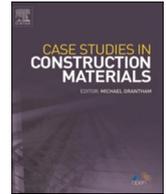




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Short communication

Evaluation of refined cement-based matrix systems for extrusion of wood fiber cement

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ABSTRACT

Different cementitious matrix formulations incorporating various chemical, polymeric and mineral additives were evaluated for use in extruded fiber cement products. These alternative formulations were designed to enhance the end product performance characteristics under aging conditions and to facilitate the extrusion process. The impact of different matrix formulations on mechanical, durability and physical characteristics of extruded cellulose fiber cement products were compared. Test results show that most of the additives lowered the bulk specific gravity but increased the moisture absorption capacity of extruded fiber cement products when compared with the basic mix formulation. Generally, the additives caused drop in flexural strength but did not significantly affect the flexural toughness of the matrix formulations. Upon saturation the frictional pull-out of swollen fibers increased the ductility (deformation) capacity of extruded cellulose fiber cement products. These results provide further insight into performance characteristics of extruded cellulose fiber cement products, and help with selection of matrix mix formulations to meet performance requirements of a particular application.

1. Introduction

The use of natural fibers (vegetable and wood) for the reinforcement of cement-based matrix offer many advantages, such as cost effectiveness, ease of availability, environmentally-friendly use, and having renewable source when compared with synthetic fibers [1–4]. These fibers also offer good mechanical characteristics that suite to overcome the limitations of the cement-matrix and improve some of its key attributes namely; compressive strength, tensile strength, toughness, rheological properties and resistance to early age cracking caused by thermal and impact loadings [5–10]. However, it has been observed that significant degradation occurs in cellulose fibers in the high alkaline environment of hydrated cement in addition to the loss of fiber-matrix bond with the passage of time [11,12]. To overcome these issues, some techniques have been successfully employed to enhance the durability and mechanical characteristics of cellulose fiber-based cement-composites. These include carbonation of the matrix and the use of supplementary cementitious materials to lower the alkalinity of the cement matrix thereby making it more suitable environment for the natural fibers [11,13,14].

On the other hand, extrusion presents an alternative mass production system for processing of wood fiber cement products. A number of the shortcomings of the 100-year-old Hatschek and other conventional processing methods can be overcome by adopting

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the versatile processing method of extrusion for the development of wood fiber cement composites. These shortcomings include limited freeze-thaw durability, serious limitations on profile and thickness, high sorptivity and permeability, and esthetics [15–19]. The diversity and high performance of extruded products promise to provide a strong support for continued growth of wood fiber cement in traditional and new markets. The extrusion process, widely practiced within the ceramic and plastic industries [20,21], offers attractive attributes as an alternative to the traditional wet and dry processes of wood fiber cement production. The extrusion process consists of forcing a highly viscous plastic mix through a shaped die. The auger-type extruder is capable of continuous mass production. It consists of a pug mill, a deairing section, and a compaction chamber followed by a die [22,23]. Pug mill provides a high-shear mixing action which squeezes the mix between the blades and wall of pug mill. The mixture then enters a deaired chamber that uses auger motion plus an applied vacuum to remove as much air as possible. The mix finally moves to the compaction chamber where the auger motion precompacts the mix to remove as much void space as possible prior to extrusion through a shaped die. The plasticized mixture is then forced under high pressure through the shaped die. The end product of desired shape is then cut to targeted length and cured.

The extrusion process places particular demands on fresh mix qualities [7,24,25]. The process requires a relatively viscous mix which is highly resistant to segregation (loss of moisture) when compressed during extrusion. The fresh mix should further provide relatively low friction at interfaces with the extruder die to facilitate the process. For the purpose of extrusion, the fresh mix should assume characteristics approaching those of dough, which limits the amount of water which could be used in fresh cement-based mixtures. Yet another requirement concerns the integrity of fresh mix as it exits the die. In order to meet these requirements on fresh mix performance while providing cured end products of high performance, special additives should be used in cement-based mixtures which are amenable to extrusion.

