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To cite this article: H Abu Bakar *et al* 2024 *IOP Conf. Ser.: Earth Environ. Sci.* **1347** 012084

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The effect on tensile and surface morphological properties of oil palm empty fruit bunch fibre through hot water treatment

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Abstract. The utilisation of Oil Palm Empty Fruit Bunch (EFB) fibre, a significant crop in Malaysia, is a viable option for incorporation into cement-based products due to the substantial amount of waste it generates. Nevertheless, the presence of extractives has a significant impact on the performance of EFB fibre and leads to an inherent incompatibility between EFB fibre and cement. Hence, the purpose of this study was to investigate the impact of varied temperatures and soaking durations on the physical and mechanical properties of EFB fibre as a reinforcement material. This research aimed to contribute new insights into the performance of these qualities under varying conditions. The result from the tensile strength shows that the EFB fibre increased dramatically with temperature at all soaking times. Nevertheless, extended periods of soaking have been found to have a detrimental effect on the tensile strength of fibres due to the process of deterioration. This leads to a weakening or damage to the fibres. Thus, the surface morphology observation proved that as temperature increased, the number of silica bodies removed from the fibre surface increased (80°C, 90°C and 100°C) at all soaking hours. However, starting at 70°C (3 hour soaking time), the circular craters on the surface and the lignin layer start to damage. Therefore, these findings exhibit considerable potential for future investigation in substantiating the viability of employing EFB fibres as reinforcement agents in composite materials.

1. Introduction

Malaysia is a significant global producer and exporter of palm oil. Oil palm production in Malaysia has witnessed a consistent upward trend, rising from 4.1 million tonnes in 1985 to 6.1 million tonnes in 1990, as reported by the Malaysian Palm Oil Board (MPOB). In 2019, it produced about 19.9 million tonnes of palm oil and exported 93% (18.5 million tonnes) of it to the world's oils and fats market [1]. Thus, palm oil accounted for the largest proportion (32.2%) of worldwide oil and fat output, with its primary cultivation being dedicated to the production of cooking oil [1]. The total number of palm oil mills is about 457 with an entire processing capacity of 116.81 million tonnes of fresh fruit bunches (FFB) [2]. Concurrently, the increased production volume gave rise to a significant concern within the palm oil sector, namely the substantial quantity of biomass disposal [3]. The biomass produced at the plantation consists of the following components: oil palm trunk fibres (OPT), oil palm frond fibres (OPF), 23% empty fruit bunch fibres (EFB), 6% palm kernel shell (PKS), 7% palm kernel cake (PKC) and 15% oil palm mesocarp fibre (OPM) [3,4,5]. According to the study conducted by Faizi et al. [6], it was shown that palm oil accounted for just 10% of the total output of the oil palm tree, while the other 90% was composed of biomass waste. The management of this substantial volume of oil palm biomass



presents difficulties and has a detrimental impact on the environment [6,7]. In addition, it should be noted that palm oil fibre exhibits favourable physical and mechanical characteristics, as indicated by previous studies [8,9]. Nevertheless, in light of ongoing advancements in the field of science and technology, it is imperative for researchers to explore alternative options for sustainable building materials [10]. This necessitates the investigation of novel green composites that utilise natural plant fibres. Hence, natural fibres possess the potential to serve as alternative waste resources that have the capacity to substitute synthetic fibres in the realm of engineering fibre composites [11-13].

Furthermore, green technologies have enabled the conversion of oil palm biomass into various products, including biofuels, bio composites, and green chemicals. This accomplishment is based on Malaysia's government's policy of supporting green technology, which encourages industry participants and other stakeholders to take part in the conversion of biomass into value-added products. The Malaysian government implements new technologies and market accessibility to enhance oil palm biomass utilization, but implementation is still nascent due to the low conversion rate to value-added products [3]. However, based on previous research by Maynet et al. [14] and Owoyemi et al. [15], composite-based products have the potential to reduce EFB fibre from being a main waste. For instance, composite boards, such as cement boards, can be effectively utilised in both interior and exterior applications within the realm of building construction. While, according to Akasah et al. [2016], the substitution of wood fibre with EFB fibre in cement-bound fibreboards offers potential environmental and socio-economic advantages. Thus, compared to traditional concrete materials, composite-based products offer enhanced thermal insulation, workability, and acoustic qualities [17]. Therefore, the utilisation of EFB fibre would not only alleviate the strain on the plants in forests [13], but also mitigate the need for biomass disposal [3,6,7].

