

Research Article

Air-Cured Fiber-Cement Composite Mixtures with Different Types of Cellulose Fibers

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This present study was carried out to check the feasibility of different cellulose fibers obtained from cropped virgin cellulose, bleached eucalyptus, and araucaria pulps through different new environmentally friendly curing processes for fiber-cement production. The aim is to introduce the different sources of cellulose fibers with lower cost to produce the “fiber-cement without autoclave” (FCWA). The slurries used in the experiments contain approximately 8% wt. of cellulose. The influence of the waste marble powder addition to the cement mixture was also studied. The physical and mechanical properties of the products which were prepared with this method under different curing conditions were investigated. The mechanical properties of eucalyptus cellulose appear to offer the best combination, especially after longer air-cure cycles. The results showed that the production of FCWA is very economical by using waste marble powders. And moreover, two new types of cellulose fibers (eucalyptus and araucaria celluloses; EuC and ArC, resp.), which provide a better density and packing in the fiber-cement leading to better modulus of rupture (MOR) and modulus of elasticity (MOE) values as virgin cellulose (ViC), are very usable for production of the fiber-cement in industrial scale.

1. Introduction

Cellulosic fibers contribute to the development of high-quality fiber-reinforced cement composites [1–4]. Fiber-reinforced cement (FC) is used in a wide range of applications from building facade cladding systems to advertising and promotional and decorative applications, and it is becoming increasingly common throughout the day. Information on the use of natural fibers as reinforcements in composites has increased in the 2000s [5, 6]. Natural fiber-based fiber-cement sheets are used in all kinds of interior and exterior coverings. Flat-shaped and wooden-patterned fiber-cement panels are hardened in autoclaves. FC products are produced in two different production methods in the industry. The first method is hardening with high pressure and temperature in autoclaves, and the other one is in nonautoclave systems. The

fiber-cement produced by nonautoclave systems has been termed as “fiber-cement without autoclave” (FCWA). Prescription to be used in production of FC: there are two different curing methods. First one is air/humid curing, and the other curing is at high-temperature and high-pressure conditions in autoclaves [7]. Autoclave production method is well known across the world, and commercially, they are widely used in the construction industry [8]. When producing FCWA, the synthetic PVA (polyvinyl-alcohol) fibers are generally used due to increasing distortion and abrasion resistance [9]. PVA fibers have been used in dramatically low amounts, <1% in this study, as compared to the content of total cellulosic fibers. Environmentally friendly cellulosic fibers, derived from renewable materials, are produced with a low-cost fiber-cement production method, air-curing in this study. General practice, however, involves

use of autoclaves, processing at high temperatures which increases cost in manufacturing of fiber-cement.

Wood fibers have many advantages such as nonhazardous, relatively low costs, biodegradability, renewability, and recyclability. They are also widely used in cementitious composites due to easy manufacturing process [10]. Wood fibers are examined in two categories such as softwood and hardwood fibers, which have long and short length, respectively. Characteristically, eucalyptus as a hardwood fiber has short fiber length and is dispersed better than pinus which is known as a softwood fiber with long fiber length in the sludge matrix and contains more cellulose fibers per unit volume [10, 11]. Therefore, the amount of paper clay obtained from one gram of softwood fiber is greater than the amount of paper clay obtained from one gram of hardwood fiber [12]. Eucalyptus can be used to produce the FCWA materials which are cheaper than other fiber cements in the market, and raw materials are easily available. FCWA is harmless since it contains renewable materials [13].

FC materials offer several advantages compared to conventional construction materials. These advantages are widespread resource availability, high fiber strength, advanced fiber extracting technology, and relatively low cost [14]. The primary problem in the production of the fiber cement by air-cured nonautoclave system is the increase of the material and manufacturing costs [15]. The most important reasons for the increase of the production cost in the conditions of Turkey are the high cost of virgin cellulose (ViC) used as raw material and autoclave costs. In this study, eucalyptus and araucaria celluloses (EuC and ArC, resp.), which are cheaper than relatively virgin cellulose, were produced without autoclaving in order to reduce the cost of raw materials. The characteristics of the produced FCWA products were also compared. PVA is used in minimal proportions, and its strength contribution is assumed by other fibers in the system.

2. Experimental

2.1. Materials. Three different types of American (Brazilian and Canadian) and European (Spaniard, Portuguese, and Swedish French) fibers were selected to produce the fiber-cement materials. Virgin, eucalyptus, and araucaria celluloses (ViC, EuC, and ArC) were used in this study as different cellulose sources. And the recycling water (pH = 13) was used to prepare the mixture. The virgin, eucalyptus, araucaria celluloses, and PVA fibers were supplied by Cellulose Lerox International, Kombassan, and Kordsa. Powder materials such as cement CEM I 42.5 R and waste marble powder (WMP) were supplied by Cimsa and Durmus Mermer which is a producer in the Bilecik region.

