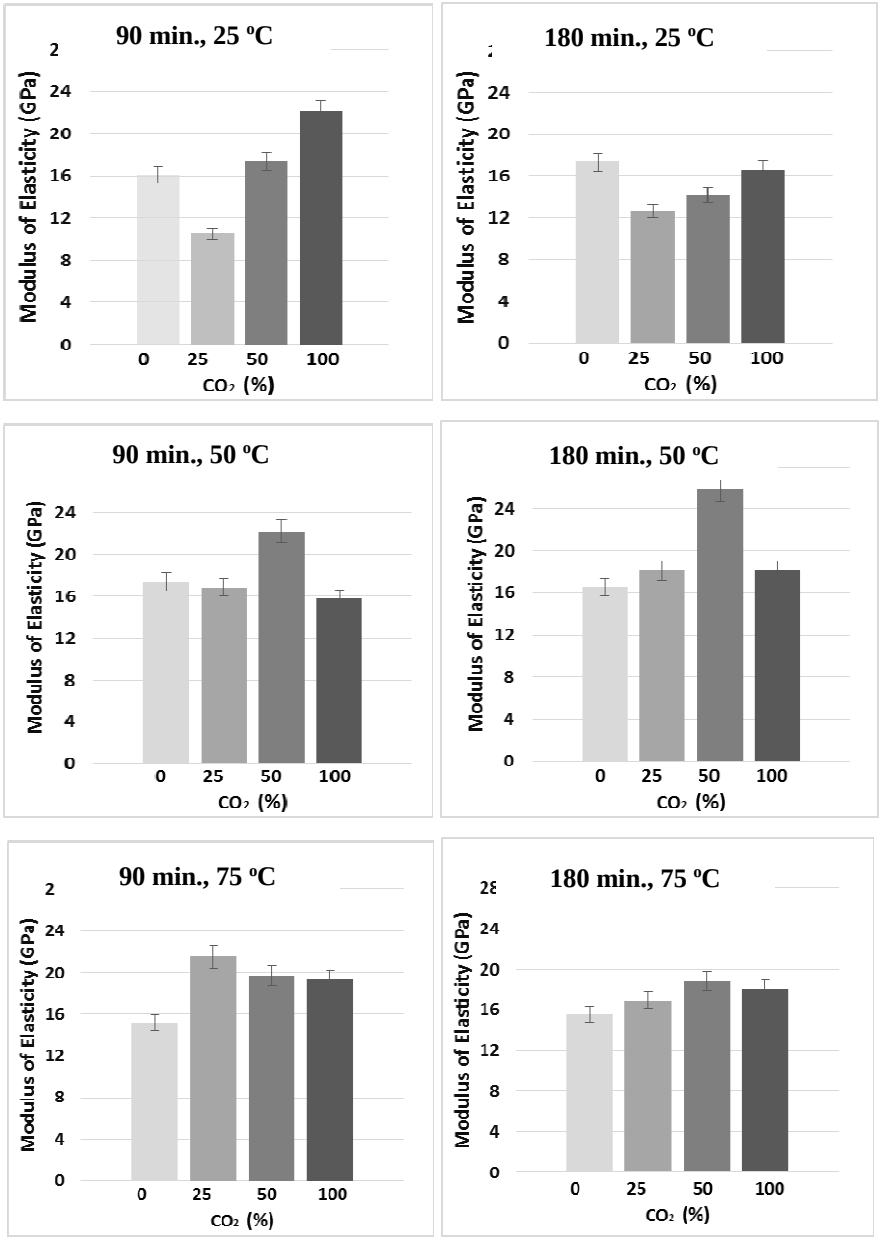


Figure 4: Modulus of elasticity of date palm-cement boards at different (CO_2 concentrations, chamber duration and chamber temperature), mean and S.D.

The test results in this Figure shows that the modulus of elasticity almost fluctuated as the CO_2 concentration is increased from 0 % to 100 %. Moreover, the stiffness of the wood cement fiberboards increases linearly with the increase of CO_2 concentrations to 50 % at 90 min. of chamber duration. However, increasing the CO_2 concentrations to (100 %) leads to a reduction in the static modulus by 25% and 36% for 90 min. and 180 min. respectively.