Compatibility is another major problem when producing fibre-cement-based products [13-20]. Several studies have addressed the enhancement of the compatibility between fibre and cement through the application of specific treatments, such as surface modification treatments, including chemical and physical treatments [21-23]. Furthermore, the process of surface modification treatment has the capability to alter the surfaces by effectively eliminating contaminants, hence enhancing surface roughness. This, in turn, improves the bonding capacity between the surface of the fibres and the matrix material [19]. Previous studies were mostly conducted on the surface modification of EFB fibre with chemical treatment, such as research by Nasidi et al. [24], Nik Soh et al. [9], Bakhtiar et al. [25], Akasah et al. [16], Akasah et al. [26], Omoniyi [27], Iskandar et al. [21], Bonnet-Masimbert et al. [28], Ramlee et al. [22], Faizi et al. [6] and Maynet et al. [29]. Based on a thorough analysis of the literature, it was observed that numerous researchers have utilised sodium hydroxide (NaOH) pre-treatments as a means to enhance the compatibility between fibre and cement. However, alternative chemicals such as acetic acid (CH_3COOH), sodium perchlorate (NaClO_4), and various others have also been employed for this purpose. But the knowledge of hot water treatment that affects the compatibility of cement composites is very limited. Several previous studies used hot water treatment for natural fibres such as oil palm broom (OPB) fibres [5], EFB fibre [27] and sugarcane bagasse fibre [30] before being used to produce cement-based composites. The application of a treatment temperature of 100°C to OPB fibre led to the occurrence of radial cracking. In order to prevent harm to the fibres, it is advisable to conduct hot-water treatment at a temperature below 100°C . In line with the study conducted by Omoniyi [27], an assessment was carried out on the physical and mechanical characteristics of cement-bonded composites manufactured using EFB fibres. The study examined different production variables, specifically the pre-treatment of fibres with water at different temperatures (cold, 60°C and 100°C) and five concentrations of a chemical additive (NaOH) (2%, 4%, 6%, 8%, and 10%). The findings indicate that the pre-treatment of fibres with hot water at 60°C and a NaOH concentration of 8% had a substantial impact on improving and altering the performance of the composites. While Cabral et al. [30] observed that composites containing treated sugarcane bagasse fibres subjected to a temperature of 100°C for a duration of 30 minutes exhibited reduced physical attributes compared to composites containing non-treated sugarcane bagasse fibres. The mechanical properties of the composites with pre-treated sugarcane bagasse fibres exhibited higher values compared to those without treatment, indicating the effectiveness of pre-treatment in enhancing the performance of sugarcane bagasse fibres in cement compositemanufacturing. Finally, there is a gap in studies on the treatment of EFB fibre whereas no research

previously focused on using hot water treatment with variation temperature and soaking hour. Therefore, this study aims to investigate the impact of different temperatures, starting with room temperature (RT) as the control, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C with different soaking hours 1, 2 and 3 to provide new knowledge on the relationships between the performance of physical and mechanical based on the control sample and treated EFB fibres as a CB reinforcement.

2. Materials and methods

The EFB fibre utilised in this study was procured from Pamol Kluang Palm Oil Mill, located in Kluang, Malaysia. All preparation was carried out at the Timber Fabrication Laboratory and Construction Engineering Laboratory, Faculty of Civil Engineering and Built Environment, Universiti Tun Hussein Onn Malaysia. To avoid the effect of weather exposure during the storage period, EFBs are gathered the same day as fruits are removed. Upon transporting the raw EFB from the mill, it was sun-dried for 2 to 3 days, depending on the weather conditions to reduce the excessive moisture. The dried EFB was then cut into shorter fibres using a hammer mill machine. The EFB fibre was initially soaked in the water bath at different temperatures and soaking times. For this study, the temperatures of the hot water used were 50°C, 60°C, 70°C, 80°C, 90°C and 100°C. The switch needs to be turned on and the temperature pre-set as desired. Finally, after the temperature reached the pre-set value, the fibres were loaded, and the soaking procedure was done up to the setting soaking hours required. After the fibres are rinsed, they must be dried under the sun for a few days and put in the oven for 24 hours at 100°C.

