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Characterization of chemically treated waste wood fiber and its potential application in cementitious composites

Merve Sönmez Tuğluca [a,b], Emine Özdoğru [c], Hüseyin İlcan [a,b], Emircan Özçelikci [a,b], Hüseyin Ulugöl [d], Mustafa Şahmaran [a,*]

[a] Department of Civil Engineering, Hacettepe University, Ankara, Turkey
[b] Institute of Science, Hacettepe University, Beytepe, Ankara, Turkey
[c] Department of Civil Engineering, Yildiz Technical University, Istanbul, Turkey
[d] Department of Civil Engineering, Ankara University, Ankara, Turkey

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ABSTRACT

In recent years, the utilization of various fibers in cementitious composites gained remarkable popularity worldwide. Unlike well-known fibers, this study focused on examining the incorporation of waste wood fiber (WWF) assorted from construction and demolition sites into the cementitious matrix. In the first stage of the study, alkali-treatment of the WWF was carried out with different concentrations of sodium hydroxide (NaOH) solution (i.e., 1.0 M, 2.0 M, 2.5 M, 5.0 M, 10.0 M) and treatment durations (i.e., 2 h, 4 h, 6 h, 12 h) to eliminate impurities and enhance fiber properties and fiber-matrix compatibility through surface treatment. To investigate alkali-treatment efficiency, microstructural analyses were conducted on the untreated and alkali-treated WWFs by Scanning Electron Microscopy and Fourier Transform Infrared Spectrophotometer measurements. Afterward, physical and mechanical performance assessments were conducted by water absorption test, compressive and four-point bending tests on the WWF-incorporated cementitious composites to monitor the compatibility behaviour of the WWF-matrix interface. Microstructural examinations showed that after the alkali treatment, impurities (*i.e.,* painting, hemicellulose, lignin, etc.) on the WWF surface were successfully removed; however, higher alkali concentrations (beyond 5.0 M) and longer treatment duration (after 6 h) lead to deterioration, fragmentation, and a more heterogeneous structure due to the degradation of long cellulosic chains of WWF. Four-point bending test results showed that the flexural strength capacity of 2.5M-2 h-WWF-composite was 7.9 MPa, which was higher than untreated WWF-composite and plain mortar by 34.7% and 39.8%, respectively. Furthermore, the 2.5M-2 h treated WWF composite had the highest compressive strength of 46.3 MPa, which was higher than plain mortar, and the untreated WWF composite by 6.4% and 21.5%, respectively. The elimination of impurities such as painting, lignin, hemicellulose, and other inorganic compounds at the optimal alkali treatment concentration and time increases the WWF surface area and strengthens the fiber-matrix interface adhesion, resulting in a stronger matrix-WWF interface connection and an increase in load-carrying capability. The study's findings are believed to encourage the usage of alkali-treated WWF in cementitious matrix with improved mechanical performance and high waste upcycling.

1. Introduction

The continuous growth in the world population has yielded an increment in construction activities, hence a tremendous amount of construction and demolition waste (CDW) generation due to repair/renovation/maintenance/demolition activities [1–3]. Such increment in the production of CDW has severe consequences, including an increment in uncontrolled waste disposal sites, increased pollution and contamination, and economic burdens [4]. To overcome such drawbacks, the recycling/valorization of CDWs has gained popularity among researchers. Although some portions of CDWs (i.e., concrete rubble and masonry units) were utilized as a base material in road construction, and in the production of conventional cementitious systems, and geopolymer binders, the waste wood fractions correspond to %7 CDWs, were not included in the value-added chain considerably [5]. 75% of the waste wood fraction is generally deposited in sanitary landfills. The

* Corresponding author.
E-mail address: sahmaran@hacettepe.edu.tr (M. Şahmaran).

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remaining part is used as secondary material such as fuel, compost, or component manufacturing other materials [5]. However, some literature studies showed that wood could be utilized as a veneer, chip, and panel [6–8]. Besides, the usage of waste wood as fiber also recently attracted the attention of researchers to improve the mechanical properties of building materials [9].

