

Mechanical Performance of CO₂ and Autoclave Cured Date Palm Fiber Reinforced eco-mortar Composites

Dr. Maan Salman Hassan

Building and Construction Engineering Department, University of Technology/Baghdad

Email address: maan_s_h@yahoo.co.uk

Wahad Marwan Salih

Building and Construction Engineering Department, University of Technology/Baghdad

Email address: wahad.marwan@yahoo.com

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ABSTRACT

Using nature waste fibers in construction industries had shown important character "environmental friendly" which paid a great interest around the world. The aim of this paper was to investigate the suitability of agriculture waste date palm fibers as lignocellulosic materials for the production of wood-cement composite, in addition to enhance their compatibility with cement using accelerated curing like carbonation or autoclave.

Three percentages of date palm fibers were used (2, 6, and 10)% by weight of cement in cement composites specimens. Compressive, flexural and direct tension strengths were examined as strength properties and X-ray diffraction (XRD) as microstructure properties. The results show that carbonation curing was the most effective curing for compressive strength property while autoclave curing leads to better performance in flexural strengths. Curing type effects on direct tension property were fluctuated. This could be attributed to the variation in cellulose fiber roles with respect to each property evaluated. X-ray diffraction confirmed that CO₂-curing led to increased CaCO₃ content compared with autoclaved composites.

Keywords: Eco-friendly mortar, Date palm fibers, Carbonation, Autoclave curing, Mechanical properties, X-ray diffraction.

INTRODUCTION

In recent years, conserving of natural resources, using alternative constituents in construction materials and saving energy considered the perspective of global concern due to scarcity of raw materials, high demand and high price of energy caused increment in the cost of construction materials [1].

Natural fibers like sisal, jute, cotton and hemp were considered as innovative materials when it used in construction and manufacturing industries due to its environmental advantages like availability in fibrous form, reliability, biodegradability, sustainability lower pollutant emissions, enhanced energy recovery, lower greenhouse gas emissions and many others of its benefit that considered the main reason to overcome the industrial pollution which responsible on the increment of environmental concern and awareness [2,3]. The ideas of using the industrial and agro-industrial material in sustainable building construction has been paid a great attention due to the effect of building sector which were large spent of resources in materials and energy, highly polluting (emission of CO₂) and a generator of residues [4,5].

Although cellulose fiber is cheap and relatively low energy demand besides of its strengthen and stiffness, cellulose fiber behavior shown increment in fracture energy of fiber-cement matrixes as well as enhancement in some mechanical and physical properties due to the fiber work of micro cracks bridging in addition of pull out action at these cracks, as well as thermal behavior

improvements. This improvement encourage to use unprocessed or processed cellulose fiber in various application as a reinforcement in cement based products around the world such as thin sheet cement products [6,7]. Large quantity of date palm trees approximately 100 million found normally in Middle East Northern Africa, the Canary Islands, Pakistan, India, and in the United States, when seasonal pruning started a huge quantity of plant biomass obtained as an essentially agricultural practice [8, 9,10]. Date palm fibers was one of the important resources of cellulose fiber as agro wastes that used in recycle composites, and as reinforcement materials due to four types fiber that date palm tree offered [4, 9, 11] :

- | | |
|-------------------------------|-----------------------------------|
| 1-Leaf fibers in the peduncle | 3-Wood fibers in the trunk |
| 2- Bast fibers in the stem | 4-surface fibers around the trunk |

Special techniques such as carbon dioxide and autoclave to accelerate the curing of wood-cement and to improve the properties of wood-cement composites have been used [12].

Rapid curing considered one of the solutions to overcome the retarding effect in hydration cement due to using local fibrous agricultural residue and the inhibitory effects of using straw in cement matrix can obtained by carbonation reactions due to using CO₂ curing [13].

Accelerated CO₂ curing for cellulose fibers cement composites shown increment in setting and hardening rates of fibers-cement matrix substantially compared with autoclave curing that required several hours to get the sufficient strength due to the chemical reaction between CO₂ and Ca(OH)₂ from cement hydration resulted CaCO₃ (limestone) [13], however this reaction resulted in accelerating the setting time and the materials hardening were increased but also it may reduce the accelerated curing time [14].