Lately, the extrusion technology has been successfully used for the production of complex shapes such as honeycomb panels, window and door frames, and wave-shaped roof tiles without the use of molds resulting into economical production of these products. Even high-performance building materials such as bricks, tiles, and pipes have been manufactured using extrusion process [16]. Since extrusion, products are formed under high compression and shear, they show improved performance under various loading conditions. Furthermore, since the extruded material is in semi-solid state, hence the overall curing period of the cement-based products is considerably reduced, which means the products are ready to be used after comparatively shorter duration [26]. Besides being a continuous process for manufacture of variety of sections, extrusion makes use of simple machines, low water to cement ratio and produces less liquid and solid wastes. The low water to cement ratio of the cementitious mixture results into lower porosity and improved fiber-matrix interface of the extruded composites which significantly enhance their mechanical characteristics [27]. The use of extrusion process fulfills the requirements of sustainable production practices as various agricultural and industrial wastes such as sugarcane bagasse ash and blast furnace slag have been successfully incorporated as mineral additives in the extrusion process of cement-based composites [28,29].

This research evaluated the effect of various additives that include polymers, minerals, and chemicals towards enhancement of the physical, mechanical and durability attributes of the extruded cellulose fiber cement products reinforced with softwood fibers, and to facilitate the extrusion process and expand its range for manufacture of diverse profiles exposed to severe weathering conditions.



Fig. 1. Picture of the auger extruder (with barrel removed) [19].

2. Materials and methods

The basic mix ingredients used for extrusion of wood fiber cement products in this experimental work included; ordinary Portland cement; silica fume, hydroxyethyl cellulose, methyl cellulose (cellulose ether), high-range water reducer, wood fiber and water. Silica fume having fineness ($\% + 45 \mu\text{m}$) of 0.76%, loss on ignition of 3.16%, and bulk density of 0.286 g/cm^3 was used as mineral additive in all mixtures at a silica fume to cementitious material (cement plus silica fume) ratio of 0.4. The high-range water-reducing admixture was ASTM C 494 Type F. Methyl cellulose (cellulose ether) was used in the extrusion mixes to facilitate the dispersion of fibers in the mixes, retainment of water in the mix and reduction of friction during extrusion. The hydroxyethyl cellulose (250,000 molecular weight) was used to enhance the viscosity, extrudability and segregation resistance of the fiber cement mix. The wood fibers used in this investigation were softwood (southern pine) kraft pulp with an average length of 3.3 mm and an average diameter of $37 \mu\text{m}$. In all mixtures, the fiber to cementitious binder (cement plus silica fume) weight ratio was fixed at 0.10. The fibers were not fibrillated before use in the extrusion mix. Following optional ingredients were added to some fiber cement mixtures (with the key reason for their trial noted within parenthesis):

Sodium stearate (lubricating effect for ease of extrusion); sodium silicate (improved fiber-to-matrix bonding); Polyethylene glycol (PEG) with 600 molecular weight, styrene butadiene latex and wax emulsion (all for reduced water absorption and moisture-sensitivity of end product); and calcium chloride and sodium bicarbonate (both for acceleration of strength gain to facilitate handling of fresh product after extrusion). A minimum of three replicated specimens were tested in each unaged and aged condition for all mix designs considered.

Preparation of the mix involved separate mixing of cementitious materials with 25% of methyl cellulose in a mortar mixer, and high-speed mixing of water first with 75% of methyl cellulose and all other additives, and then with fiber. The wet fiber mix was then added to the dry mix in mortar mixer, and mixed until a homogenous blend was achieved.

Fig. 1 shows the laboratory-scale de-airing ceramic extruder used in this experimental program. The de-airing auger has an outside diameter of 76 mm; the exterior diameter of the extrusion auger ranges from a maximum of 102 mm at the beginning to a minimum of 76 mm at the die. The lengths of the de-airing and extrusion augers are 171 mm and 305 mm, respectively. The auger extruder speed was kept at 15 rpm. Fig. 2 shows the schematics of the die used with the extruder for production of flat specimens with 60 mm width and 8 mm thickness. The width of the die is such that it is comparable with the barrel diameter of extruder. Hence, its main function was to reduce the thickness of the fresh product from about 60 mm (in barrel) to 8 mm. The die has a slope of 24 degrees, with a 100 mm long flat finishing part.