From the recorded results through the experimental work, it can be noticed that the 28-days modulus of elasticity for date palm fiberboards ranges between (10.5-22.5) GPa, (15.98-22) GPa and (15-21.3) GPa for 25 ^{O}C, 50 ^{O}C and 75 ^{O}C respectively at 90 min. While these results revealed that the 28-days modulus of elasticity for date palm fiberboards ranges between (12.4-17) GPa, (16.28-26) GPa and (15.8-18.3) GPa for 25 ^{O}C, 50 ^{O}C and 75 ^{O}C respectively at 180 min.

Dry Density

Apparently, the overall densities of control date palm fiberboards were lower than those for CO_2 cured fiberboards, as shown in Fig. 5; this could be attributed to the extra formation of $CaCO_3$ due to carbonation curing and the filling effect of these products in the fiber lumen. Moreover, an increase in the CO_2 concentration from (25 to 100) % increases the density of palmdate fiberboards by (2.1-3.7) %, (4.3-5) % and (6.4-6.6) % for 25 ^{o}C, 50 ^{o}C and 75 ^{o}C respectively at 90 min. The rate of increase in density was between (2.5-4.8) %, (6.2-6.9) % and (5.6-6.4) % for 25 ^{o}C, 50 ^{o}C and 75 ^{o}C respectively at 180 min.

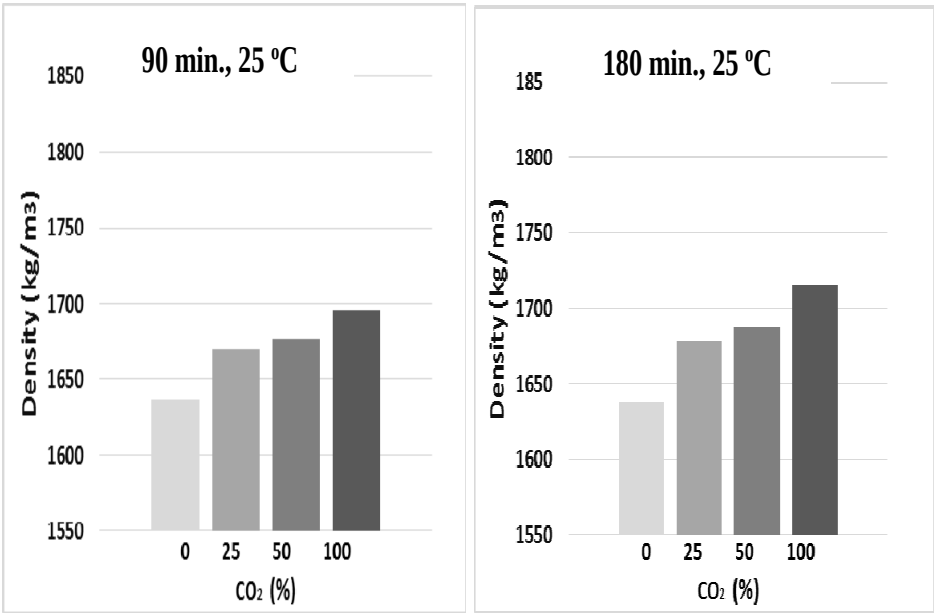
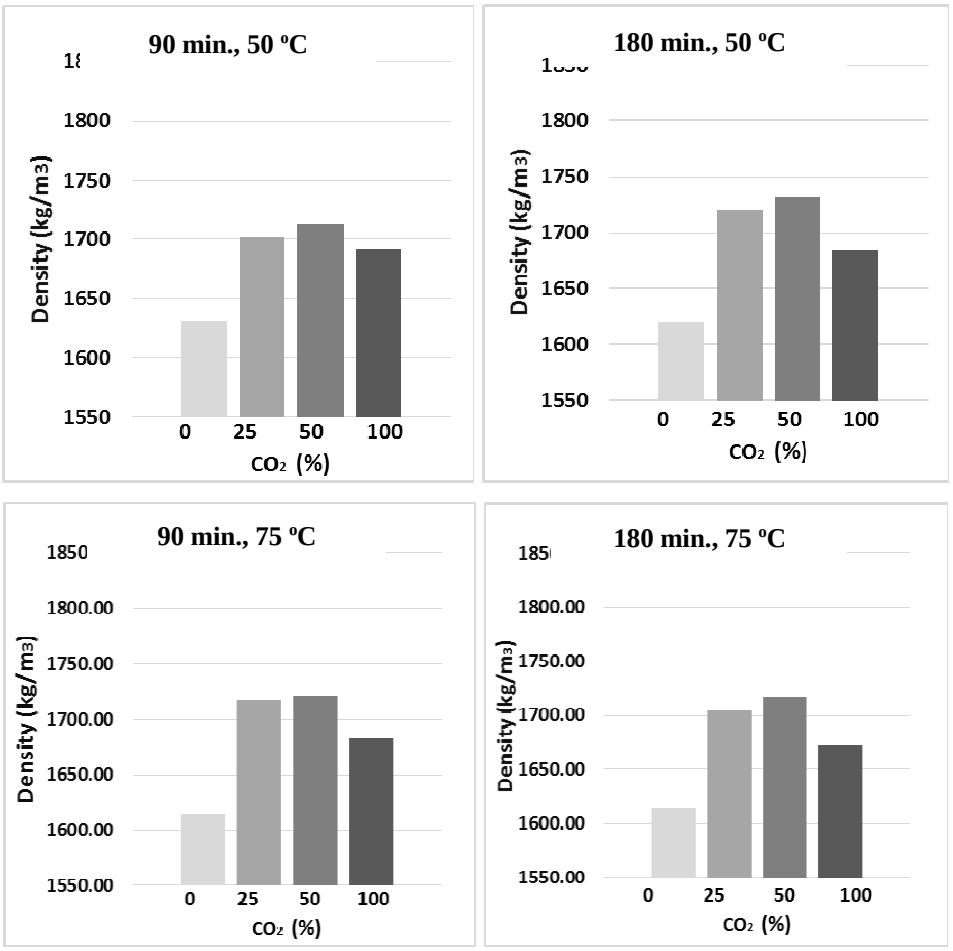


