Durability Evaluation of Cement Board produced from Untreated and Pre-treated Empty Fruit Bunch Fibre through Accelerating Ageing

P Peter¹, N M Z Nik Soh² Z A Akasah³, and M A Mannan⁴

¹,²,³ Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia.
⁴ Department of Civil Engineering, Faculty of Engineering, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia.
Corresponding author: nikzaini@uthm.edu.my

Abstract. Durability issue known that natural fibre tends to degrades in cementitious matrix particularly when in hot and humid exposure for a period of time. This experimental approach evaluates the potential of EFB cement board’s durability in accelerated ageing condition. Initially, the EFB fibres are pre-treated for 24 hours by being soaked in 1% NaOH solution and oven-dried at 103±2°C. Cement boards of 1300 kg/m³ density are fabricated in the laboratory, with 2.5:1:2 ratio of ordinary Portland cement, EFB fibre and water with 3% CaCl₂. Accelerated ageing assessment of hot water immersion for 10 days, 30 days and 60 days also wet dry cycle for 5 cycle, 15 cycle and 30 cycle at 70°C temperature are then conducted. The tested samples reveal finding ranges in modulus of rupture (MOR) of 3.98 – 9.11 MPa; modulus of elasticity (MOE) of 1056 – 4699 MPa; internal bonding (IB) of 0.28 – 0.53 MPa and thickness swelling (TS) of 1.66 – 9.25%. In addition, the field emission scanning electron microscope (FESEM) showed degradation mechanism of EFB cement board of before and after the accelerated ageing assessment. Conclusively, the findings showed that accelerated ageing approach for durability assessment of cement boards could be done in quantifying the durability of samples in shorter time.

1. Introduction
It is generally known that most countries cultivated oil palm as a major plantation and in Malaysia, oil palms are the country’s largest and most valuable crops thus further developments for a long-term strategy are needed for its potential as a material in structural and non-structural applications [1–5]. The natural fibre composite is known as more cost-effective, material with low energy consumption, environmentally friendly and sustainable [6, 7]. On top of that, it is important to take into account the adaptation of biodegradable composites, not simply for costs and availability but also for the consideration of occupants’ health factor in the long-term service of the building [8,9,10]. Substantially, various research and development activities globally as well as in Malaysia, are looking towards discovering an alternative source and an innovatively reliable raw material from natural fibres and the oil palm empty fruit bunch fibre (EFB) is a fascinating discovery in Malaysia to specifically explore its durability potential as a composite material [11]. However when exposed to various ageing condition, the fibre itself experiences severe degradation in the cement matrix thus causing premature deterioration to the cement composite material [12, 13]. Amongst such climate factors as temperature, humidity, solar radiation, precipitation as well as pollutants throughout the year; both high temperature
and humidity are the factors that most greatly affects the durability performance of building materials especially for a prolonged outdoor application [14-15].

2. Relevant previous studies

According to several studies on EFB fibres, chemical composition contents of lignin, hemicellulose and cellulose are about 13-43.51%, 17-47.7% and 22.90-57.8% respectively [16-18]. Due to the cell structure, natural fibre has limitation of its utilization as cement-based materials because of weathering condition [7,13]. Therefore, the fibres are necessary to be pre-treated and in alkaline pre-treatment with sodium hydroxide (NaOH) solution, studies reported concentration ranges from 0.2–6% solution has the ability to remove impurities and most residual oil on the EFB fibre surface [2,5,9,17,20–24] regardless of whether it is soaked for a period of 30 minutes or up to 24 hours. Whereas in the thermal treatment of fibre by oven-drying approximately at about 100°C–150°C, the composite results in 60% less thermal conductivity and also yield acceptable properties for a residential building material [22]. It is proved by the study of sisal fibre composite of Wahab [5] and Wei [9] that the thermal treatment improved cellulose crystallinity that provides high initial strength as well as maintained the reinforcement of fibre cement matrix in long period of time.