Different matrix mix proportions considered in this investigation are presented in Table 1. Extruded fiber cement products were maintained at 22°C and 100% relative humidity for 8 h, and then subjected to steam curing at 60°C for 16 h. Products were then stored at 22°C and 50% relative humidity for 14 days before they were tested. The test procedures were conducted following the guidelines of ASTM D 1037 and ASTM C 1185. Extruded fiber cement products in unaged state were subjected to the following tests (three replicated specimens were subjected to each test): flexure by center-point loading on a span of 230 mm (with specimen width of 60 mm); flexure in saturated condition (after 48 h of immersion in water at 22°C); water absorption capacity; and bulk specific gravity. Three different accelerated aging conditions that is wet-dry, freeze-thaw, and hot water immersion were used in the experimental program. Table 2 presents the details of these aging conditions along with references of the relevant specifications. After accelerated aging, the specimens were stored at 50% relative humidity and 22°C before they were tested in flexure. Three replicated samples were subjected to accelerated aging and flexure testing. Lastly, the ease of extrusion of various matrix formulations was judged based on physical observation on a scale of 1 (poor extrusion) and 5 (excellent extrusion). Based on this observation, an ease of extrusion index was defined for all of the mix formulations.

3. Results and discussions

Figs. 3 and 4 summarize the bulk specific gravity and water absorption capacity test results for extruded fiber cement sheets with different matrix formulations, respectively. Compared to the basic matrix formulation (specific gravity of 0.9), modified ones tend to provide lower bulk specific gravity with the exception of Sodium Stearate + Sodium Bicarbonate based matrix, which results into

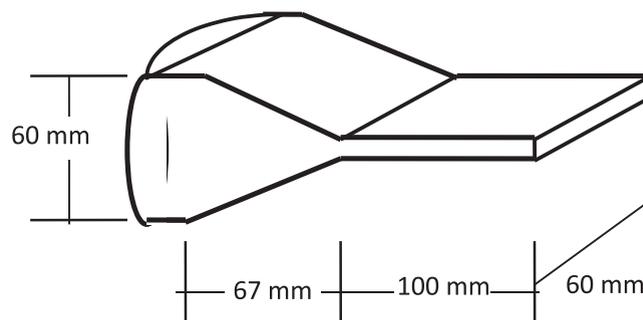


Fig. 2. Die geometry [19].

Table 1

Alternative matrix proportions in fiber cement mixtures suiting extrusion (weight ratios to cement plus silica fume unless specified otherwise).

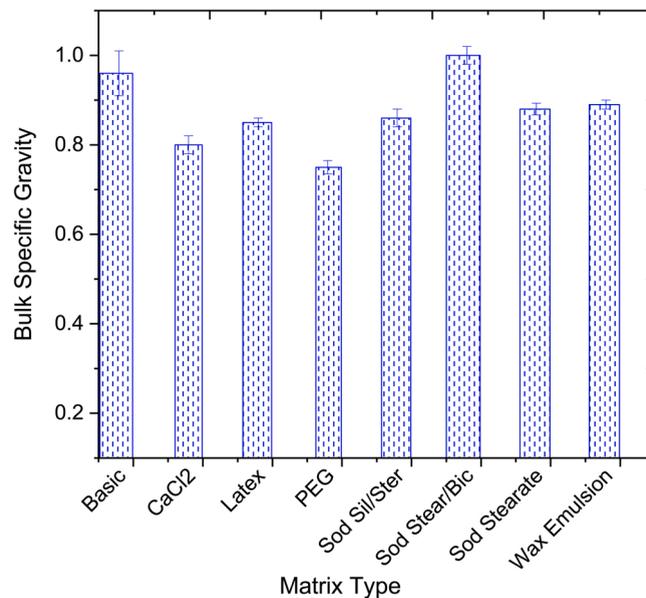
Mix Additive	PC	SF	MC	EC	SP ^a	Water	fiber/cement.	Additive 1	Additive 2
Basic	0.6	0.4	0.02	0.004	0.83	0.85	0.1	–	–
Sodium Stearate	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.04	–
Sodium Silicate + Stearate Sodium	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.02	0.02
PEG	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.04	–
Latex	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.02	–
Wax Emulsion	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.05	–
Calcium Chloride	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.04	–
Sodium Stearate + Sodium Bicarbonate	0.6	0.4	0.02	0.004	0.83	0.85	0.1	0.005	0.004