Figure 5: Density of date palm-cement boards at different (CO_2 concentrations, chamber duration and chamber temperature).



Continue to Figure 5: Density of date palm-cement boards at different (CO_2 concentrations, chamber duration and chamber temperature).

Scanning Electron Microscopy (SEM)

SEM analysis of the carbonated material offered a qualitative view of the mineralogy that lies inside the board structure. Fig. 6 views the SEM micrographs of fractured surface of composite samples before and after accelerated carbonation. Further, it seems clear that the surface of palmdate fiber covered with Ca $(OH)_2$ (arrow 1) Fig. 6a. In general, it is obvious that the palm date fibers have a comparatively simple and uniform structure. It is appear that they are made up of a large number of cell types with the long pointed fibrous cells termed tracheids providing both the structural support and the conducting pathways in composite. These tracheids are empty (arrow 2), for non-carbonated samples, (0 % CO_2), and completely filled with $CaCO_3$ for carbonated boards.

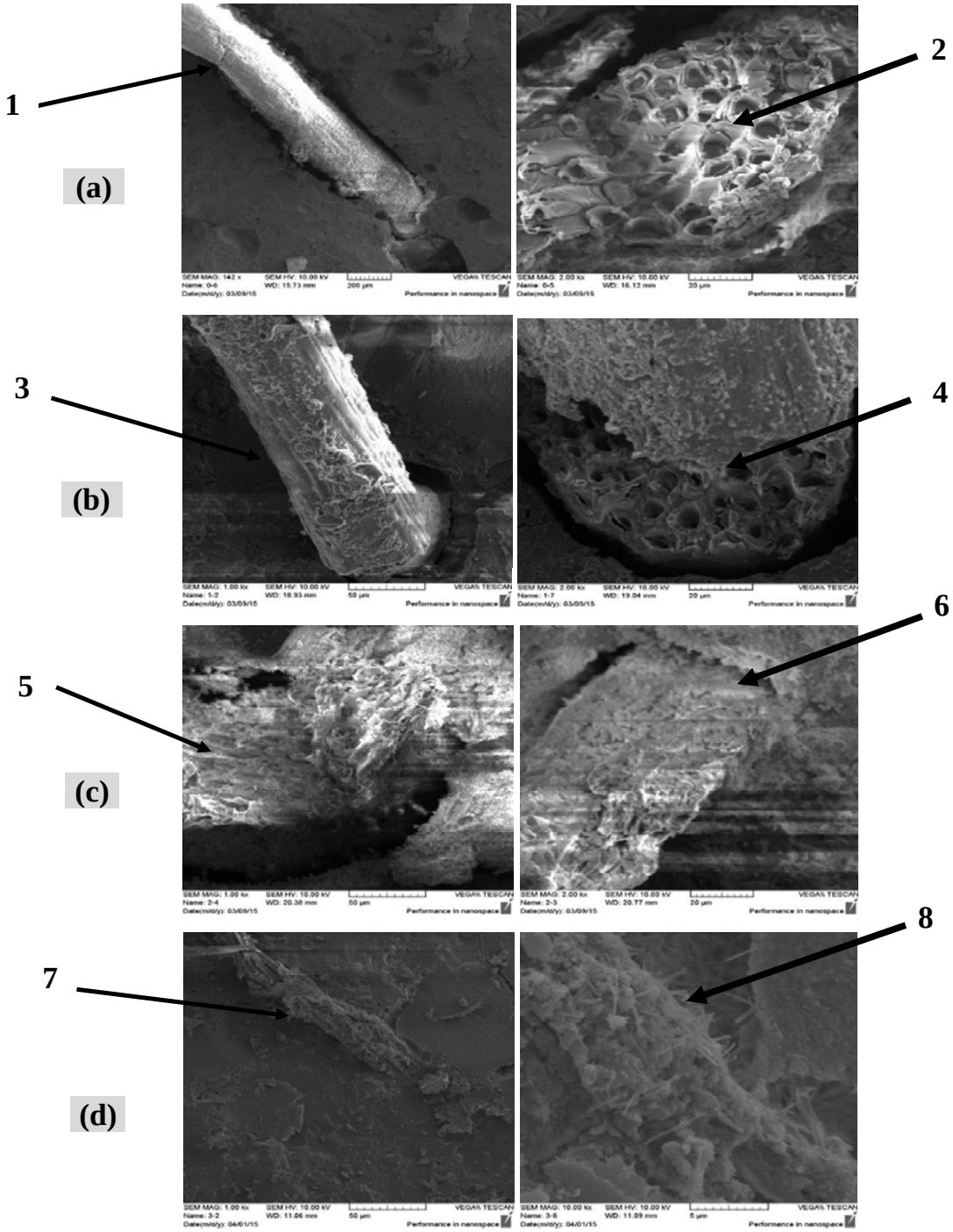


Figure 6: SEM images of fractured surface of the 28-day date palm-cement boards: (a) non carbonated (0%CO_2), (b) carbonated (25%CO_2), (c) carbonated (50%CO_2) and (d) carbonated (100%CO_2).

Arrows 3 and 4 in Fig. 6b, arrows 5 and 6 in Fig. 6c and arrows 7 and 8 in Fig. 6d, show needles of $CaCO_3$ formed on fiber surface, in the fiber pores and around the cellulose fibers after CO_2 curing. Therefore, the cement matrix of the carbonated composite is denser, compact, leading to improvement in the interfacial transition zone between fibers, and cement matrix, and certainly favoring the flexural performance of the end composite. The second benefit of using accelerating CO_2 curing is a physical effect of pore size refinement and filling up capillary pores and crack spaces, thus improving the microstructure of cement paste **[14]**. A very important phenomenon that can be noticed, control boards tailed mainly pulling out of palm date fibers (arrow 1). Consequently, a combination of fiber rupture and fiber pull out occurred in the carbonated boards. $CaCO_3$ 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 and thicker ITZ (arrow 6).