The aim of this research work is to assess the effect of two types of accelerating curing, CO₂ and autoclave curing, on the compressive, flexural, direct tension strengths of the produced composites and compare their performance with control composite specimens.

Eco-friendly cement composites

Recently, environmental pollution considered one of the main forces that responsible for economic and social transformations in society [15] thus, the environmental protection has been highlighted with greater interest which can obtained by new technologies [16] such as green concrete technology that do not effected on high performance and life cycle sustainability or causes environmental destruction who consisted of using at least one waste materials of concrete component or concrete production process [15].

Using natural fiber as reinforcement in cement composites considered ecofriendly that considered important from environmental aspect due to [16,17]:

- 1-These fibers were from natural resources thus it reduced the consumption of virgin wood as well as the rate of deforestation.
- 2-Variou of environmental problems such as air pollution, emissions of greenhouse gases and useful land occupation have been occurred due to the dealing ways of agricultural wastes [18].
- 3-Agricultural wastes have been caused some problems in its land filled areas like biodegradation of lignocellulosic, emits methane, a greenhouse gas that responsible of heat effect increment (about 72 times) due to CO₂ emission. Using these fibers caused reduction in the landfill areas thus large useful land areas could save for productive uses.

Experimental program

Materials and methods

Ordinary Portland cement (Type I) commercially known (Krasta) was used in this study. The physical properties are given in **Table (1)**. The test results of cement used show that the cement conforms to Iraqi Specification No.5/1984 [19]. Al-Ekhaider natural sand was used as a fine aggregate throughout the experimental work. Physical properties of sand are shown in **Table (2)**. The tests results were presented the gradation of the sand lies in zone (III) and the sulfate content (0.343%) which are within the requirements of the Iraqi specification NO. 45/(1984) [20]. A high-range water reducing admixture commercially known as GLENIUM 51 was based on modified polycarboxylic ether which was used throughout the experimental work. The dosage recommended by the manufacturer is from 0.5 to 1.6 liters per 100 kg of cement (cementitious materials). This type of admixture conforms to ASTM C494-06 type F [21]. Densified silica fume produced by the chemical company BASF commercially known as MEYCO/MS610 was used as pozzolanic materials in mixtures as replacement by weight of cement. The result of Accelerated pozzolanic strength activity index conform the requirements of ASTM C1240-03 [22] and ASTM C311-05 [23] specifications.

Cellulose fibers in this study were extracted from the wastes of the date palm trunk nearby and around province. Various wood pieces were cutting by hummer mile to produce fibers passing throughout of 3 mm mesh as shown in **Plate (1)**. The average lengths and widths of fibers were tested using Metallurgical Microscope (MBL 3300) as shown in **Table (3)**. After the wood pieces were cutting with hummer mile, the fibers were treated by 1% of NaOH solution to remove the extractive which considered the main reason that responsible on inhabitation of cement curing. In this study the procedure of **Ahmed et.al** [16] was applied for treatment the fibers with NaOH. Density and moisture content of fibers were investigated according to ASTM D2395-02 [24] and ASTM D4442-03 [25] as shown in **Table (3)**. The organic constituents and chemical compositions of treated date palm fibers are shown in **Table (4)**.

Mix Proportions and Casting

Mixing procedure and mix proportion that used throughout the work were according to ASTM C305-02 [26]. Mixing proportion and contents for each mix are shown in **Table (5)** while **Table (6)** was illustrated the details of mixtures used throughout the work. Trial mix were carried out to specify %flow equal to 110±5% at same water/cement ratio for four types of mixes (control, control+2% of fibers, control+6% of fibers, control+10% of fibers) by using different percentages of super plasticizer as shown in **Table (5)** according to ASTM C-1437-01 [27]. All mixtures types were casted into cubic molds of 5x5x5 cm³, prism molds of 4x4x16 cm³ and direct tension molds as shown in **Plate (2)**, after de-molding the specimens, the curing was applied, at the end of curing period the specimens were stored into the sealed bags until age of 28 days. Aging processes were applied after 28 days from casting. After de-molding the specimens were subjected to different types of curing according to the mixtures groups, as shown in **Tables (6)**.