2.1. Tensile properties of EFB fibre

Tensile strength was used to determine the mechanical properties of EFB fibre. The testing was conducted at the Structure Engineering Laboratory, Faculty of Civil Engineering and Built Environment, Universiti Tun Hussein Onn Malaysia. The tensile strength test for EFB fibre was conducted based on ASTM D3379 [31] for single fibre [9,21,22]. Seven categories of EFB fibre were tested after being treated in hot water at different temperatures: room temperature (RT), 50°C, 60°C, 70°C, 80°C, 90°C and 100°C at soaking hours 1, 2 and 3. Twenty samples are randomly chosen for each different temperature for testing [9,28] and the average strength of the fibres is determined [28]. 140 samples were tested for each soaking hour with a total of 420 samples for all soaking times. The individual fibre is affixed to a cardboard frame in accordance with the provided schematic diagram, as depicted in Figure 1 (a). These specimens were left for 24 hours to produce perfect bonding between cardboard and EFB to avoid failure occurring at the joints of the samples [9]. The frame sides were meticulously severed at the central section subsequent to securing the ends of the paper frame using the clamps of the INSTRON machine [28]. The breaking load of EFB was determined using the Instron Universal Testing Machine equipped with a 10 kN load cell, as depicted in Figure 1 (b) [9].

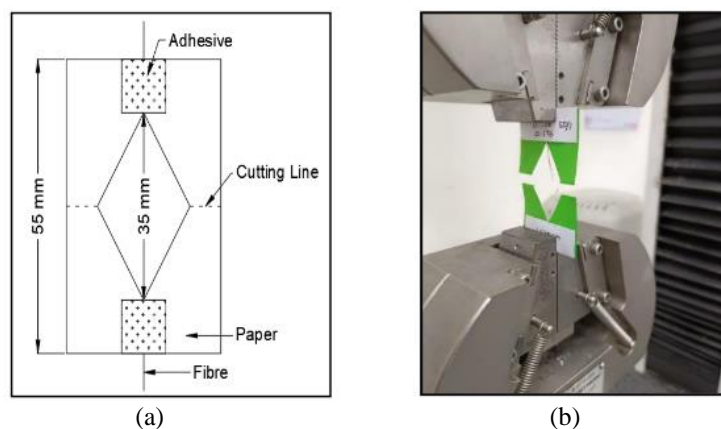


Figure 1. Tensile test (a) Schematic sketch [9] (b) The experimental setup for tensile test

2.2. Surface Morphological properties of EFB fibre

The physical properties of EFB fibres are monitored from the surface morphology of EFB fibres using ZEISS Scanning Electron Microscope (SEM) at the Environmental Engineering Laboratory, Universiti Tun Hussein Onn Malaysia, as shown in Figure 2 (a). In this study, SEM investigations were carried out at magnifications between 1500 to 2000 to better understand the surface characteristics. It is appropriate to make the conclusion through the observation of changes in EFB fibre surface morphology due to the variational treatment effect. The EFB fibre used in this physical investigation consists of RT, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C at soaking hours 1, 2 and 3 and is located based on plate numbering as shown in Figure 2 (b).