Wood-based fibers typically consist of live and dead cells, depending on their origin, and are mainly composed of cellulose, hemicellulose, lignin, and pectin [10,11]. The dominant part of the fiber, cellulose, is comparable to other natural fibers regarding laying down microfibrils with substantial hydrogen bonding, resulting in a strong crystalline structure [11]. Hydrogen bonds in these cellulose fibrils not only have a strong influence on its physical properties (e.g., solubility, hydroxyl reactivity), but they also play an essential role in its mechanical properties [12,13]. The microfibril structure of wood fibers has the exceptional performance property of high strength to weight ratio [11]. While another part of wood fiber, hemicelluloses, is the primary factor determining moisture absorption capacity, accessible cellulose, nanocrystalline cellulose, and crystalline cellulose's surface also play important roles [6]. Also, as another component of wood fibers, lignin can effectively bind and connect cells. However, the lignin separation that remains on the fiber surface during processing can reduce inter-fiber bonding [11]. Pectin, which exists in a small amount, presents a less critical role in cellulosic material due to the dominance of other components and the complexity of modifying pectin [14]. Additionally, wood fiber surfaces contain a high concentration of hydrophilic groups that may cause poor interfacial compatibility [15].

Interfacial compatibility problems between the wood fiber and composite matrices were related to the wood fibers' microstructural element properties. Sandermann et al. [16] reported that non-cellulosic materials, including sugars, starches, and tannins, can hinder the cement hydration period. This situation has arisen because the sugar-acid anions disrupt the calcium silicate hydrate nucleation sites on the initially positively charged surfaces, causing cement hydration to be inhibited [17]. In addition, resins, fatty acids, and salts are some of the other extractives that pose problems with cement wood compatibility [18]. Therefore, to increase the compatibility of wood fiber-composite structure, in the literature studies, various chemical and physical surface treatment procedures have been conducted to change the surface morphology of the fibers [11,19,20]. Stretching, calandering, and thermo-treatmentment are physical procedures that do not modify the fibers' chemical composition, which must be vital for better compatibility [21–24]. On the other hand, in the literature studies, various surface chemical modifications of wood fibers, such as alkali treatment [15], acid treatment [25], isocyanate treatment [26], acrylation [27], silane treatment [28], and peroxide treatment [29] have been applied to improve fiber strengths and fiber-matrix adhesion in wood-fiber composites to varying degrees of success [30]. Among them, alkaline treatment, also known as mercerization, is the most commonly used chemical treatment for wood fibers due to its low cost, excellent efficiency, and easiness [25,31–33]. Alkali treatment of cellulose fibers can be affected by the alkaline type and concentration of the alkaline solution, as well as the temperature, period of treatment, and material characteristics [34,35]. The fundamental mechanism of alkaline treatment reinforcement is the degradation of non-cellulosic impurities [36] by (i) alkaline solution dissolves materials other than cellulose to expose more reactive hydroxyl sites on the surface and also contributes to roughness and high surface area by providing strong interfacial contact and adhesion between fibers and polymer, and intermolecular hydrogen bonds are partially removed, fiber bundles are fragmented, (ii) Na^+ (or other such as K^+) ions replace the H^+ ions on the fiber surface and break the hydrogen bonds inside and between the fibers [37].

Alkali treatment on various natural fibers was studied in the literature to investigate fiber characteristic properties. In the study of Mishra and Basu [38], alkali treatments resulted in weight loss (9.3 ± 1.5%) and fiber color change from brown to dark brown due to the removal of wax oil and lignin. In another study, Rahman et al. [39] observed that treatment with 20% NaOH results in the highest weight loss of 12.65% at 25 °C in coir fiber compared to other NaOH ratios of between 5 and 50%. In another study [40], bamboo fiber was immersed in 2, 4, 6, and 8% aqueous NaOH solutions for 1, 2, 6, 12, 18, and 24 h and with the treatment of bamboo fibers for 12 h with 4% NaOH, tensile strength presented the highest increment of 28%. However, increasing the soaking time to 18 or 24 h has a negative impact on the tensile properties of bamboo fibers. Another study investigated the alkali treatment of bamboo fibers with NaOH concentrations of 1%, 3%, and 5% for 24 h at room temperature, and it was concluded that the increase in alkali concentration eliminates impurities and other organic elements through the fiber surface [41]. Barman et al. [42] examined the effect of various (2.5–20%, with the increment of 2.5%) NaOH concentrations on the pinewood fibers. Results showed that 10% NaOH concentration eliminated the amorphous lignin considerably, and cellulose content decreased beyond 10% NaOH concentration. They also stated that the increase in NaOH concentration beyond the 10% ratio caused the degradation of the crystallinity structure of cellulose together with the elimination of lignin and hemicelluloses. According to another study, the surface roughness increases as aqueous NaOH concentration increases because of raises the crystallinity index by removing the amorphous phase [43]. Based on the preceding literature, it is possible to conclude that alkali treatment is useful for modifying the chemistry on the surface, cleaning the fiber surface, reducing moisture uptake, and improving surface roughness.