CO₂ curing method

The specimens were oven dried for 30 min. at (50-60) °C after removed from the mold. The drying process is applied to evaporate the free water from pores and facilitates the CO₂ gas penetration through the specimen. Then the specimens subjected to the carbonation chamber and make sure that the upper cover is closed tightly to prevent gas leaking. Carbonation curing conditions used throughout this study were according to previous studies [5,13] and are including 50% of carbonation concentration, 2 hours of curing duration and 50 °C of chamber temperature. **Plate (3)** illustrates the instrument used and the produced specimens.

Autoclave curing

The specimens were subjected to the autoclave chamber after removing the specimens from molds. The procedure of autoclave curing consisted of adding 2 liter of distilled water into the chamber then putting the specimens inside the bearer and close the chamber tightly to prevent pressure leaking. After water vapor began to release, the vessel valve was closed and the pressure began to rise, then the pressure reaches 2 MPa and stabilized for 2 hours. This procedure applied according to ASTM C-151-00 [28]. **Plate (4)** illustrated the instruments used and specimens.

Evaluation methods

Compressive strength

The compressive strength test was measured by using cubic specimens with 50x50x50 mm³ as dimensions on perpendicular direction of the surface cast according to ASTM C109/C 109M-02 [29]. The test was obtained by digital concrete compression machines of ELE International company with loading rate of 1,5000 kN/ min. as well as this test machine is giving load in kN and the compressive strength in MPa.

Flexural strength

The flexural strength test was measured by using prism specimens with dimensions of 160 mm length, 40 mm width and 40 mm thickness. The specimen was subjected on simply supported over the length of the prism on perpendicular direction of cast and the load was applied in the center of the prism. The test was obtained by 10 kN Flexural/Tensile testing machine of ELE International company. This machine is given the maximum load of flexural process in kN and the flexural strength was calculated according to ASTM C293-02 [30]

Direct tensile strength

The direct tensile strength was measured by using dumb-bell briquette test specimens. The specimen was subjected in axial alignment into the test machine with pull direction and symmetrical position in the jaws. The test was obtained by 10 kN Flexural/Tensile testing machine of ELE International company with loading rate of (1 ± 0.5 mm/min). This machine is given the maximum load of tensile process in kN and the direct tensile strength was calculated according to BS-6319-7-85 [31]

X-ray diffraction (XRD):

The crystallographic structure of natural and manufactured materials can be reveal in detailed information by a versatile, non-destructive technique called X-ray diffraction (XRD). This test was attained by diffractometer (Model: XRD 6000 / Shimadzu / Japan) that includes a high voltage generator 3 kW X-ray tube with copper anode, running on at 40 kV and 30 mA, LYNXEYE detector, bearing an anti-scatter slit of 3 mm and a filter of Ni K-beta (0.5 %) and no monochromator. This test was achieved by Nanotechnology and Advanced Materials Research Center at the University of Technology.

Results and Discussion

Compressive strength

The better compressive strength values was obtained by CO₂ curing for fibrous and non-fibrous mixes, but the fibrous mixes were showed a reduction in compressive strength comparing with the plain mix by about (30%) for all fibers percentages.

However the compressive strength values of fibrous mixes for autoclave curing were shown an increment by about (50%, 20%, 5%) for fiber percentages (2%, 6%, 10%) respectively, but their values still less than CO₂ curing values as shown in Fig. (1). Also, it was noticed from **Fig. (1)** that compressive strengths of CM6 and CM10 were convergent.

Statistical analysis of the compressive strength results (Tables 7 and 8) suggests that the effects of curing are significant. The interaction of various variables (curing types and fiber) are seemed

also to be strong and significant. Furthermore the statistical effects of cellulose fiber percentage added are relatively small. This is probably due to conflict of fiber role combined with other factors.

Flexural strength

Although, the non-fibrous mixes were shown the better flexural strength values for both types of curing (CO₂ and autoclave) comparing with fibrous mixes, but the better flexural strength values of fibrous mixes were obtained by autoclave curing.