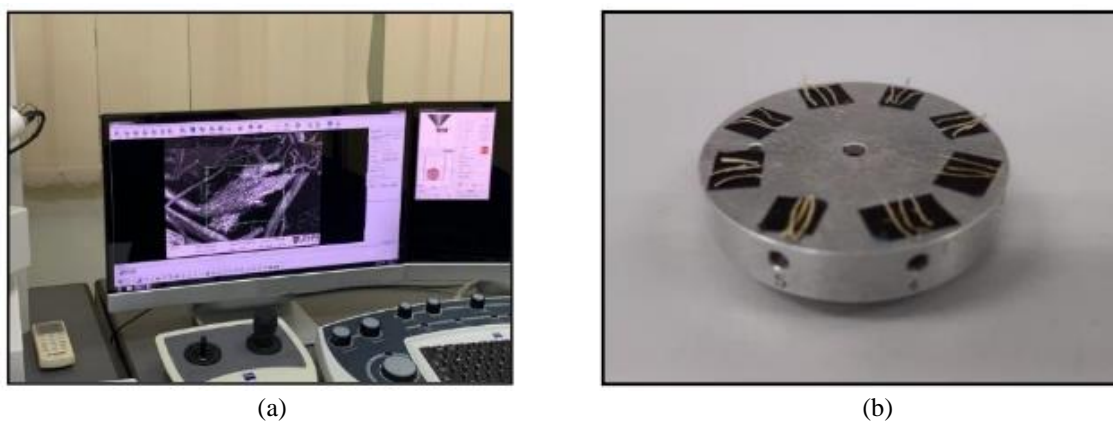


Figure 2. SEM test (a) Image EFB fibre from SEM equipment (b) Sample preparation

3. Result and discussion

3.1. Tensile Strength of EFB fibre

The average tensile strengths of the control sample (RT) and heat-treated EFB fibre are presented in Figure 3. All figures show the increment trend from RT, 50°C, 60°C, 70°C, 80°C, 90°C and 100°C. Soaking EFB in hot water for 1, 2 and 3 hours has increased the tensile strength of the fibre. The treated EFB appeared to be significantly increasing the tensile strength of the fibre compared to the control sample. As demonstrated in Figure 3, the control sample recorded an average value of 169-204 N/mm². This value is within the range of tensile strength obtained by previous researchers such as Nik Soh et al. [9] at 180 N/mm². The tensile characteristics of EFB fibre were shown to increase significantly when treated with NaOH at concentrations of 0.4%, 1%, and 4%, in comparison to the untreated fibre. The highest recorded value for tensile properties was 422.90 N/mm², which was achieved at a NaOH concentration of 4%. This observation aligns with the findings of Iskandar et al. [21], who also reported that the tensile properties of treated EFB fibre were higher than those of untreated fibre. This improvement can be attributed to the improved crystallinity of the fibres resulting from alkali treatment. Similar to hot water treatment, the trend gradually increases for 1 hours soaking time with a value of tensile strength of 171, 215, 289, 304, 352, 435 N/mm² and 2 hour soaking time 195, 229, 301, 348, 363, 450 N/mm², respectively. The possible reason for the finding was that hot water treatment also led to major changes in the fibrillar structure of fibres and removed the amorphous components causing changes in the deformation behaviour.

The tensile strength decreases when the EFB is subjected to a longer duration of soaking. In this research, the value of tensile strength for 70°C, 80°C, 90°C and 100°C at 3 soaking hours has been reduced. The percentage reduction was almost 22% when compared to 2 hours. However, findings from Momoh et al. [5] show the fibre in radial cracking due to the treatment temperature of 100°C at 2 soaking hours. According to their research, the authors concluded that the use of hot water treatment is a more ecologically sustainable approach. They determined that the optimal treatment duration is 30 minutes at a temperature of 100°C. This treatment resulted in a significant enhancement of 40% in tensile strength. Meanwhile, the effectiveness of the treatment decreased at 2 soaking hours. However, the findings may be affected by the different types of fibre and its plantation region, which may affect the fibre properties [32].

The distribution of tensile strength against EFB diameter is presented in Figure 4. An increase in the soaking time with temperature has reduced the diameter of EFB fibre, simultaneously increasing the tensile strength of the fibre. However, inconsistency in EFB fibre diameter has also affected the results,

as some samples did not break in the middle of the predicted breaking zone. The predicted breaking zone is considered in the void area between mounting tab of 35mm length as shown in Figure 1 (a). The diameter of each sample must be measured at three points in the predicted breaking zone (top, middle, bottom) using a micrometre. The tensile strength calculation of EFB fibre was measured based on the diameter of the fibre at the breaking zone. This study produced a diameter range 0.17-0.26mm after hot water treatment. It was proven by research conducted by Nik Soh et al. [9] and Bakri et al. [33] that the diameter of the fibre affected the properties of the fibres; when the diameter was increased, the strength of the fibre decreased. Bakri et al. [33] reported that the diameter of EFB fibre is varies between 0.4 mm and 0.72 mm and influences the tensile strength of the fibre where the smaller the diameter of fibre, the higher tensile strength. This is caused by the change in surface morphology of treated fibre by removing certain components such as wax, lignin, and oil from the EFB strand [6,28]. The average tensile strength of EFB is predicted to influence the mechanical properties of the EFBCB sample as the previous study revealed that the tensile properties treated with NaOH have increased with the increment of the tensile strength of single fibre [9]. Additionally, Omoniyi [27] proved that pre-treatment of EFB fibre with water at a temperature of 60°C has produced better physical and mechanical properties, as well as a treatment in 8% NaOH that increased MOE from 5.5 to 8.9 GPa and MOR from 3.6 to 7.3 MPa, and decreased WA from 26.2 to 12.8% and TS from 2.5 to 0.5%. It was concluded that hot water pre-treatment increased the mechanical properties (MOE and MOR) and enhanced the physical properties (WA and TS) of the cement composite.