PC = Portland cement; SF = Silica fume; MC = Methyl cellulose; EC = Ethyl cellulose; SP = Superplasticizer.

^a Liters per 100 kg of cement plus silica fume.**Table 2**

Details of aging conditions.

Aging condition	Description	Specification
Wet-dry	30-cycles of wetting and drying	ASTM C 1185
Freeze-thaw	300 cycles of freeze-thaw	ASTM C 666
Hot water immersion	56 days of immersion in hot (60 °C) water	–

**Fig. 3.** Bulk specific gravity test results (means and standard errors).

slight increase in the specific gravity of the matrix formulation. This effect may be attributed to the ability of the sodium bicarbonate to form ettringite which results in the densification of the cement matrix, consequently causing increase in the specific gravity of the relevant matrix formulation [30]. The lower bulk specific gravities achieved by the modified matrix formulations is quite satisfactory when compared with fiber cement products processed by Hatschek (slurry-dewatering) and other techniques. On the other hand, all of the modified matrix formulations showed higher water absorption compared to the basic matrix formulation which had moisture absorption capacity of 30%. It is observed that even the polymer-based additives did not reduce moisture absorption capacity of extruded cellulose fiber cement products, possibly due to their lower dosage. These results are similar to the ones reported earlier [2].

Fig. 5 presents the flexural load-deflection behavior of various matrix formulations. Two quantitative features of these curves were used to compare the effects of matrix type on flexural performance: (1) flexural strength (peak flexural stress); and (2) flexural toughness defined as the total area underneath flexural load-deflection curve. With the exception of PEG-based matrix formulation, other modified matrix formulations showed comparable performance to that of basic matrix formulation. The lower flexural strength and stiffness of PEG-based formulation may attributed to its higher dosage in the mixture which has been reported to negatively impact the fresh and hardened properties of the cementitious mixtures [31,32]. Fig. 6a and b compare the flexural strength and toughness, respectively, of extruded wood fiber cement products with different matrix formulations (conditioned at 50% relative humidity) in

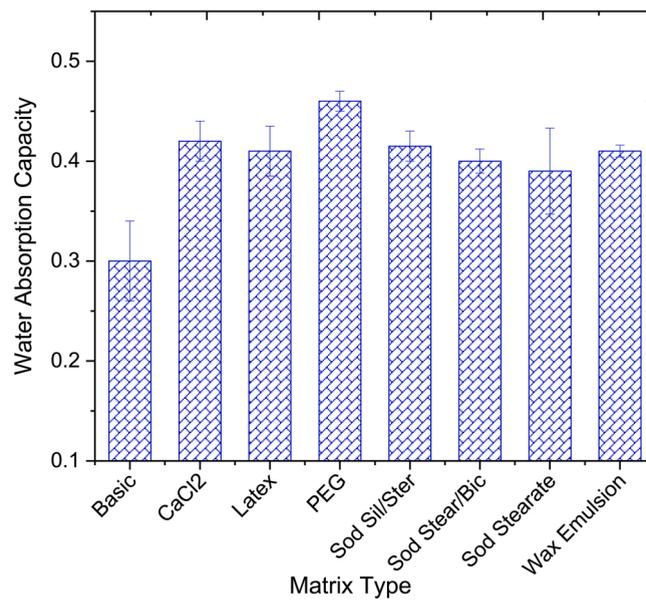


Fig. 4. Water absorption capacity test results (means and standard errors).