The fibrous mixes cured with autoclave curing were showed a reduction in flexural strength from the plain mix ranged between (11% to 23%), While for CO₂ curing ranged between (14% to 19%) for all fibers percentages as shown in Fig. (2)

Statistical analysis of the compressive strength results (Tables 7 and 8) suggests that the effects of curing are significant. The interaction of various variables (curing types and fiber) are seemed also to be strong and significant. Furthermore the statistical effects of cellulose fiber percentage added are significant.

Direct tension strength

Although, the non-fibrous mixes were shown the better direct tension strength values for both types of curing (CO₂ and autoclave) comparing with fibrous mixes, but the better direct tension strength values for plain and (10% of fibers) mixes were obtained by CO₂ curing, while for (2% and 6%) of fibers were obtained by autoclave.

The fibrous mixes cured with autoclave curing were showed a reduction in direct tension strength from the plain mix ranged between (16% to 25%), While for CO₂ curing ranged between (3% to 46%) for all fibers percentages as shown in Fig. (3)

Statistical analysis of the compressive strength results (Tables 7 and 8) suggests that the effects of curing are significant. The interaction of various variables (curing types and fiber) are seemed also to be significant. Furthermore the statistical effects of cellulose fiber percentage added are significant.

X-Ray diffraction

Fig. (4 a) shows the X-ray diffraction patterns for mix containing 10% of fibers cured with CO₂ and Fig. (4 b) for mix containing 10% of fibers cured autoclave. It was obvious from Fig. (4) that CO₂ curing was given the highest CaCO₃ and Ca(OH)₂ contents compared with autoclave and that considered a proven of carbonation process occurrence. The behavior of CO₂ curing consist on increment of CaCO₃ content due to the conversion of Ca(OH)₂ to CaCO₃ during the carbonation process that applied as curing for cellulose fibers cement composites [13].

CONCLUSIONS

1. CO₂ curing was the most effected curing for compressive strength for all mixes. Moreover it was generally noticed that whenever the fiber percentages increased, the compressive strength decreased (decreased by about 30%).
2. Autoclave curing leads to better performance in flexural strengths. Among diverse cellulose fibers content used, 6% and 10% lead to better flexural performance for autoclaved and CO₂- cured composites, respectively.
3. Both types of accelerated curing effects on direct tension performance were shown to be fluctuated. This could be attributed to the variation in cellulose fibers role with respect to each property evaluated. Among diverse cellulose fibers content used, 10% leads to better direct tension performance for both autoclaved and CO₂- cured composites.

4. For non-fibrous (plain) mortar specimens, application of CO₂ curing improves compressive and direct tension strengths more significant than autoclaved specimens. In contrast, non-fibrous autoclaved specimens perform better in flexural performance, compared with CO₂-cured specimens.

5. X-ray diffraction test shows that carbonation process was took place and causing an accelerated curing for composites. CO₂ curing leading to increased CaCO₃ content (intensity =4300 CPS) compared with autoclaved composites (intensity =2000 CPS).

Table 1: Physical properties of cement*

Physical properties	Test results	Limits of (IQS NO.5/1984)
Fineness (Blaine Methods),m ² /kg	376	≥230
Setting time (Vicat's Method) hrs:min		
Initial	2:05	≥45min
Final	4:00	< 10hrs
Soundness using Autoclave Method%	-0.12	≤0.8
Compressive strength, MPa		
3 Days	20.0	≥15
7Days	25.0	≥23

*Tests results were carried out by National center for Construction Laboratories and Researches (NCCLR)

Table 2: Physical properties of fine aggregate*

Physical properties	Test results	Limits of (IQS NO.45/1984)
Specific gravity	2.6	-
Bulk Density(kg/m ³)	1729	-
Sulfate content%	0.343	≤0.5%
Absorption%	2	-

*Tests results were carried out by National center for Construction Laboratories and Researches (NCCLR).

Table 3: Physical properties of date palm fibers

Property	Value
Average length (mm)	5.81
Average diameter (mm)	1.96
Aspect ratio (%)	2.96
Dry density (kg/m ³)	269
Saturated density (kg/m ³)	1.082
Moisture content to saturation point (%)	81.3%

Table 4: Organic constituents of date palm fibers*

Type of fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Ash (%)
Treated with 1 % of NaOH	21.673	21.247	30.463	2.123

*Chemical tests were done by the College of Agriculture in Bagdad University

Table 5: Mixing proportion.