2.2. Characterization of the Raw Materials. The chemical analysis of the raw materials, cement, and waste marble powders used in the experiments was performed by XRF analysis. Particle size and distribution of the raw materials were measured by Mastersizer from Malvern Instrument. The specific surface area (SSA) of the raw materials was determined by Quanta Chrome Autosorb 1B using the nitrogen adsorption BET method. The structure and morphology of the raw

materials, fibers, and products were investigated using a Philips XL30 SFE scanning electron microscope (SEM). Samples were dried first and then coated with gold. Each sample was examined with the SEM at different magnifications. The crystal structure of the materials was identified by X-ray diffraction (XRD) recorded on a Rigaku Dmax 2200 diffractometer using Cu-K α radiation with a characteristic wavelength of $\lambda = 1.5406 \text{ \AA}$ ($2\theta = 10\text{--}80^\circ$) at room temperature.

2.3. The Properties and Microstructural SEM Analysis of the Raw Materials: Fibers, Cement, and Waste Marble Powders. The chemical XRF analysis of the cement material and waste marble powders (WMPs) is shown in Table 1. WMP contains CaO as main phase only, but the cement contains the main phases CaO (63.50%), SiO $_2$ (19.35%), and Al $_2$ O $_3$ (4.74%). The XRD analysis results of WMP and the cement material, shown in Figure 1, are in accordance with the XRF analysis (Table 1). This means that WMP consists of calcium carbonate, and the used cement materials consist of calcium silicate, aluminium silicate, and alumina as crystalline phase.

Particle size and size distribution analysis of WMP and cement is given in Figure 2. The median sizes (d_{50}) of the cement and WMP were measured as $16.08 \mu\text{m}$ and $5.52 \mu\text{m}$, respectively, as shown in Figure 2. The measuring results of the specific surface area of the cement and WMP are $1.23 \text{ m}^2\cdot\text{g}^{-1}$ and $2.17 \text{ m}^2\cdot\text{g}^{-1}$, respectively. It is an expected result that the cement material with large grain size has a specific surface area less than WMP.

The SEM images of the CEM I 42.5 R cement from Cimsa and PVA fibers from Kordsa used in the experiments are shown in Figures 3(a) and 3(b). The scaled SEM images of the natural fibers, papers, and pulps according to the cellulose types were taken to determine the structure and diameters of natural fibers (ViC, EuC, and ArC) as shown in Table 2. The diameters of the natural fibers (ViC, EuC, and ArC) and also the average diameters/thickness were determined as shown in Table 3. Table 4 shows the measured freedom degrees of natural fibers (ViC, EuC, and ArC). Moisture measurements of powder materials, PVA, and all kinds of fibers were determined using a moisture analyzer. The results are shown in Table 5.

3. Preparation of the Samples

Different ratios of fiber have been tried in the prescription. The fiber ratio was tried between 2 and 12% and optimized to 8% [16]. The ratio of water used is between 25 and 40% in many concrete works. The water/cement ratio is optimized to 30%, and these ratios were used in these experiments [17]. The cellulose pulps, PVA, cement, waste marble powder (WMP), and recycling water with the pH value of 13 were used in the desired proportions as shown in Table 6 to prepare the FCWA slurry used for the production of the fiber-cement composite materials in the experiments.

First step of the production is preparing mixture of the fiber-cement sludge. After that, the sludge is poured into the vacuum box. The excess water in the slurry is discharged out of the press with the help of vacuum pump. Following the

TABLE 1: XRF analysis of powder materials.

Number	Component	Cement	WMP
1	Na ₂ O	0.38	—
2	MgO	1.88	1.69
3	Al ₂ O ₃	4.74	—
4	SiO ₂	19.35	0.27
5	P ₂ O ₅	0.08	—
6	SO ₃	2.72	—
7	K ₂ O	0.53	—
8	CaO	63.50	54.27
9	TiO ₂	0.36	—
10	Cr ₂ O ₃	0.11	—
11	MnO	0.08	—
12	Fe ₂ O ₃	3.29	0.22
13	LOI	3.01	43.55

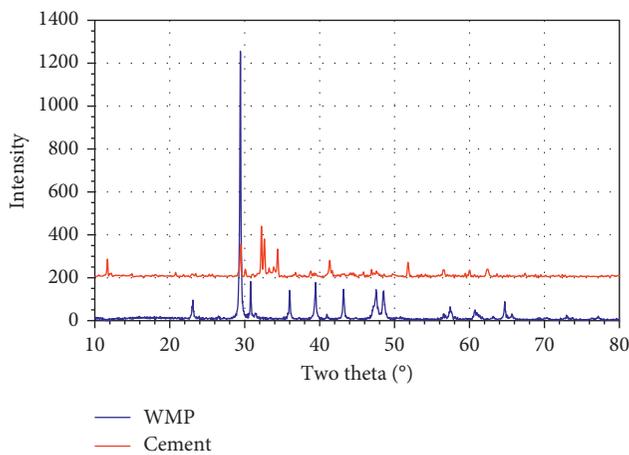


FIGURE 1: XRD analysis of the raw materials.

vacuuming process, the fiber-cement samples are placed in the press machine and compressed with a pressure of 7 MPa for 120 seconds. Thus, 10 specimens were produced for each fiber type. Figure 4 shows the flow chart of the fiber-cement production. All prepared samples were cured in the air by itself. However, the samples were allowed to cure after curing the samples at 30°C for 6 hours in order to maintain the stability of each sample.