The causes of fibre degradation resulted into the reduction of fibre strength and its flexibility [25, 26]. As reported by Wei, natural fibre in pore solution of cement matrix experienced a linear decrease when exposed after 10 days in accelerated ageing and deteriorated completely in 2 years. Thus, further investigation on natural fibre in exposure of aggressive environment and various humidity for a long-term stability outdoor application is crucial [8-9]. In studies of durability by natural weathering condition, it usually takes period of approximately 3 months up to 12 months and reported that at 10 months exposure, the mechanical properties of hybrid reinforced composite showed better values compared to conventional concrete [25]. Likewise at 6 months of exposure, findings showed poor durability in toughness due to fibre-matrix adhesion [14]. Other than that, a complete reduction value of MOE and MOR also found [28]. In contrast to accelerated ageing condition in a laboratory, numerous totals of cycles and days at various temperatures are studied. According to Wei [9], 70°C or higher are the most effective temperature of accelerating condition to evaluate durability in hot soak immersion for days and in wet-dry cycle. At similar temperature, David [12] reported that ageing in lime saturated hot water is effective to evaluate the degradation of a specimen and the wet-dry ageing effective in evaluating the stability of fibre in cement-matrix. In order to evaluate the durability of a composite in shorter time, it is reported that the toughness of a composite is reduced with the increasing number of soaking and drying cycles [30] where the average values of MOE, MOR and IB are lower than composite without accelerated ageing condition [31].

Furthermore, most manufacturing of natural fibre composite material used calcium chloride (CaCl₂) as chemical accelerator to improve composites’ mechanical and physical properties. In study by Onuorah et. al. [18], usage amount of 3% CaCl₂ is reported to have the highest obtained mechanical properties compared to 1%-2% CaCl₂. In conjunction with Kishar et. al. [19], it is reported that cement accelerator of CaCl₂ showed higher compressive strength in a cement paste and has lower porosity compared to samples without addition of CaCl₂. Although there are research studies done in regards to fibre pre-treatment and manufacturing improvement methods, rarely reported on the durability aspects particularly of EFB fibre as a composite material. Thus, significant assessment on durability potential of EFB cement board is obviously necessary depending on the type of materials used and methods involved.

3. Experimental materials and method

The EFB fibres were obtained from the Global Seed Sdn. Bhd. factory in Kawasan Perindustrian Simpang Renggam, Johor. It was compacted in blocks of bunches that comprised of long EFB fibres and weighed approximately 2 tonnes. The compacted blocks were loosened to be extruded by the crusher machine for the chipping process at Timber Fabrication Laboratory, Universiti Tun Hussein Onn Malaysia to obtain the short EFB fibres.

The sieving processes was done at Malaysia Palm Oil Berhad, Bangi where it separates the dust from the required fibre sizes for cement board fabrication in which the sieve machine has nominal
openings of 4.76 mm (4 mesh), 2.83 mm (7 mesh), 1.41 mm (14 mesh) and 0.177 mm (80 mesh). The lengths distribution of the EFB short fibres are approximately 80 mesh, 14 mesh and 7 mesh of EFB fibres of about 8–10 mm, 10–13 mm and 10–18 mm respectively. As mentioned in previous studies [20,21,24], the most effective amount for EFB fibre is 2% NaOH concentration yet it is not discovered as the amount for the durability of EFB cement board properties specifically. Thus, the fibre is pre-treated by soaking for 24 hours in the concentration of 1% NaOH solution. For heat treatment, the fibres are oven-dried at a temperature of 103±2°C for 24 hours, along with being soaked in water at ambient temperature for 30 minutes. After completion of the pre-treatments period, the EFB fibres are washed in running tap water and then dried under the hot sunlight [5, 17]. Substantially, the size of 14 mesh is used for fabrication in this study where it achieves the provisions of Malaysian Standards 1984 similar to the cement board reported by Onuorah et. al [18]. However, only the properties of cement board were reported. Thus, this research attempts to continue by assessing the durability of the material as subjected to an accelerated ageing condition as recommended by Wei [9].