Literature studies highlighted the benefits of using natural fiber in composites for the purposes such as better flexural strength, post-crack load-bearing capacity, improved bending strength capacity, fire resistance, and low thermal conductivity [17,44–47]. Li et al. [44] studied the compressive and flexural properties of hemp fiber reinforced concrete, and their findings revealed that the flexural toughness of hemp fiber reinforced concrete was enhanced by 144%. In another study, Li et al. [45] found that the flexural toughness of cementitious composites containing coir fiber treated with 1% NaOH solution has increased approximately more than ten times. Besides, the microstructural examinations showed that the alkaline treated coir fiber reinforced cementitious composite yields superior interfacial bonding. According to Soroushian et al. [46], the flexural strength values of composites (incorporate 2% fiber mass fraction) with kraft fiber were greater than three times that of plain mortar, whereas composites with pulp fiber were greater than two times that of plain mortar. Andiç-Çakir et al. [47] evaluated the mechanical characteristics of NaOH-treated (5% NaOH solution for 2hr) coir fiber composites under compressive, flexural stresses and toughness. They reported that composites' flexural and compressive strength increased as the fiber content increased. A similar increment was also observed with the alkali-treated fibers compared to the untreated ones. Inclusion of fibers into composite resulted in a reduction in unit weight, hence enhancement in strength/weight ratio. Eventually, it was highlighted alkali treatment improves the fiber-matrix interfacial bonding property resulting in the development of flexural strength and toughness. Wong et al. [41] also investigated the feasibility of using bamboo fibers (untreated and treated with 1, 3, and 5% NaOH concentrations) as reinforcement for polyester composites. It was revealed that removing hemicelluloses, lignin, and surface impurities from the fiber surface via alkaline treatment may decrease the density of the refined fibers and make the bamboo fiber surface much rougher compared to the untreated. Alkali treatment improved the ductility compared to untreated ones. However, the opposite trend was detected in density, yield strength, ultimate tensile strength, and Young's modulus using alkali-treated bamboo fibers in this study.

Based on the aforementioned issues, the main aim of the study is the evaluation of the possible upcycling performance of the CDW-based wood fiber in cementitious composites. Although CDW-based wood fiber is also known as a lignocellulosic nature similar to well-known natural fibers such as bamboo, sisal and, coir, it can be considered low

quality end-of-life material due to the exposed external influences throughout its long period of service life. Besides its low quality, the great variety of wooden units as a result of utilizing different types of substances in their manufacturing for the building industry causes great reluctance to recycle these units as wood fibers. Moreover, it is thought that the common treatment methods (i.e., alkali type/molarity, treatment durations) that apply to the natural fibers and their reinforcement ratios may not be fully employed identically on the waste wood fiber due to its low quality and varied nature. As documented elsewhere, the compatibility of natural virgin fibers such as bamboo, sisal, hemp, and coir has been improved by the alkali treatment method [41,47,48]. Although a tremendous amount of CDW-based wood fiber was obtained, the literature has not fulfilled the valorization of those fibers in construction applications [5]. The number of studies on improving natural fibers used in the construction industry is limited and inconsistent. Particularly, the improvement of waste wood fiber (WWF) from different sources and the compatibility of WWF into cementitious systems remains unknown, according to the authors' best knowledge. This study, therefore, targeted to present comprehensive research for the utilization of alkali-treated WWF with different molarities and durations in cementitious composites. In this context, NaOH solution molarities of 1.0 M, 2.0 M, 2.5 M, 5.0 M, and 10.0 M and durations of 2hr, 4hr, 6hr, and 12 h were applied to enhance the CDW-derived wood fiber. Changes in the microstructural properties of WWF were revealed through Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) analysis, along with, the mechanical and microstructural properties of WWF-composite were reported by load-carrying capacity and compressive strength test.