4. Tests on the Materials: Freeness Test

The degree of freedom is defined as the measurement of the drainage rate at the discharge of the wood pulp suspension (3 g of pulp in 1 L of water). The drainage rate and the degree of freedom help determine the behavior of the fibers in the cement matrix or determine the fiber elasticity of the fibers or fine particle content of the fiber. According to the results of the measurements, stock preparation depends on the temperature, surface properties, and water quality [18, 19]. It is often referred to as Canadian Standard Freedom (CSF) because it was developed by the Canadian Cellulose and

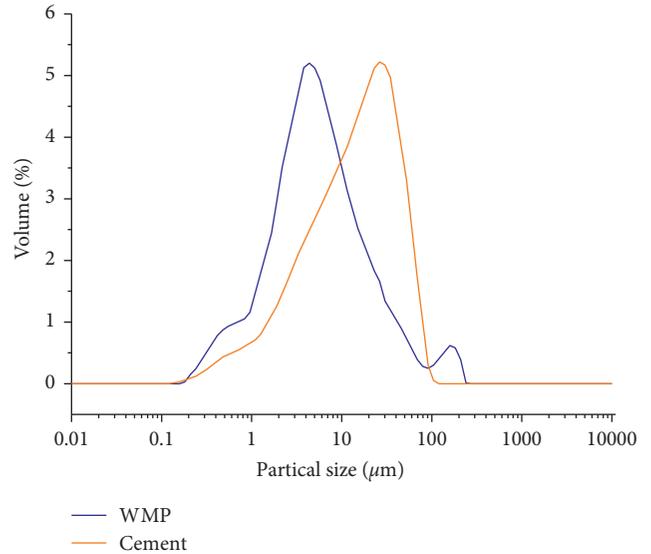
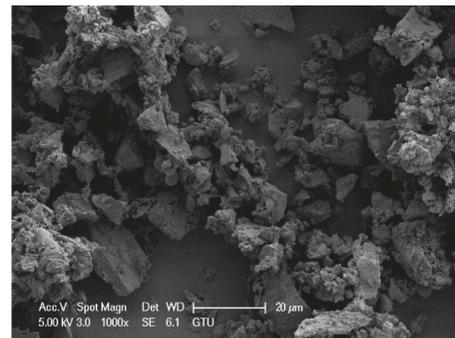
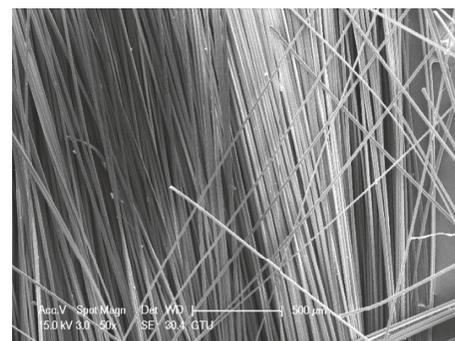


FIGURE 2: Particle size distribution of cement (Cimsa) and waste marble powder (Durmus Mermer).



(a)