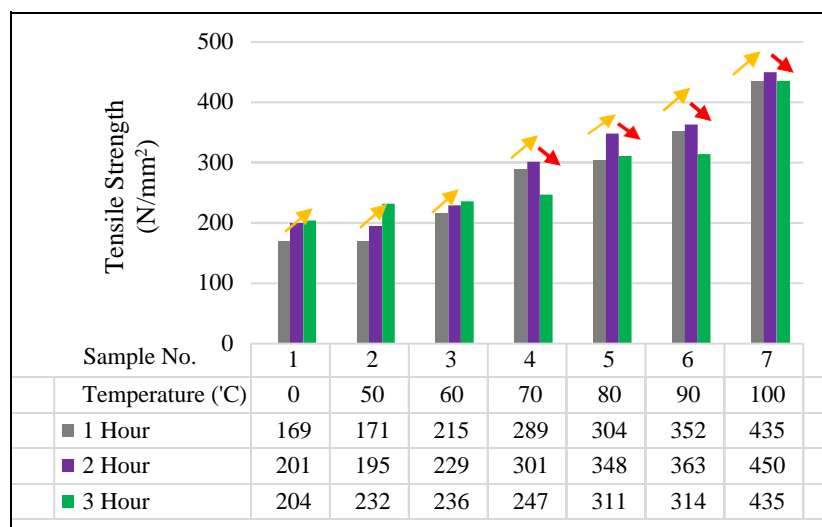


Figure 3. Effect on tensile strength for different hour soaking time of hot water treatment

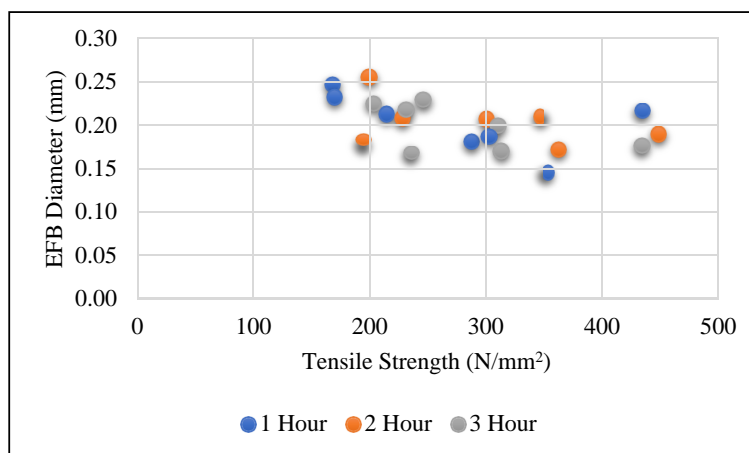


Figure 4. Effect on EFB fibre diameter for different hour soaking time of hot water treatment