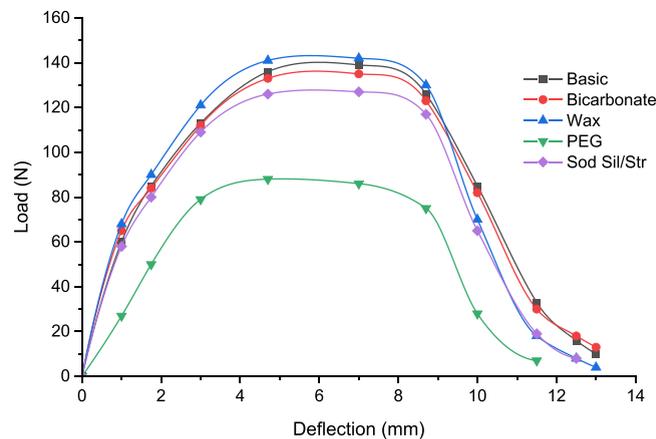


Fig. 5. Load-deflection curves of various matrix formulations.

unaged condition and after different accelerated aging effects. Most of the additives (except sodium bicarbonate) considered in this investigation (in addition to methyl cellulose, ethyl cellulose and superplasticizer which are part of the basic mix as well) cause a drop in flexural strength of unaged extruded wood fiber cement products. The drop in flexural strength is thought to have been caused by the increases in the leanness of the mixtures caused by the additives. Flexural toughness in the unaged condition, however, is less influenced by the use of various additives. The sodium bicarbonate and wax emulsion-based formulations show better stability under different accelerated aging effects as compared to basic formulation matrix. The early strength gain and reduction in the expansion of cementitious matrix under aging condition, brought about by the presence of sodium bicarbonate seems to enhance the flexural strength of the relevant matrix formulation. It is noted that flexural strength and flexural toughness of the mix formulations are also reduced under the wet-dry aging cycles. Although due to the rehydration of cement during such cycles, there is likelihood of increase in these attributes. It seems that an inhomogeneous moisture distribution caused by the wet-dry cycles promotes the evaporation of moisture from the surface of the cementitious formulations which results into salt crystallization and cracking of the cementitious matrices, ultimately leading to loss of strength. Furthermore, it has been reported that the wet-dry cycles cause fiber-cement debonding that leads to strength loss [33]. Thus, the damaging effect of wet-dry cycles overcomes their strength enhancing effect resulting into loss of the strength and toughness of the matrix formulations [34,35]. These results confirm the findings reported by earlier researchers [1,19]. Fig. 7 summarizes the effect of saturation on flexural performance of extruded fiber cement products with different matrix formulations. Saturation generally lowers the flexural strength of extruded cellulose fiber cement products (Fig. 7a); wax emulsion seems to produce more stability under moisture effects. Moisture absorption results into swelling of the fibers which in turn negatively