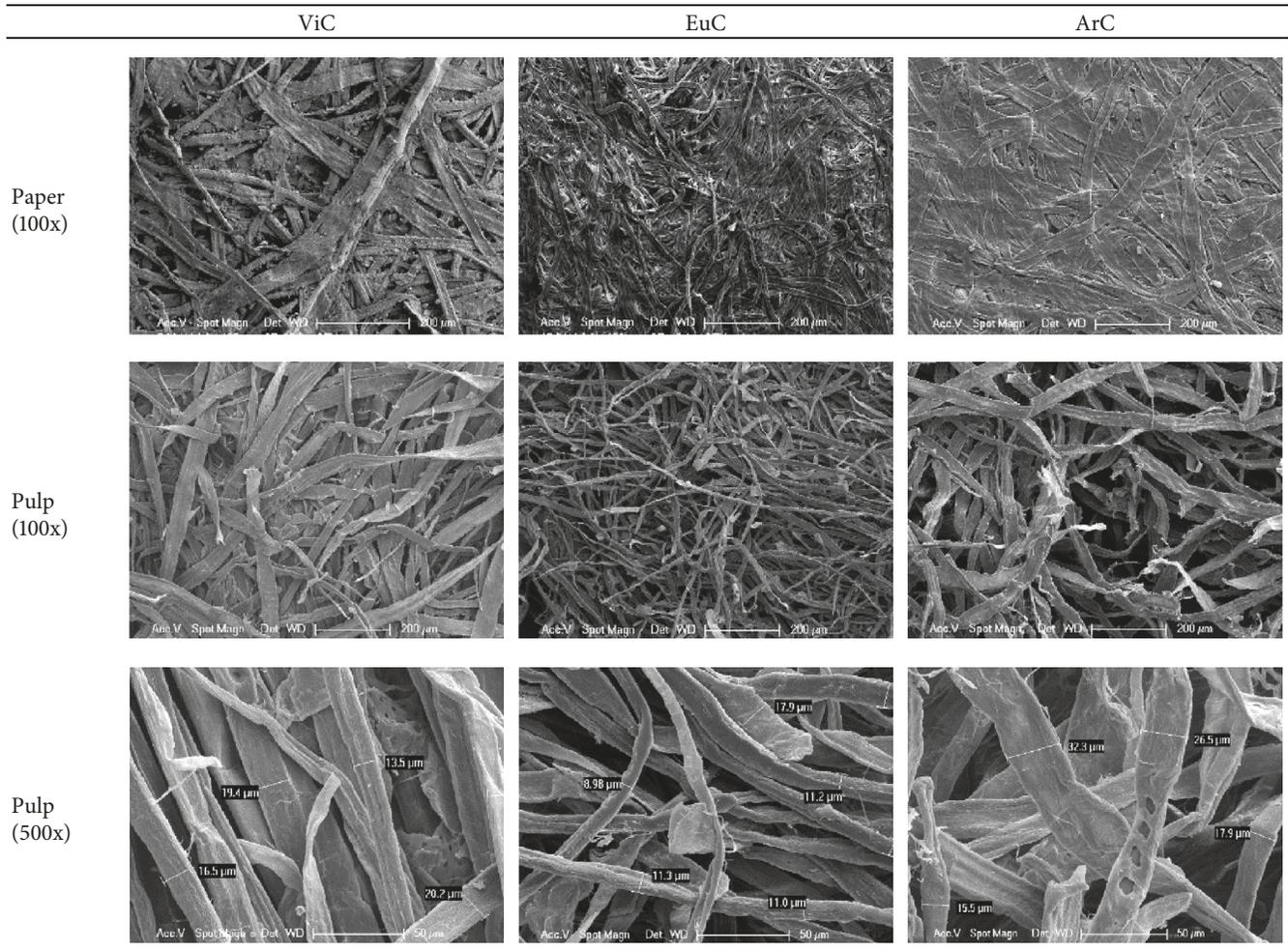


(b)

FIGURE 3: SEM micrographs of CEM 1 42.5 R cement (a) and of PVA fibers (b).

Paper Research Institute [18, 19]. Figure 5 shows a test machine according to the CSF. The freeness degree of celluloses pulp such refined fibers as ViC, EuC, and ArC was measured using a CSF, according to ISO 5257/2. In this study, the degree of freedom and drying rate of ViC, EuC, and ArC are measured.

TABLE 2: SEM micrographs of papers and pulps according to the cellulose types.



5. Tests on the Materials: Three-Point Bending, Strain-Stress Test

The effects of the fiber-cement produced without autoclaving on the physical and mechanical properties of all three fiber types were investigated. All fiber types were used 8% wt. in the mixtures to investigate differences in fiber's physical properties. The modulus of rupture (MOR) and modulus of elasticity (MOE) were determined using the three-point bending configuration for the mechanical characterization of the produced fiber-cements. Tests were performed using the Intelli Jack 6 kN device. The equations required to calculate the modulus of rupture (MOR) and the modulus of elasticity (MOE) with the three-point bending configuration are given in (1). Five flexural specimens were tested for each composite formulation during the experiments:

$$\text{MOR (MPa)} = \left(\frac{L_{\text{Max}}}{b \cdot h^2} \right) \cdot (S_{\text{down}} - S_{\text{up}}),$$

$$\text{MOE (GPa)} = tg\alpha \cdot \left(\frac{L_{\text{Max}}}{\delta} \right) \cdot \frac{(S_{\text{down}} - S_{\text{up}})^3}{b \cdot h^3} \times 10^6, \quad (1)$$

TABLE 3: Fiber wideness according to SEM analysis.

	ViC	EuC	ArC	
Average thickness of cellulose (μm)	L_{Avr}	30.34	9.79	43.55
	L_{Max}	48.27	21.7	59.1
	L_{Min}	17.2	4.18	27.8

TABLE 4: Freeness of fibers.