3.1 EFB cement board fabrication
The 350 mm × 350 mm × 12 mm cement board samples of targeted density 1300 kg/m³ are prepared for a total of 36 samples. The mixture’s material of ordinary Portland Cement Type 1, calcium chloride (CaCl₂), water and EFB fibre are used to fabricate the cement board. The mixing ratio of cement:EFB fibre:water is 2.5:1:2, CaCl₂ is 3% based on cement weight and is used in the form of powder diluted in water while the water-cement ratio is 40% [18,27]. The mixtures are mixed in the mixer machine for 5 minutes at 60–80 rpm. It is then pre-formed by hand lay-up method unto a wooden mould through a wire mesh. Initially, a polythene sheet place unto the plywood. The complete mixture is then pre-pressed with another polythene sheet upon it with the metal plate mould placed on the upper part before being transferred to a 50 ton/m² cold compressor machine. For approximately 5-7 minutes, the cement board mixture is compressed at 180 mm/min pressure rate. Upon being removed from the machine, the board are let to cure for 24 hours at laboratory ambient temperature then be de-moulded. The EFB cement board is cured for 28 days in the laboratory before being subjected to an accelerated ageing assessment as referenced by Wei [9].

3.2 Experimental tests of accelerated ageing
The totals of samples are cut each to be tested for MOE, MOR, IB and TS properties and to be subjected to an ageing process as recommended by previous research [9]. The durability test is conducted at 70°C for accelerated ageing conditions of 10 days, 30 days and 60 days and 5 cycles, 15 cycles and 30 cycles for hot water immersion and wet-dry cycle, respectively.

Figure 1. Fabrication processes of EFB cement board.
Table 1. Durability assessment of EFB cement board.

| Sample     | Properties | Control | 5 C* | 15 C | 30 C | 10 D* | 30 D | 60 D |
|------------|------------|---------|------|------|------|-------|------|------|
|            |            | U*      | T*   | T*   | T*   | U*    | T*   | T*   |
|            |            | T       | N    | H    | T*   | U*    | T*   | T*   |
| Mechanical | *MOR       | *5      | *5   | *5   | *5   | *5    | *5   | *5   |
|            | *MOE       | *5      | *5   | *5   | *5   | *5    | *5   | *5   |
|            | *IB        | *5      | *5   | *5   | *5   | *5    | *5   | *5   |
| Physical   | *TS        | *5      | *5   | *5   | *5   | *5    | *5   | *5   |

Notes: C = cycles, D = days, UT = untreated EFB, TN = 1% NaOH pre-treated EFB, TH = heat pre-treated EFB, MOR = modulus of rupture, MOE = modulus of elasticity, IB = internal bonding, TS = thickness swelling, * = number of samples, Total samples = 105

For the accelerated ageing condition of hot water immersion, the samples are immersed completely in a water bath machine. Whereas for the wet-dry cycle, the samples are immersed in water for 15 hours and then oven-dried for 25 hours for each cycle. The Universal Testing Machine (UTM) INSTRON 3369 shown in figure 2 is used for the experimental test of MOE, MOR and IB properties for before and after ageing assessment.

4. Result and analysis
The cement board produced by heat treated EFB fibre showed higher MOE value followed by cement board produced by NaOH treated EFB fibre where in figure 3, showed values range in 2879 – 4699 MPa and 1056 – 3459 MPa respectively. In ageing through warm water immersion, declined value reported approximately 49.58% and 37.15% for NaOH and heat treated EFB fibre as cement board in 10 days to 30 days respectively. Whereas in 5 cycle to 15 cycle of wet-dry cycle, declined value reported about 32.76% and 20.71% for NaOH and heat treated EFB fibre as cement board respectively.
In figure 3b, the MOR value obtained shows a gradual declined for ageing of wet-dry cycle and warm water immersion. Value ranges are 3.98 – 4.94 MPa and 4.77 – 9.11 MPa for the NaOH and heat treated EFB fibre as cement board respectively. From the obtained result for MOE and MOR for accelerated ageing condition, warm water immersion as well as wet-dry cycles is observed to be the aggressive ageing for the composite as its strength declined, similarly stated by Wei [9].