Materials	Percentages	Contents
Cement	C: S= 1:2.75	450 gm
Sand		1375 gm
Water	W/C= 0.485	242 ml
Silica fume	10% by Wt. of cement	50 gm
Fibers	2% by Wt. of cement	10 gm
	6% by Wt. of cement	30 gm
	10% by Wt. of cement	50 gm
Superplasticizer For control mix	0.9% by Wt. of cement	4.5 ml
For 2% of fibers mix	1.1% by Wt. of cement	5.5 ml
For 6% of fibers mix	1.7% by Wt. of cement	8.5 ml
For 10% of fibers mix	2.1% by Wt. of cement	10.5 ml

Table 6: Details of the experimental program *

Groups	Mix symbol	Description	Curing type
1	CM	Control	CO ₂
	CM2	2% of fibers	
	CM6	6% of fibers	
	CM10	10% of fibers	
2	AM	Control	Autoclave
	AM2	2% of fibers	
	AM6	6% of fibers	
	AM10	10% of fibers	

*All fiber percentages were taken by weight of cement or each type of mixtures.

Table 7: Statistical analysis of compressive, flexural and direct tension strengths test results of fiber percentages effect.

	Compressive Strength				Flexural Strength				Direct tension Strength			
	0%	2%	6%	10%	0%	2%	6%	10%	0%	2%	6%	10%
0%	-				-		*	*	-	*	*	
2%		-				-			*	-		*
6%			-		*		-		*		-	*
10%				-	*			-		*	*	-

-: Statistically insignificant difference.

*:

Statistically significant difference at 95% level of confidence.

Table 8: Statistical analysis of compressive, flexural and direct tension strengths test results of curing type effect.

	Compressive Strength		Flexural Strength		Direct tension Strength	
	CO ₂	Autoclave	CO ₂	Autoclave	CO ₂	Autoclave
CO ₂	-	*	-	*	-	
Autoclave	*	-	*	-		-

-: Statistically insignificant difference.

*: Statistically significant difference at 95% level of confidence.



Plate 1: Fibers of wood date palm were used.



Plate 2: Specimens after casting



Plate 3: (a): control panel of system, (b): CO₂ bottle supply, (c): O₂ bottle supply, (d): valves of CO₂, O₂ and vacuum, (e): carbonated specimens.