Properties	ViC	EuC	ArC
Drainage rate	30–50	73	68
% drying rate	80 ± 2	94.1	93.52
% humidity	3.98	5.9	6.48

where L_{Max} is the maximum stress of the linear portion of the stress-strain curve, $(S_{\text{down}} - S_{\text{up}})$ is the major span, b and h are the specimen width and thickness, respectively, $tg\alpha$ is the initial slope of the stress-strain curve, and α is the deflection of the composite. The mechanical properties of the samples were measured 7, 15, and 28 days after the production of the sheet [9].

TABLE 5: Drying mass and humidity of components.

Samples	% drying mass	% humidity
PVA	97.2	2.8
Waste marble powder (WMP)	99.2	0.8
CEM 1 cement (Cimsa)	100	0.0

TABLE 6: Usage rates of raw materials.

Raw materials	Mass rate (%)
Celluloses (eucalyptus, araucaria, and virgin cellulose)	8.0
PVA	0.6
CaCO ₃ (reduce of marble powder)	31.4
Cement (Cimsa CEM I)	60

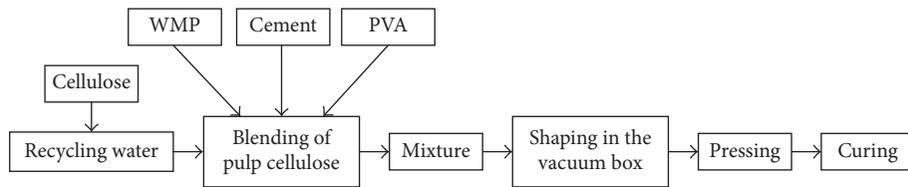


FIGURE 4: The production flow chart of fiber-cement sheets.

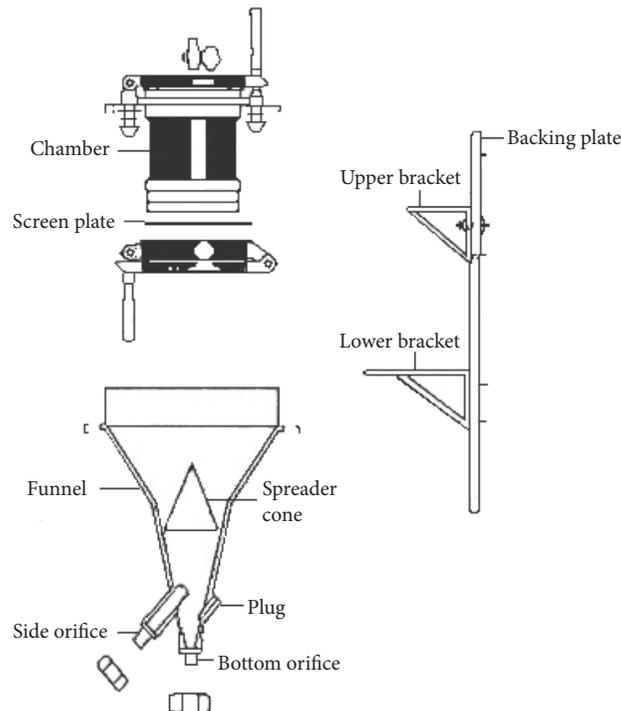
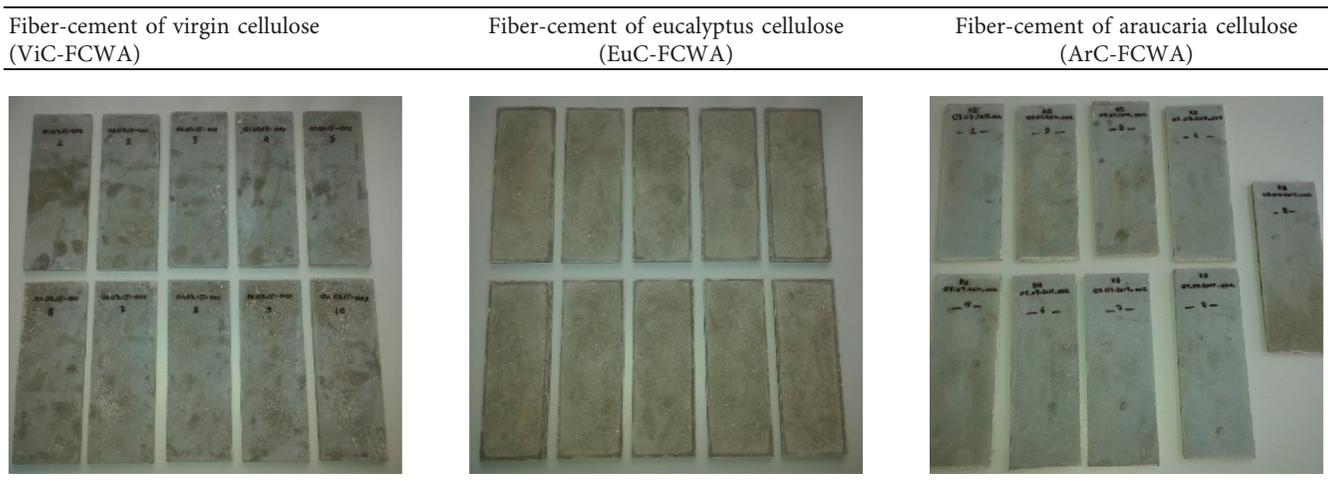


FIGURE 5: Freeness test machine [18].