3.2. Surface Morphology of EFB fibre

The surface morphology of EFB fibres was conducted using a ZEISS Scanning Electron Microscope (SEM) with magnifications of 1500-2000x to obtain a better view of surface characteristics. Obviously, there is a large amount of silica bodies attached to the fibre surface for the room temperature (RT) sample compared to the treated sample (50°C, 60°C, 70°C, 80°C, 90°C and 100°C) at soaking hours 1, 2 and 3 as shown in Figures 5, 6 and 7. Silica body is still present in large amounts at lower temperatures as can be seen on 50°C, 60°C, 70°C at soaking hours 1, 2 and 3 which would inhibit the hydration process of the cement-*EFB* mixture as shown in Figures 5, 6, 7 (a-d). As the temperature increased, the number of silica bodies removed from the fibre surface increased at high temperatures (80°C, 90°C and 100°C) at all soaking hours and leaving empty circular craters on the fibre surface (Figures 5, 6, 7 (e-g)). The empty circular craters will enhance the bonding ability of the cement matrix, aligning with previous research by Iskandar et al. [21]. Surface modification treatments can increase surface roughness, remove impurities, and enhance the number of hydroxyl groups or new functional groups, thereby improving the bonding between the fibre surface and matrix. Thus, a strong fibre-matrix interface is important, and fibre surface treatments can initially improve the interfacial adhesion between the fibre-matrix and enhance the good mechanical properties of composites [34]. It can be shown as in Figure 8 that pore diameter and area of empty circular of EFB fibre treated increased between different temperatures from 60°C and 70°C at 1 hour soaking time which are an average diameter and area for 60°C of 10.42µm, 86.41µm² and 70°C of 11.07µm, 96.90µm². Similar results to 1 and 2 hours at an average diameter and average area for 70°C for 2 hour soaking time increased to 13.02µm, 134.33µm². However, at a level of 70°C at 3 soaking hours as shown in Figure 7 (d), the lignin layer starts to damage. While Figures 7(e) and (f) also show a similar image, fibre starts to damage when empty circular craters change to a linear surface. Meanwhile, a study by Nik Soh [35] proved that the limitation of alkaline treatment when increment in NaOH concentration until 5% has damaged the lignin and part of the cellulose component in fibre, preventing better interlocking with the cement matrix and inhibiting cement hydration. This finding was in line with the research done by Nordin et al. [36], the result decreased after 6% NaOH treatment, as excessive treatment removed boundary layers and deteriorated fibre particles, affecting the strength of EFB fibres.

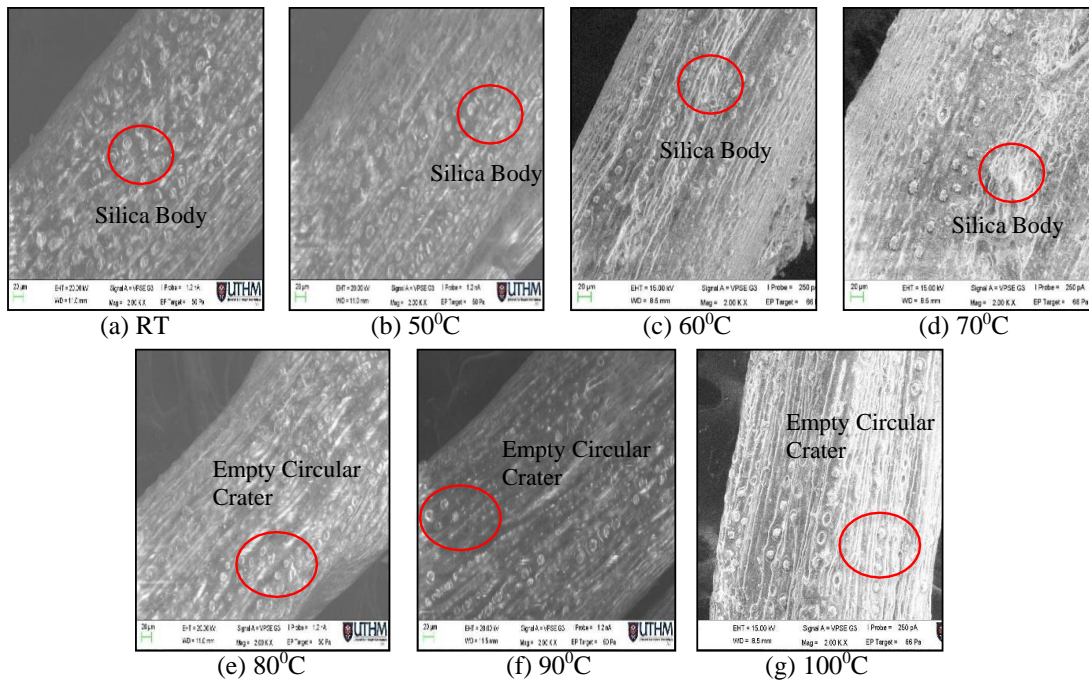


Figure 5. SEM image of EFB treated with hot water treatment (1 hour)

