Preliminary study of natural fibers with various treatment processes on properties of fiber-reinforced cement

Piroon Siriput¹, Teewara Suwan²,³,*, Hemwadee Thongchua¹, Gunamon Thongchua¹, Yanisa Thammapradit¹, and Sarach Jitsakulchok¹

¹Department of Civil Engineering, Faculty of Engineering, Chiang Mai University, 50200, Thailand
²Center of Excellence in Natural Disaster Management, Chiang Mai University, 50200, Thailand
³Chiang Mai University Advanced Railway Civil and Foundation Engineering Center (CMU-RailCFC), Chiang Mai University, 50200, Thailand

Abstract. Natural fiber has remarkable engineering properties, especially tensile strength. It can be used as a fiber reinforcement in cement composites with some environmentally friendly with economic advantages. Recycling of natural wastes could reduce PM 2.5 pollution during the burning season. However, those natural fibers require any pre-treatment processes to achieve more bonding ability with the cement matrix. The main aim of this study is to investigate the effects of various pre-treatment processes on the properties of fiber-reinforced cement. This test used rice straw and hemp fibers to represent the natural fibers. The results show that even the presence of natural fiber causes density and mechanical strength reductions, the elasticity through the deflection test is evidently increased up to 40.5%. All tested pre-treatment processes, i.e., using NaOH, HCl, and boiled in water, provided quite similar results in density and mechanical properties. The usage of NaOH seems to achieve good properties with reasonable price and simple preparation method. Moreover, applying less NaOH concentration or alternative non-chemical method (i.e., boiled water) could provide acceptable results with much less environmental impacts and costs.

1 Introduction

Cement or Ordinary Portland cement (OPC) is a primary concrete component. Concrete generally has excellent compressive strength but is brittle with deficient tensile ability. Therefore, steel rebars or steel fibers are applied in concrete structures to support this weakness, known as ‘Reinforced concrete (RC)’structures. Nowadays, with the Bio-Circular-Green (BCG) economy model, many research works have been studied on recycling natural fibers as a fiber reinforcement in cement and concrete [1]. The literature reviews found that natural fibers have remarkable engineering properties, especially tensile strength. Moreover, it also provides some environmentally friendly and economic advantages. For example, the natural fibers used as construction materials and composites are sisal, flax, coir, bamboo, hemp, rice straw, and many others [2].

* Corresponding author: teewara.s@cmu.ac.th

© The Authors, published by EDP Sciences. This is an open access article distributed under the terms of the Creative Commons Attribution License 4.0 (https://creativecommons.org/licenses/by/4.0/).
At present, the demand for using natural materials is being increased as they can be cultivated, renewable, and have such sustainable development. This preliminary study used rice straw and hemp as natural fibers for fiber-reinforced cement production. Rice straw was selected as a high-potential natural fiber waste due to Thailand's massive rice production. In the Northern region, around 30% of the rice of the whole country (approximately 8,000 million tons) is harvested annually. In the same amount of rice production, rice straw becomes organic waste left in the farm fields. Utilizing those massive straw wastes as natural fibers for fiber-reinforced cement positively reduces air pollution from the PM 2.5 during the burning season [3,4]. Apart from that, the latest government policies allow cultivating hemp as one of the economic crops. Moreover, the Highland Research and Development Institute (HRDI), located in Chiang Mai, Thailand, also strongly supports the Thai industrial hemp species for the test. Thus, hemp fiber participated in this preliminary study as an alternative natural fiber reinforcement [5].

The function of fibers in the cement matrix needs to be high in bonding ability between the fiber and cement binder. However, many research studies revealed that natural fibers require some pre-treatment processes before use. The reasons are to remove some waxy substances, e.g., oil, waxes etc., and to increase the roughness and bonding of fiber’s surface to be suitable in practice [5,6]. Many pre-treatment processes exist, such as biological, physicochemical, and chemical treatment. For example, soaking in a mild acid, soaking in an alkaline solution, boiling in a hot water and many others [7,8]. In addition, it is reported that incorporating natural fiber with alkaline activated cement or geopolymer is also one of the alternative approaches to pre-treat the fiber [9,10]. Even though the chemical treatment using NaOH is commonly used, there are many other effective pre-treatment processes to be applied.

By this, the main objectives of this study are to investigate the effects of various pre-treatment processes on the properties of fiber-reinforced cement. Rice straw and hemp fibers were used as representative of natural fibers, while the top three widely-use techniques, i.e., NaOH, HCl and boiled in water, were carried out in this work. The outcomes may increase a value-added concept to achieve greener production for environmentally friendly alternative construction materials using renewable sources of natural materials.