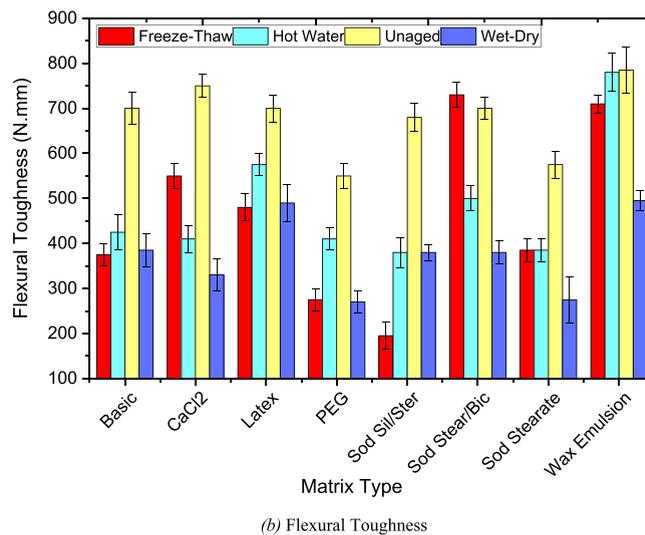
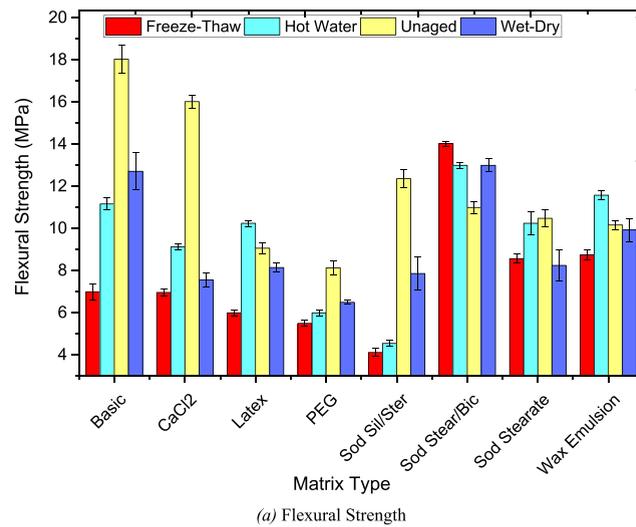
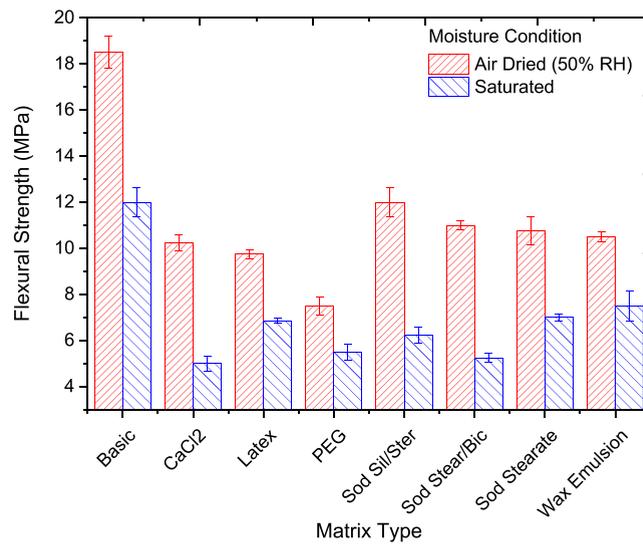


Fig. 6. Effects of aging condition on flexural strength and toughness of extruded fiber cement sheets with different matrix formulations (means and standard errors). (a) Flexural Strength, (b) Flexural Toughness.

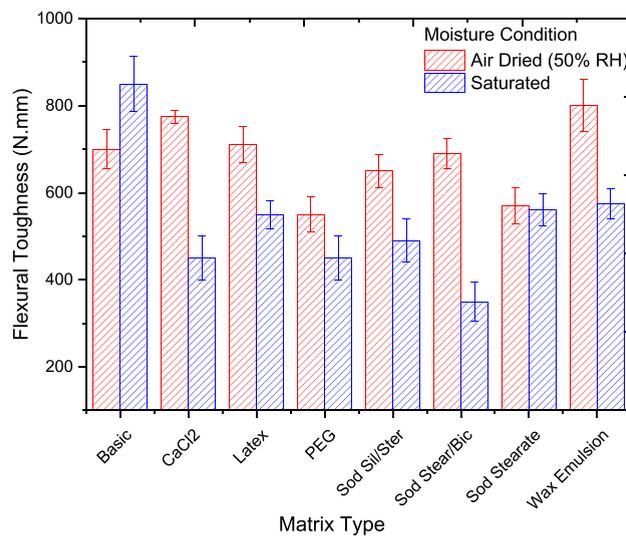
affects the fiber-matrix interaction and causes strength loss. Strength loss in natural fiber reinforced composites due to the moisture uptake has been reported by other researchers as well [36]. Any adverse effects of moisture on flexural toughness are less pronounced than those on flexural strength (comparison of Fig. 7a and b). The basic mix actually shows some gain in flexural toughness upon saturation. Moisture tends to reduce (in a reversible way) the hydrogen bonding between cellulose fibers and cement-based matrix which increases the tendency towards fiber pull-out (in lieu of fiber rupture) and consequently reduces the flexural strength. Frictional pull-out of wet (swollen) fibers, on the other hand, provides for some energy absorption which reduces the severity of moisture effect on flexural toughness. Hence, saturated cellulose fiber cement products exhibit a higher ductility (deformation capacity), as shown in Fig. 8. Table 3 shows the ‘ease of extrusion’ index, which was developed based on the physical observations during the extrusion process of various matrix formulations. Sodium stearate-based matrix formulation showed the best extrudability (index value of 5) followed by the sodium stearate plus sodium silicate and latex based matrix formulation with index value of 4. These observations show that the stabilizing and viscosity enhancing characteristics of the sodium stearate (fatty acid) significantly help in the extrusion process.