CONCLUSIONS

Based on the tests results of the present study, the following conclusions can be drawn:-

1. Date palm fibers were suitable for using as reinforcement with cement only if they were pretreated with hot water since maximum flexural strength exceeds 1.89 MPa (ASTM C-208/08).
2. The overall date palm fiberboards mechanical properties were better than those of control fiberboards by between (42%-59%), (19%-72%) and (33%-45%) for 25 ^oC, 50 ^oC and 75 ^oC respectively.
3. Cement bonded cellulose fiberboards had exhibited very significant CO_2 absorption behavior that enhanced the overall flexural performance and CO_2 curing have yielded better matrix and board qualities.
4. Excessive carbonation rate associated with pure gas carbonation does not necessarily led to high strength and was found even strength development limiting factor. Further, as compared to control fiberboard, the palm date fiberboard yielded higher flexural toughness by (152%-84%) at 50 % CO_2 concentration for 90 and 180 min respectively.
5. The carbonation of $Ca(OH)_2$ is accompanied by an increase in the volume of solids. This is the consequence of the increase of $CaCO_3$ in the composites, which is denser than $Ca(OH)_2$.
6. $CaCO_3$ 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.
7. Part of the carbonate crystals was found diffused into the fiber cell walls and cavities and were found to protrude from fiber surfaces.
8. Petrification of fibers and densification interface zones caused tendencies toward increased strength and embrittlement. Petrified fibers with excess bonding tend to rupture rather than pullout at fracture surfaces, thus eliminating the desirable toughness characteristics associated with frictional energy dissipation during fiber pullout.

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Appendix

Table 1: Chemical composition and main compounds of cement. *

Oxide composition	Abbreviation	Content %	Limits of I.Q.S No. 5/1984
Lime	CaO	61.9	
Silica	SiO_2	20.1	
Alumina	Al_2O_3	4.75	
Iron Oxide	Fe_2O_3	3.21	
Sulfate	SO_3	2.42	< 2.8 %
Magnesia	MgO	3.01	<= 5 %
Potash	K_2O	0.60	
Soda	Na_2O	0.22	
Total Alkalis	$(Na_2O+0.658K_2O)$	0.61	
Loss on ignition	L.O.I.	2.18	<= 4%
Insoluble residue	I.R.	0.81	<= 1.5 %
Lime Saturated factor	L.S.F.	0.97	0.66 – 1.02
Main compounds			
Tricalcium Silicate	C_3S	٥٥.83	-
Dicalcium Silicate	C_2S	15.51	-
Tri calcium Aluminate	C_3A	٧.16	-
Tetra Calcium Aluminoferrite	C_4AF	9.٧7	-

* Chemical test were made by the National Center for Geological Survey and Mines.

Table 2: Physical properties of cement. *

Physical properties	Test results	Limits of Iraqi Specification No. 5/1984
Specific Surface area (Blaine Method) m^2/kg	280	≥ 230
Soundness by Autoclave	0.15 %	≤ 0.8 %
Setting time (Vicates Method):		
Initial setting time, hrs: min.	1:58	≥ 00:75
Final setting time, hrs: min.	3:24	≤ 10:00
Compressive strength of mortar		
3- day MPa	19	≥15
7- day MPa	26	≥ 23

* Physical test were made by the National Center for Geological Survey and Mines.

Table 3: Grading of fine aggregates *.