TABLE 7: Nonautoclave products: ViC-FCWA, EuC-FCWA, and ArC-FCWA.



6. Tests on the Materials: Determination of Bulk Density and Water Absorption

Archimedes test was performed to determine the density of the produced samples. Their bulk density and water absorption values were measured. The samples were dried in the oven until their weight get stable and scaled first (W1). Thereafter, the samples were kept in the water for 48 hours and waited until they had not changed their weight and the samples get scaled in the water with the Archimedes scale (W2). The samples were taken out from water to determine their saturated weights (W3). The bulk density and water absorption values of the samples were determined by the following equations:

$$\text{Bulk density} = \frac{W1}{(W1 - W2)} \times 100, \tag{2}$$

$$\text{Water absorption \%} = \frac{(W3 - W1)}{W1} \times 100,$$

where W1 is the dried weight in the oven, W2 is the weight in water, and W3 is the saturated weight (the surface of the sample removed from the water is weighed in the air by wiping with a moist cloth).

7. Results and Discussion

The densities of the produced samples were measured: dry density is 1.4g/cm³ and saturated samples density is 1.8g/cm³ according to ASTM C1185 standard [13]. The dimensions of these fiber-cements produced in the laboratory are measured as 210 mm × 76 mm × 8 mm. Table 7 shows the plates of the produced fiber cements in the laboratory environment.

The effects of the fiber-cement produced without autoclaving on the physical and mechanical properties of all three fiber types were investigated. All fiber types were used 8% wt. in the mixtures to investigate differences in fiber's

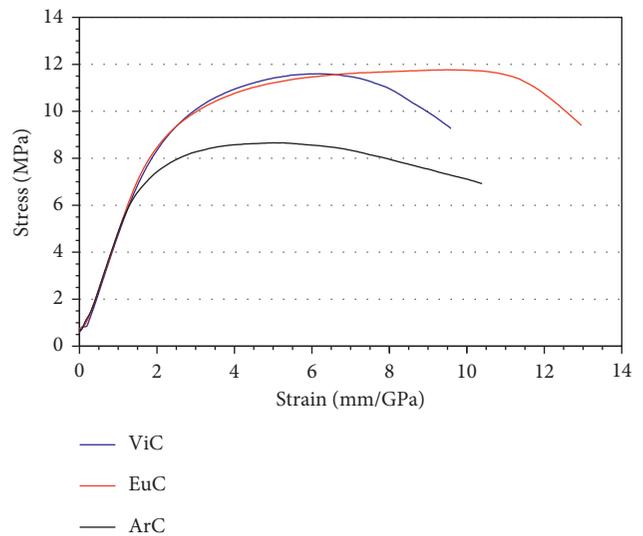


FIGURE 6: Stress-strain measure.

TABLE 8: Mechanical result of produced samples.

		ViC-FCWA	EuC-FCWA	ArC-FCWA
7-day results	MOR (MPa)	11.24	11.94	13.13
	MOE (GPa)	4.70	4.65	5.43
15-day results	MOR (MPa)	9.76	13.97	12.16
	MOE (GPa)	3.79	5.76	5.66
28-day results	MOR (MPa)	9.76	17.57	17.39
	MOE (GPa)	3.79	5.28	4.89

physical properties. Stress-strain measure graphic appears in Figure 6. It is seen that all fiber cements have the same stability under the strain up to 2 GPa. But, after 2 GPa, EuC and ViC have similar values of stress, while ArC has a lower value. EuC and ViC exhibit the same properties according to the stress-strain test. Stress-strain diagram is shown in Figure 6.