2 Materials

General purpose TPI Ordinary Portland Cement (OPC) type I, ASTM C150 and Thai Industrial Standard (TIS) 15-1, was used with a specific gravity of 3.13. Local river sand was used as a fine aggregate with a unit weight of 1,509 kg/m³. The gradation complied with ASTM C778, while the fineness modulus of the sand was 2.90. Rice straw was received from local areas in Chiang Mai (Northern region), Thailand. It was kept air-dry in atmospheric conditions for a couple of months. Then, the straws were cut into a specific length of 2 cm. for the test. Its average diameter was approximately 3.39 mm., while the unit weight of the as-received straw was 0.311 g/cm³. Its moisture content and water absorption were 17.6% and 82.4%, respectively. Hemp fiber, the Thai industrial hemp specie, was received from the Highland Research and Development Institute (HRDI), Chiang Mai, Thailand. The plain fiber of hemp was manually prepared and cut into 1, 2 and 3 cm., respectively. Its average diameter was approximately 1 mm., while the unit weight was 1.37 g/cm³. According to the results of pilot testing of flexural and deflection ability, it is noted that the rice straw was carried out for the experiment on density, compressive strength, and flexural strength. While hemp fiber was carried out for the deflection test for more clarity in the analysis.
3 Mixture designations and analytical methods

Two cm long straw was rinsed with tap water to remove some dusts and then placed in the oven at 60°C for 24h for drying. After that, it was prepared for the various pre-treatment processes, which are (i) soaking in 1 molar of NaOH solution for 24h (NaOH 1M), (ii) soaking in 5 molars of NaOH solution for 24h (NaOH 5M), (iii) soaking in 3.6%w/w hydrochloric acid for 24h (HCl), and (iv) boiling in 100°C water for 30 minutes (Boiled). Next, the straw was rinsed with tap water and put in the 60°C-oven for another 24h. The sample preparation started with dry-mixing OPC and sand of 1:2.75 by weight. The water-to-cement (w/c) ratio was set to 0.35. After thoroughly mixing cement mortar, the saturated surface dry (SSD) straw was added into the mortar mixer with 2% w/w of the OPC. Figure 1a-1e show the images of all prepared rice straw.

![Figure 1a: As-received straw](image1.png)
![Figure 1b: Straw-1M NaOH](image2.png)
![Figure 1c: Straw-5M NaOH](image3.png)
![Figure 1d: Straw-3.6% HCl](image4.png)
![Figure 1e: Straw-boiled](image5.png)
![Figure 1f: Hemp fiber](image6.png)

Fig. 1. Straw with various pre-treatment processes and Hemp fiber.

As aforementioned, the previous pilot test results showed that hemp fiber was suitable for deflection testing due to an appropriate average diameter of 1 mm with high tensile strength (Figure 1f). The hemp fiber preparation processes were in the same order as the preparation of straw fiber. However, the various lengths of hemp fiber were set to 1, 2, and 3 cm, and the amount of added hemp fiber to the mixture was 1% w/w of the OPC. After well-mixing, the mixtures were placed into the $4\times4\times16$ cm³ prism mould. After 24h of casting, all specimens were demoulded, wrapped with plastic film, and kept at room temperature until reaching their testing age. The details of the mixtures and proportions are shown in Table 1.

### Table 1. Mixtures and proportions.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Pre-treatment</th>
<th>OPC:S</th>
<th>w/c ratio</th>
<th>L (cm)</th>
<th>% Fiber</th>
<th>Experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>-</td>
<td>1:2.75</td>
<td>0.35</td>
<td>-</td>
<td>-</td>
<td>All testing</td>
</tr>
<tr>
<td>Straw-based composites</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaOH 1M</td>
<td>NaOH 1 molar</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Density,</td>
</tr>
<tr>
<td>NaOH 5M</td>
<td>NaOH 5 molar</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Compressive</td>
</tr>
<tr>
<td>As received</td>
<td>-</td>
<td>1:2.75</td>
<td>0.35</td>
<td>2</td>
<td>2</td>
<td>and Flexural strengths</td>
</tr>
<tr>
<td>HCl</td>
<td>HCl 3.6%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The specimens, 4×4×16 cm³, were prepared following a BS-EN 196-1 standard. Dimensions and weights of all samples were recorded before carrying to mechanical testing. The density was also calculated and recorded before the test. Flexural and compressive strengths were performed by using a 250kN CONTROL UTM at the age of 28 days. The deflection and the applied load of hemp-based reinforcement cement were also recorded on the UTM at their 28 day-age.