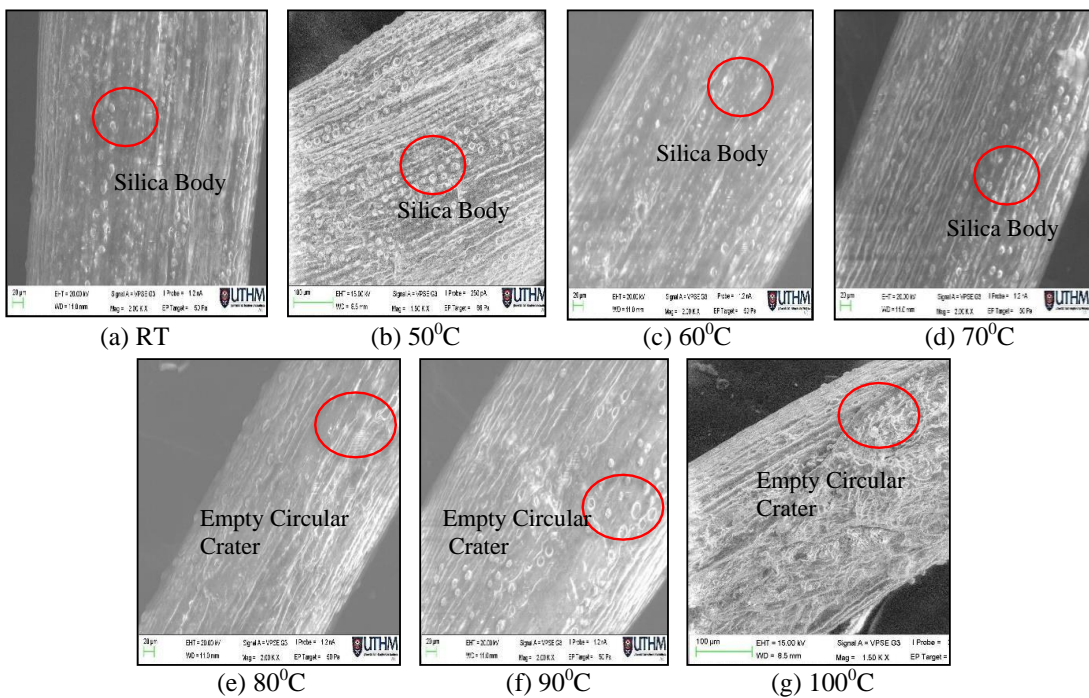


Figure 6. SEM image of EFB treated with hot water treatment (2 hour)