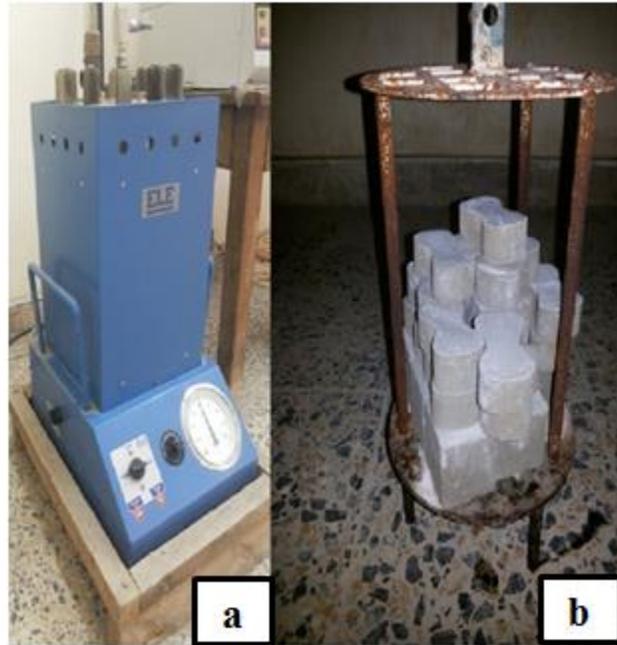


Plate 4: (a): Autoclave chamber, (b): specimens inside the autoclave bearer

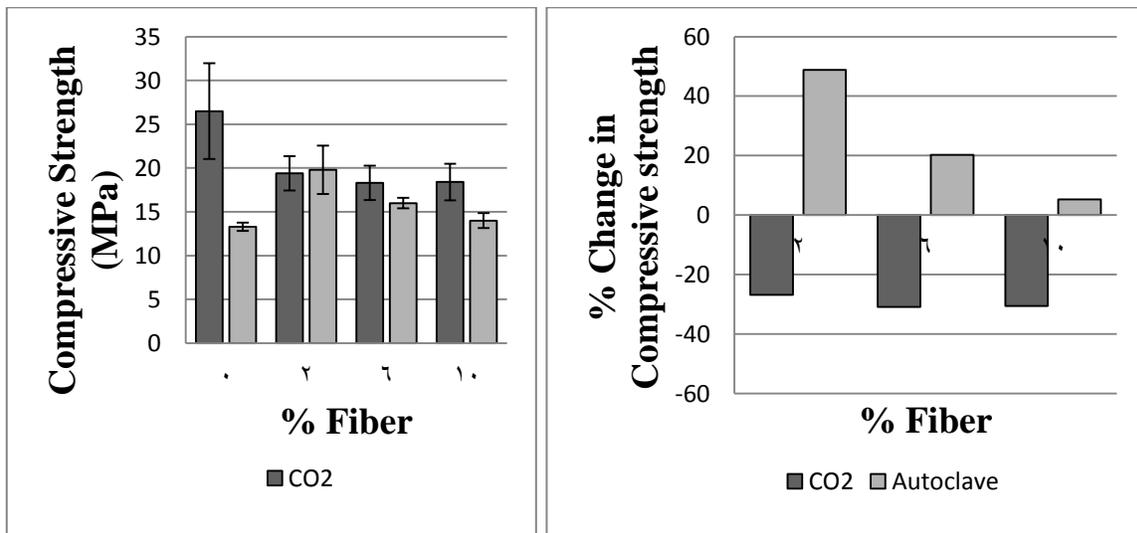


Figure 1: Compressive strengths and change in compressive strengths from plain

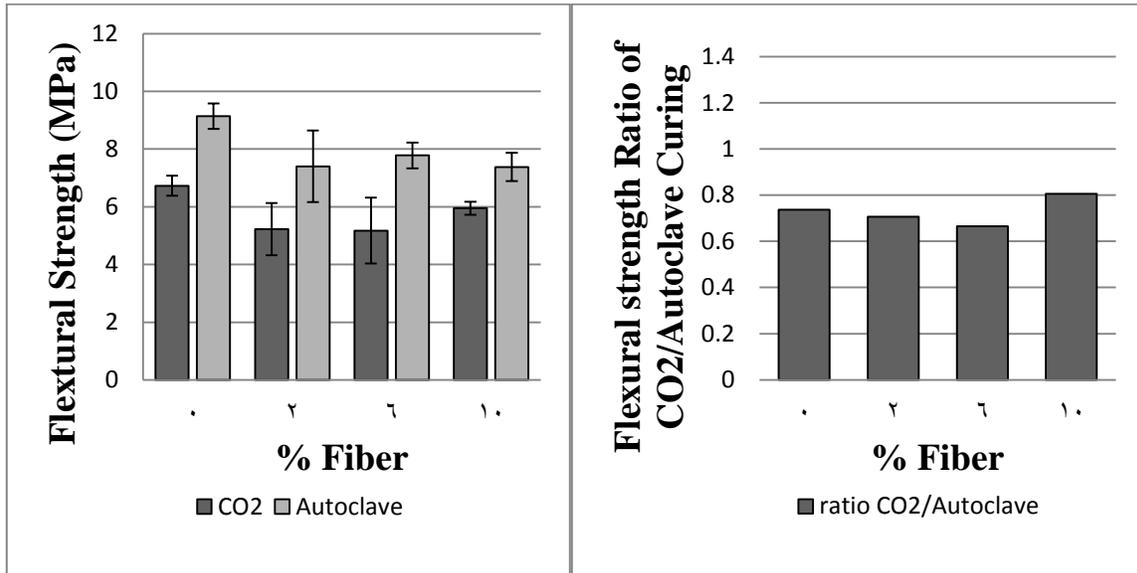


Figure 2: Flexural strengths and ratio of CO₂ flexural strength to Autoclave flexural strength.