### 4 Results and discussions

Figure 2a shows the dry density of the control sample (non-fiber reinforcement) and straw-based samples in various treatment processes. It can be seen that all samples with rice straw reinforcement had fewer dry densities than that of the control mix (2.177 g/cm³) due to the very low unit weight of the rice straw. Using NaOH 1M and boiled water provided similar densities of 2.054 g/cm³, followed by NaOH 5M and HCl, respectively. Those pre-treatment processes evidently revealed the ability of natural fiber’s surface modification. In addition, the as-received straw got the lowest density of just 2.019 g/cm³. It can be explained that the enamels and waxes on the straw’s surface obstructed water absorption during the mixing, resulting in less density.
Figure 2b shows the compressive strength of the control sample (non-fiber reinforcement) and straw-based samples in various treatment processes. A similar trend to the density was observed as the highest compressive strength was the control cement (44.9 MPa), followed by the straw-reinforced composites of 1M NaOH (35.2 MPa), 5M NaOH (34.3 MPa), Boiled (33.6 MPa), and HCl (32.6 MPa), respectively. With less density and lack of surface bonding, the as-received straw composite thus obtained the lowest compressive strength of 31.4 MPa.

Figure 2c shows the flexural strength of the control sample (non-fiber reinforcement) and straw-based samples in various treatment processes. The control mixture achieved the highest flexural strength at 8.65 MPa. In contrast, the flexural strengths were dropped in straw-based composites to around 6-7 MPa. Even the flexural strengths of straw-based composites were a little different, the straw fibers of 5M NaOH seemed to be more delicate and smoother as fine fiber than other processes (see Figure 1). Moreover, according to previous research, the thin fine fiber provided a better elastic function on the deflection than the thick one [5]. Therefore, the deflection of the natural fiber-based composite was extendedly carried out in the next section.

Fine hemp fiber with a 5M NaOH pre-treatment process was used for the deflection test with different lengths of 1, 2 and 3 cm. Figure 3 shows that the control OPC mix achieved the highest load resistance at 210 kg with only 0.37 mm deflection. The lowest load resistance was 1 cm long of hemp-based reinforcement at 153 kg with 0.35 mm deflection. In contrast, the 2- and 3-cm long hemp fibers provided superb deflection of 0.52 mm (166 kg) and 0.46 mm (192 kg), respectively. It can be explained that 2- to 3-cm long of 5M NaOH pre-treatment hemp fiber evidently improves the deflection and elasticity of the natural fiber composites. The 5M NaOH treatment hemp composite provided a maximum deflection resistance of approximately 40.5% greater than the control non-fiber cement sample.

![Figure 3](image-url)  
**Fig.3.** Load vs deflection of hemp fiber with 5M NaOH pre-treatment process

### 5 Conclusion

The main aim of this study is to investigate the effects of various pre-treatment processes on the properties of natural fiber-reinforced cement (rice straw and hemp). Pre-treatment processes with NaOH 1M, NaOH 5M, 3.6% HCl, and 30 min.-boiled in the water were
carried out to compare with the as-received straw- and the control mixtures. It was found that the densities of all-natural fiber composites were dropped when compared to the control (non-fiber) mixture, resulting in reduced mechanical strengths. The mix with no pre-treatment process (As-received) evidently showed the lowest density and compressive strength. It can be explained that the waxy enamel on the fiber’s surface of the as-received mixture obstructed the water absorption and provided less density and contact friction to the binder. However, the elasticity through the deflection test (represented by hemp fiber) is evidently increased when treated fibers are used. All tested pre-treatment processes, i.e., using NaOH, HCl, and boiled in water, provided quite similar results in density and mechanical properties. It was found that the use of NaOH seems to achieve good properties, as done in many previous researchers, with reasonable price and simple preparation method. The appropriate pre-treatment process using 5 molar NaOH with 2-cm long fiber provided the maximum deflection resistance of approximately 40.5% greater than the control non-fiber cement sample. However, the results of this work found that the usage of less NaOH concentration (i.e., from 5M to 1M) or alternative non-chemical method (i.e., boiled in hot water) could provide acceptable results with much less environmental impacts and costs. Eventually, the overall outcomes may support the construction industry in reducing the environmental impact and promote a greener and cleaner production of construction materials.

References

1. R. Sikkema, D. Styles, R. Jonsson, B. Tobin and K.A. Byrne, Sustain Cities Soc 90, 104370 (2023)
3. R. Singh and M. Patel, Biomass and Bioenergy, 106411 (2022)

Acknowledgments

Special thanks to the Faculty of Engineering, Chiang Mai University, Thailand for all support. Thanks to Chiang Mai University for the grant of this project. Also, my lovely and hardworking students who kindly helped to conduct the experimental work.