Similarly observed for the IB in Figure 4, where its value slightly decreased for both accelerated ageing to the ranges of 0.28 – 0.39 MPa and 0.32 – 0.53 MPa for NaOH and heat treated EFB fibre as cement board respectively. In spite of the utmost properties obtained for MOE, MOR and IB, the untreated fibre and followed by NaOH treated EFB fibre as cement board showed slightly higher TS compared to the heat treated EFB cement board in both accelerated ageings where observed values of 7.27 – 14.55%, 6.94 – 9.25% and 1.66 – 5.81% respectively, shown in figure 4b.
4.1. **Field Emission Scanning Electron Microscope (FESEM) analysis**

The microanalysis of EFB fibre and cement board produced from EFB fibre performed on JEOL JSM-7600F field emission scanning electron microscope. Initially, a fine layers of gold sputter-coated the EFB cement board in order for it to be conductive under the FESEM machine. The microanalysis is crucial to evaluate the effect of accelerated ageing condition on the degradation mechanism of EFB fibre in a cement matrix.

Figure 5a shows the impurities attached on the surface of untreated EFB fibre where it affected the bonding properties between EFB fibre and cement matrix. After the pre-treatment with NaOH in figure 5b and heat thermal in figure 5c, the impurities are removed. Compared to NaOH pre-treatment of the fibre, the residual and impurities that attached on the fibre are seen to be mostly removed when pre-treated with heat thermal. The cement board produced from NaOH and heat pre-treated EFB fibre shown in figure 5e and 5f respectively before subjected to the accelerated ageing condition. It is seen that the interfacial bonding between fibre and cement matrix are better compared to untreated EFB fibre as cement board.

![Figure 5](image)

**Figure 5.** FESEM images of EFB single fibre and EFB cement board without accelerated ageing

![Figure 6](image)

**Figure 6.** FESEM images of 15 wet-dry cycles and 30 days hot water immersion
Whereas in figure 6, shows the FESEM images of the cement board produced by untreated fibre as well as fibre pre-treated with NaOH and heat thermal after the accelerated ageing condition. As can be observed from the figure, interfacial failure between fibre-cement-fibre can be clearly seen after 15 wet-dry cycles and 30 days of hot water immersion. Wei [9] has reported that, the durability of fibre cement based composite is able to evaluate in a reasonably shorter time by the accelerated ageing assessment. Both wet-dry cycles and hot water immersion shows aggressive effect on the reduction strength of the EFB cement board.

5. Conclusion
Based on the experimental results, both hot water and wet-dry ageing greatly affect the degradation mechanism of EFB fibre in cement matrix with increases number of cycles and durations. In terms of MOE, MOR and IB, it was found that significantly value decreases for cement board produced from untreated and pre-treated EFB fibre through both ageing especially wet-dry cycle. Whereas for TS properties, the untreated EFB-cement board has highest percentage of swelling compared to NaOH and Heat treated EFB fibre. Furthermore, the microstructure analysis showed that after 15 cycles and 30 days of ageing, the fibre encounter severe degradation in the cement matrix. This also shows an acceptable approach to determine durability of the EFB cement board within a reasonable short time. In summary, the fibre pre-treatment is the most crucial method in the fabrication of the cement board. Upon durability assessment, the cement board achieves the ultimate properties required for MOE, MOR and IB. However, the TS value is slightly higher amongst the three EFB cement boards due to moisture absorption. Therefore, various ageing condition for durability in accelerated ageing should be conducted on the EFB cement board to explore more of its durability potential as a construction material.

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