4. Conclusions

Based on the experimental observations, following conclusions are drawn:



(a) Flexural Strength



(b) Flexural Toughness

Fig. 7. Effects of saturation on flexural characteristics of extruded fiber cement sheets with different matrix formulations (means and standard errors). (a) Flexural Strength, (b) Flexural Toughness.

1. The basic matrix composition yielded a bulk specific gravity of about 0.9 and a water absorption capacity of 30%. Various additives generally lowered the bulk specific gravity but raised the water absorption capacity of extruded fiber cement products.
2. Presence of sodium bicarbonate showed positive effects on flexural strength of the relevant matrix formulation in the aged conditions, while the presence of latex, bicarbonate, and wax emulsion positively impacted the flexural toughness characteristics of these matrix formulations
3. Most of the additives caused drop in flexural strength of unaged extruded wood fiber cement products. The drop in flexural strength is thought to have been caused by the increases in the leanness of the mixtures caused by these additives. Flexural toughness was less influenced by the addition of additives.
4. Moisture produces reversible damage to the hydrogen bonds between cellulose fibers and cement-based matrices, thus lowering the flexural strength of the system.
5. Frictional pull-out of saturated (swollen) fibers, increased the ductility (deformation) capacity of extruded cellulose fiber cement products upon saturation.

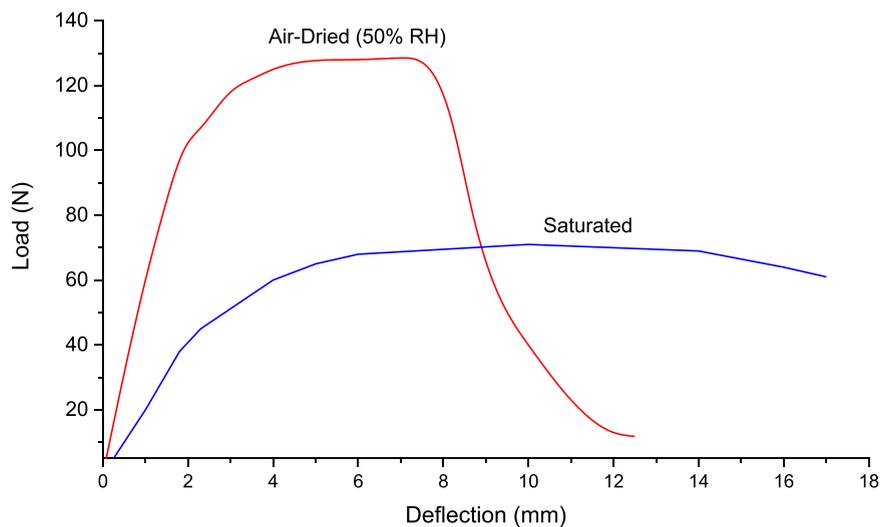


Fig. 8. Flexural load-deflection behavior with basic matrix formulation.

Table 3
Ease of extrusion index.

Matrix Formulation	Index Value
Basic	1
Sodium Stearate	5
Sodium Stearate + Sodium Silicate	4
PEG	3
Latex	4
Wax Emulsion	3
Calcium Chloride	2
Sodium Stearate + Sodium Bicarbonate	3

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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