Sieve size (mm)	Cumulative passing %	Limits of B.S. 882/1992	Limits of Iraqi Specification No. 45/1985	Cumulative retained %
4.75	90.4	89-100	90-100	9.6
2.36	70.2	60-100	60-95	29.8
1.18	48.0	30-100	30-70	52.0
0.60	26.6	15-100	15-34	73.4
0.30	13.8	5-70	5-20	86.2
0.15	5.8	0-15	0-10	94.2
Finesse Modules (F.M.)				3.452
Sulfate content = 0.126 % < 0.5% limits of I.Q.S No.45/1985				

* Test were made by the Consulting Engineering Bureau Laboratories in Bagdad University.

Table 4: The technical -specifications of micro silica.

Structure of material	Densified microsilica	Limits of ASTM C 1240-05
Color	Dark gray	
Density	0.55-0.7 kg /L	
Chlorine amount	< 0.1 %	
Fineness (Blaine)	> 15000 m^2 / kg	≥ 15000 m^2/kg
SiO_2	> 85 %	≥ 85 %
CaO	< 1 %	
SO_3	< 2 %	
Activity index	= 156 % at 7-days	≥ 105 % at 7-days
Specific weight	2300 kg/m^3	

Table 5: Typical Properties of SP.

Form	Viscous Liquid
Color	Dark brown
Density	1.1 gm/cm^3 @ 20^{o}C
pH	6.6
Viscosity	128 +/-30 cps @ 20^{O}C
Transport	Not classified as dangerous
Labelling	No hazard label required

Table 6: physical properties of cellulosic fibers.

Property	Date palm
Av. Length (mm)	3.61
Av. Width (mm)	0.23
Aspect ratio (%)	15.70
Dry density (kg/cm^3)	778
Saturated density (kg/cm^3)	1376
As received moisture content (%)	8.84
Moisture content to saturation point (%)	76.9

* Test were made by the Building Materials Laboratory in University of Technology.

Table 7: Mix proportions (by w.t.).

Fiber name	Date palm
Fiber mass fraction Mf (%)	8
Microsilica / cement by w.t	0.5
SP / cement By w.t	0.015
Sand / cement by w.t	0.75
Water / cement by w.t	0.2

Plate 1: (a) The investigated date palm fibers and (b) Hummer mill apparatus.

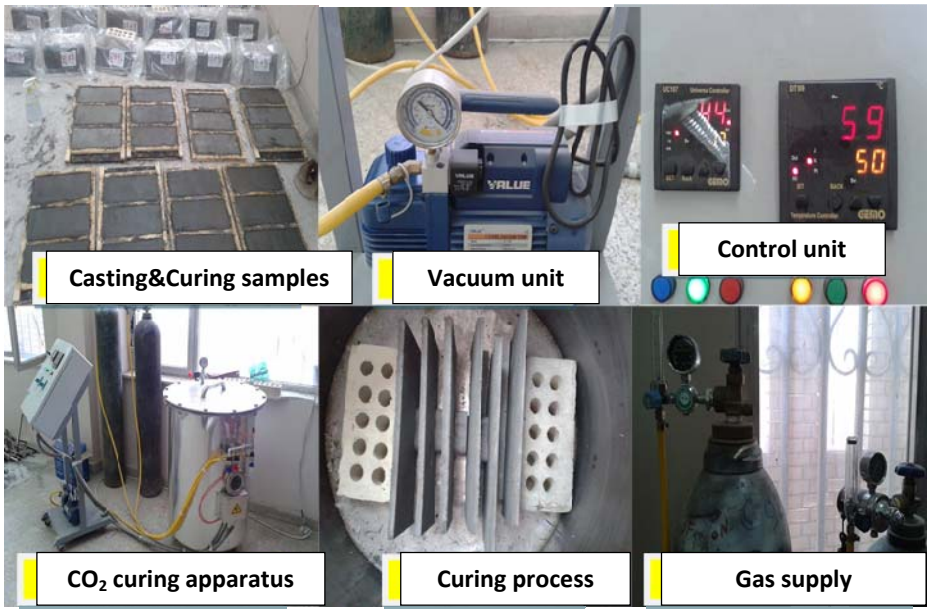

Plate 2: Processing system incorporating CO_2 curing and produced boards.