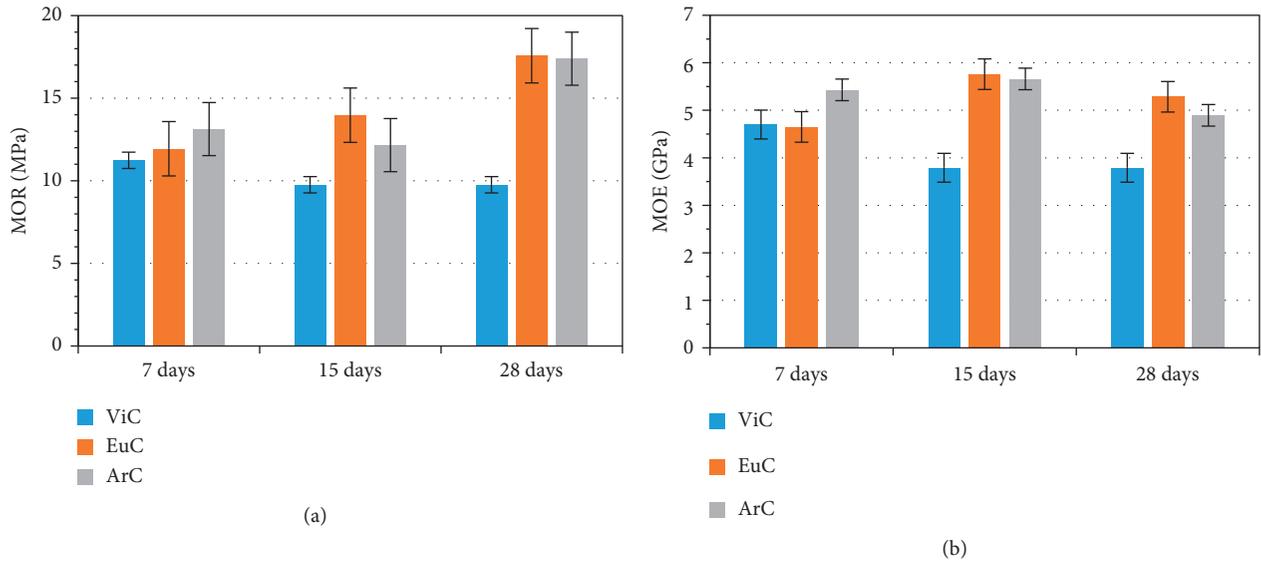
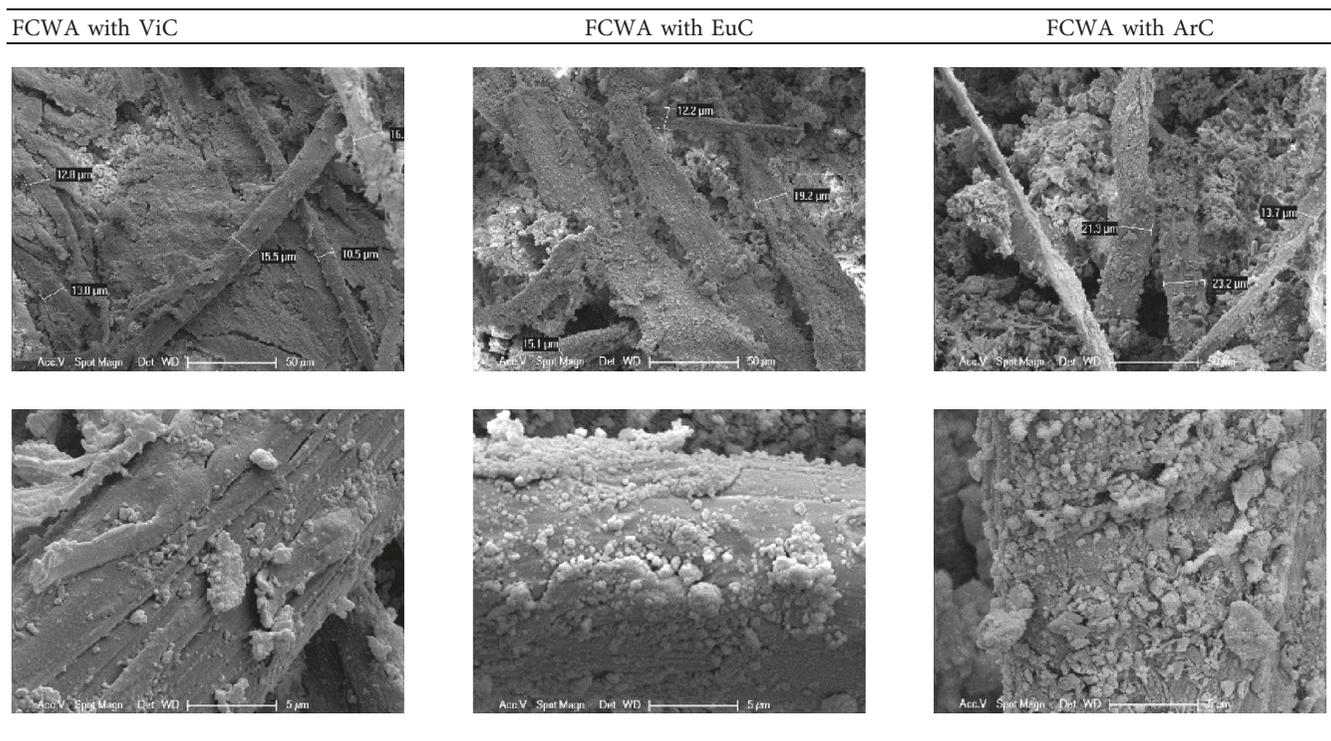


FIGURE 7: The mechanical properties of FCWA according to cellulose fiber type: (a) modulus of rupture and (b) modulus of elasticity.