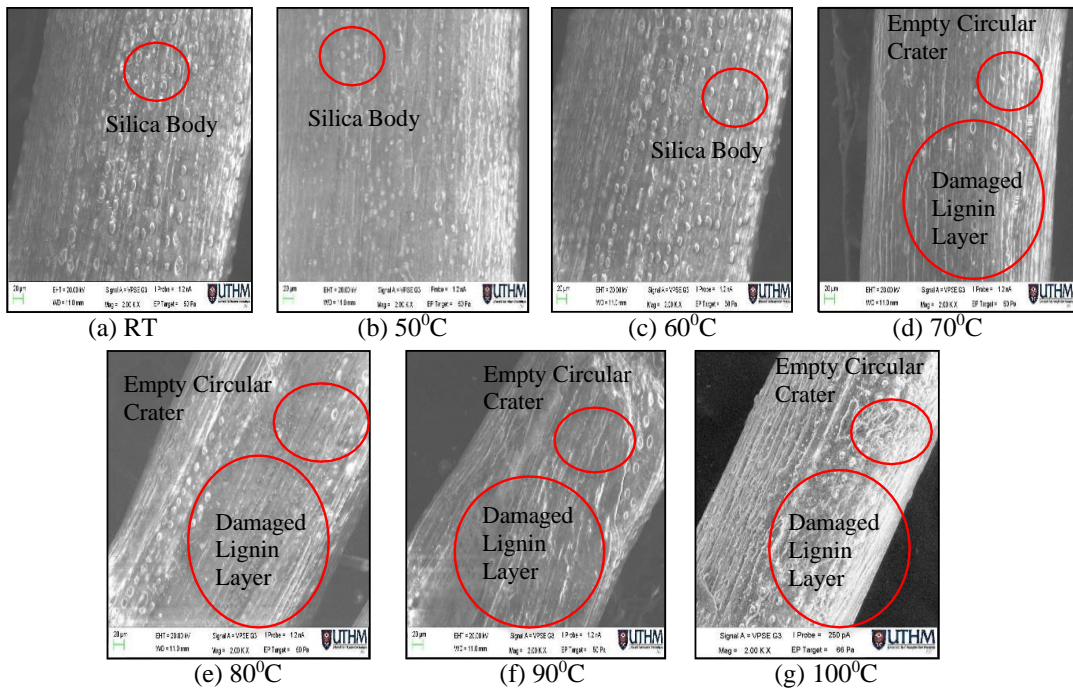


Figure 7. SEM image of EFB treated with hot water treatment (3 hour)

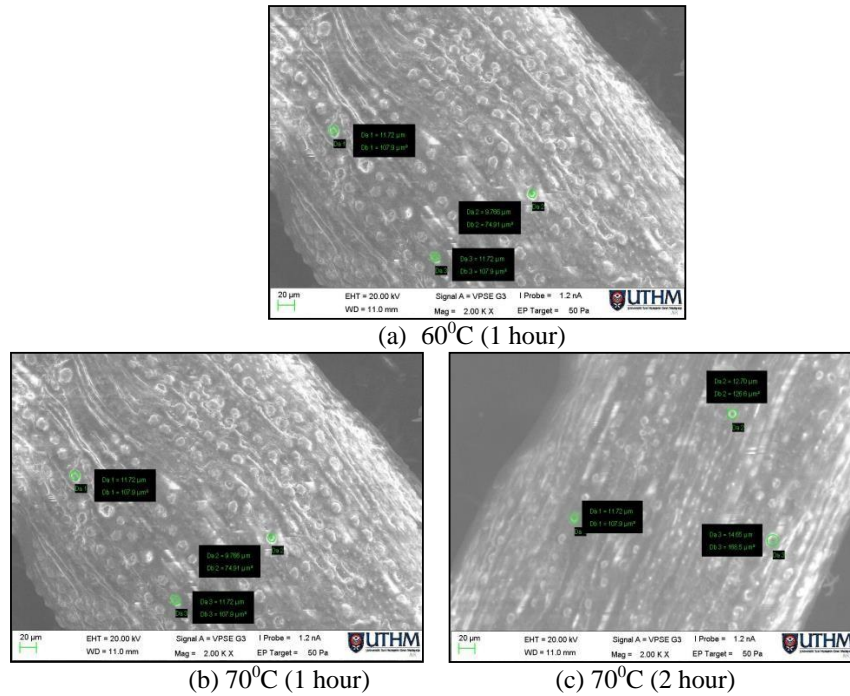


Figure 8. Pore diameter and area of empty circular of EFB fibre

4. Conclusions

The findings of this study revealed that the EFB fibre potentially contributes to the performance of the physical and mechanical properties as reinforcing materials in the composite. The impact of tensile testing on the EFB fibre increased dramatically with temperature at all soaking times. However, longer soaking times have contributed to the reduction in tensile properties. Another finding shows that an increase in the soaking time with temperature has reduced the diameter of EFB fibre, and simultaneously increased the tensile strength of the fibre. Surface observation showed that as temperature increased, the number of silica bodies removed from the fibre surface increased (80°C, 90°C and 100°C) at all soaking hours and leaving empty circular craters on the fibre surface. However, the temperature at 70°C (3 soaking hour) showing the circular craters on the EFB surface and the lignin layer starting to be damaged. Therefore, hot water treatment also has the limitations as other treatments when an increment in soaking time, damages the lignin and part of the cellulose component in fibre. This condition prevents good penetration for bonding with the cement matrix, which inhibits the cement hydration process and reduces the composite's strength. The obtained results exhibit considerable promise for future investigation, as they provide evidence for the viability of incorporating EFB fibres as reinforcement agents in composite materials.

5. References

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Acknowledgements

This research was supported by Universiti Tun Hussein Onn Malaysia (UTHM) through Tier 1 (vot H900).