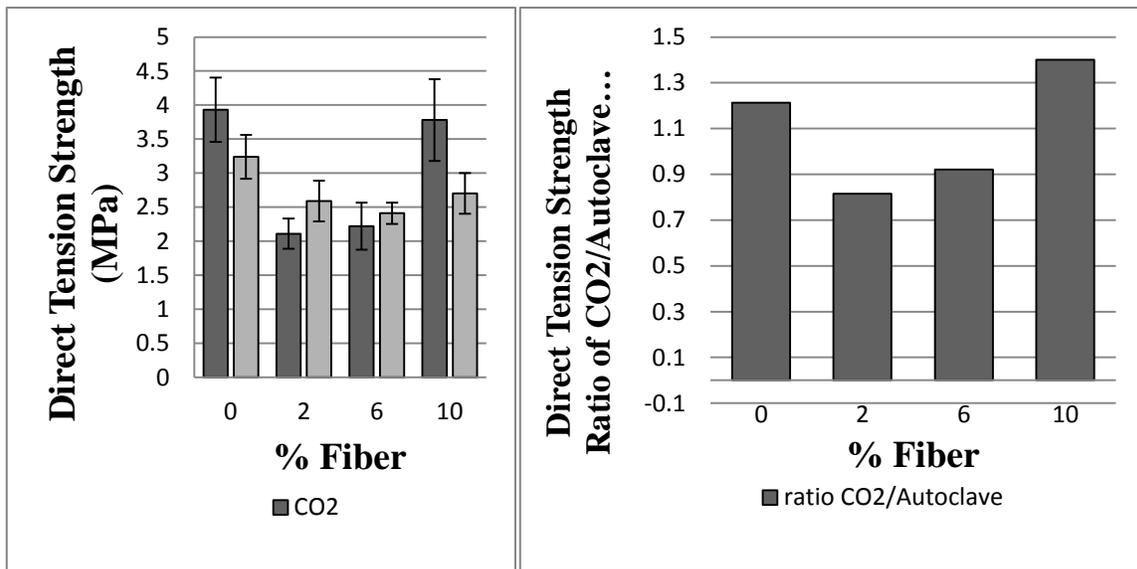


Figure 3: Direct tension strengths and ratio of CO₂ direct tension strength to Autoclave direct tension strength.

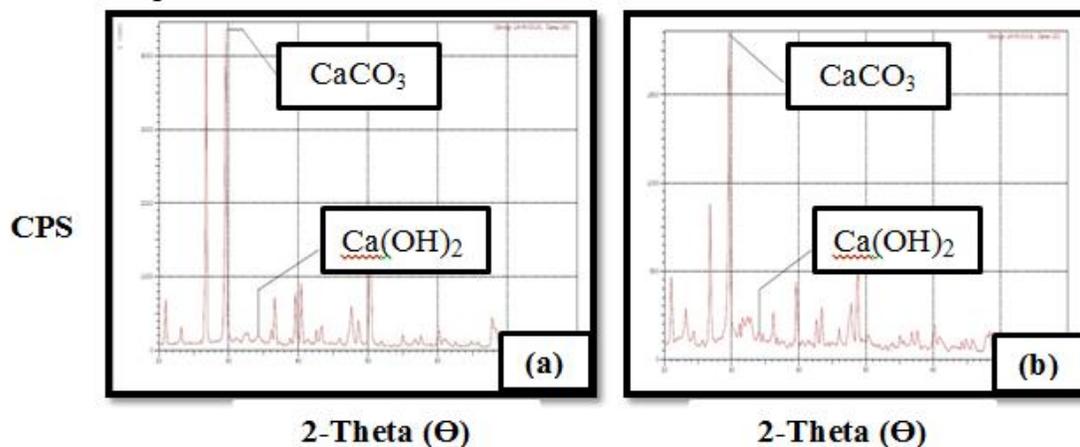


Figure.4: X-ray diffraction patterns for (a): CM10 and (b): AM10.

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