TABLE 9: SEM images of the produced samples with magnification of 500x and 5000x.



In Table 8, mechanical property test results for samples cured for 7, 15, and 28 days are provided. It was observed that MOR and MOE values of the ViC-FCWA samples remained relatively constant, but a slight drop in properties was observed from 7-day to 15-day and 28-day processes. The rupture values of EuC-FCWA samples were observed to increase from 11.94 MPa, 13.97 MPa, and 15.57 MPa, with MOE values rising from 4.65 GPa to 5.76 GPa and stabilizes

at 5.28 GPa for 7-, 15-, and 28-day curing, respectively. The MOR values of ArC-FCWA samples were observed to be 13.13 MPa, 12.16 MPa, and 17.39 MPa for 7-, 15-, and 28-day curing, respectively. There was a significant improvement in the MOR value after the 28-day process. MOE values of ArC-FCWA samples were 5.43 GPa, 5.66 GPa, and 4.89 GPa, respectively. Figure 7 shows a graphical representation of the MOR and MOE values. There was an appreciable

TABLE 10: 28-Day results of FCWA products.

	ViC-FCWA	EuC-FCWA	ArC-FCWA
Humidity (%)	9.00	8.90	9.60
Bulk density (g/cm ³)	1.52	1.55	1.56
Water absorption (%)	14.00	14.62	14.89

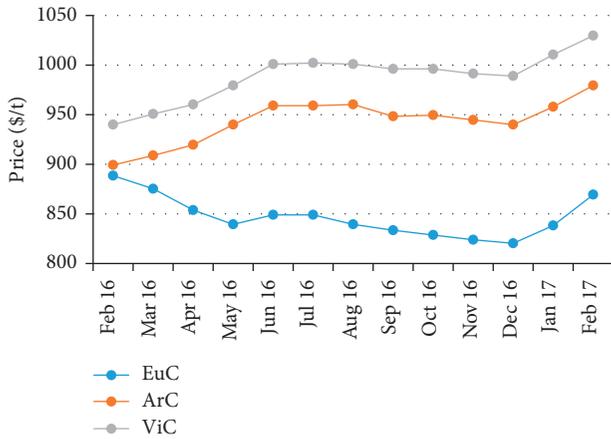


FIGURE 8: The tonnage cost of cellulose types.

improvement in MOR values as the cure duration increased in the process.

Microstructural analysis was performed on the specimens by taking SEM images of the produced fiber-cements. The morphology, size, and distributions of natural fibers are shown in Table 9. It has been observed that the cement and fibers have very good interaction, the fibers are homogeneously distributed in the matrix of the cement, and also the fibers have partly lamellar structure.

In Table 10, the bulk density of the ViC-FCWA sample was measured as 1.52 g/cm³, and the water absorption was 14%. The value for EuC-FCWA sample was 1.55 g/cm³, with the water absorption of 14.62%. The density value of ArC-FCWA sample was 1.56 g/cm³, while water absorption was 14.89%. The humidity of ViC-FCWA, EuC-FCWA, and ArC-FCWA was determined to be 9.0, 8.9, and 9.6, respectively.

In addition, the energy consumed by the autoclave in production increases the cost considerably. In this study, it has been shown that fiber-cement can be produced without autoclaving. However, this process increased the curing time of the products. According to the results of physical and mechanical tests, the properties of fiber-cement are mainly influenced by properties of the fibers. The mechanical strengths of eucalyptus and araucaria cellulose were found to be close to each other, which is considerably higher than virgin cellulose, for the cure time of 28 days. When comparing the desired properties with cost of the fiber-cement, the ones produced using eucalyptus will be more advantageous in terms of cost and mechanical performance. The cure optimization must be done based on different fiber types for optimal properties of fiber-cement products.

The biggest problem in fiber-cement technology is increasing material and manufacturing costs [15]. Approximately,

40% of the fiber-cement cost is the cellulose cost of the producers. The tonnage costs of cellulose in US dollars are shown in Figure 8 [20]. However, the cellulose prices may vary depending on the countries where they are produced and the logistics distance.

8. Conclusion

It was found from our studies that mechanical properties of FC produced using EuC and ArC are superior to ViC, especially after longer air-cure cycles. This study proposes more economical FCWA method and two new types of cellulose fibers for fiber-cement. It was believed that finer structures of EuC and ArC fibers compared to ViC provide a higher density and packing in the fiber-cement leading to better MOR and MOE values. EuC-reinforced fiber-cement offers the optimal solution in terms of shorter cure time and best mechanical properties. As a recommendation, it would be explanatory to try different curing conditions according to different fiber types.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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