2. Experimental methods

2.1. Materials and mixture proportions

CEM I 42.5R Ordinary Portland Cement, silica-rich quartz sand, and Class-F fly ash were used as the ingredient for all matrix mixtures in this study. The mixtures also included a superplasticizer (SP) (polycarboxylic ether-based) to provide sufficient workability without losing mechanical performance. The mixture was designed as an Engineered cementitious composite (ECC) matrix and to clearly observe the effect of WWF-addition and to avoid loss of workability, a 1:3 cement-fly ash usage ratio was chosen. WWF assorted from the different construction units and demolition sites with unknown origin and WWF were characterized physically and microstructurally, regardless of the wood type from which they were produced. Because various substances are mixed with the original wood in the manufacture of wooden units and CDW-based WWF is exposed different conditions to during its service life. Thus, the original wood that used in the manufacturing of wooden units can not represent the CDW-based WWF. Then waste wood was chipped by an industrial chipping machine regarding a maximum size of 2.00 mm. Then, WWF having a particle size of 0.85–2.00 mm was obtained by sieving via #20 and #10 sieves in the laboratory. It was ensured that wood dusts below 0.85 mm, which has a very high-water absorption potential, are eliminated. The dimensions of 100 randomly selected WWFs were measured, and the average length, width, and depth of the obtained WWFs were recorded as 12.45 ± 3.53 mm, 1.3 ± 0.23 mm, and 0.5 ± 0.18 mm, respectively. The aspect ratio was determined as approximately 10. Two different mixtures were designed: Plain mortar and a composite with 2.00% of total volume replaced by WWF. The replacement ratio of WWF in the mixture was adjusted as 2.00% to prevent possible workability loss. Detailed mixture proportions were shown in Table 1.

2.2. Alkali treatment protocol

The WWFs, which are also needed to clean from C&D waste-based incompatible parts on the surface such as painting was first pre-dried at 50 °C until a weight loss of less than 1% was obtained in the weight measurements of the WWFs at 3-h intervals. The dried WWFs were immersed into alkali for 2, 4, 6, and 12 h with 1.0, 1.5, 2.0, 2.5, 5.0, and 10.0 molarities of solutions prepared with flake form NaOH having a purity of 98% and a density of 2.13 g/cm^3. The views of the untreated WWF and alkaline-treated WWF which demonstrated color changes as a result of the implementation of different alkali molarities and durations were given in Fig. 1. The color changes in alkaline-treated WWF were seen from brown to dark brown due to the removal of wax oil and lignin [49].

Since the reaction of NaOH with water is an exothermic reaction, the prepared solutions were kept at room condition until reached ambient temperature. Afterward, the pre-dried WWFs were immersed into the corresponding solutions at room temperature for assigned treatment durations. At last, WWF was rinsed and dried in an oven at 50 ° C.

2.3. Casting and curing

The mixing procedure included the following steps: (i) solid ingredients of the mortar were first mixed for 90 s at low speed to provide homogenous distribution, (ii) water was added to the mixer and mixed together for 90 s, (iii) SP was included into mortar mixture slowly and mixing process continued 120 s at medium speed, (iv) WWF was added into the mixture gradually, and the resulting mixture was mixed at a medium speed until homogenous distribution was provided. Completing the mixture, composites were poured into six replicates of 50-mm cubic specimens for the compressive test and six separate prismatic specimens with dimensions of 240 × 80 × 15 mm (length × width × height) for the flexure test. After the samples hardened, they were removed from the molds and kept in a water cure at room temperature to ensure the continuity of hydration and prevent shrinkage formation.

2.4. Test methods

Scanning electron microscopy (SEM) was conducted to observe surface morphology modifications of the WWF after selected alkali treatment procedures. Besides, SEM analysis was conducted on acquired samples from the matrix-WWF interfacial zone to characterize the interfacial bonding between cementitious matrix and untreated and treated WWF. SEM analysis was performed using the Tescan GAIA3 device, which was operated at 30 kV under vacuum conditions.

To investigate the effects of treatment by observing the removal of incompatible wood components, FTIR spectroscopic analyses of the untreated and treated WWF were performed using a Thermo Fisher Nicolet iS50 spectrometer focusing on the wavenumber scanning region of 400–4000 cm^{-1}.

The samples whose curing period was completed were removed from the water curing and allowed to dry before testing to conduct

Table 1
Mixture proportions of cementitious systems.

Mixture	Mixture proportions (kg/m^3)					
	Cement	Class F-Fly Ash	Silica Sand	Water	Superplasticizer	WWF
Plain mortar	408	896	473	346	6.51	–
WWF-included composite	400	878	464	339	6.39	17.8

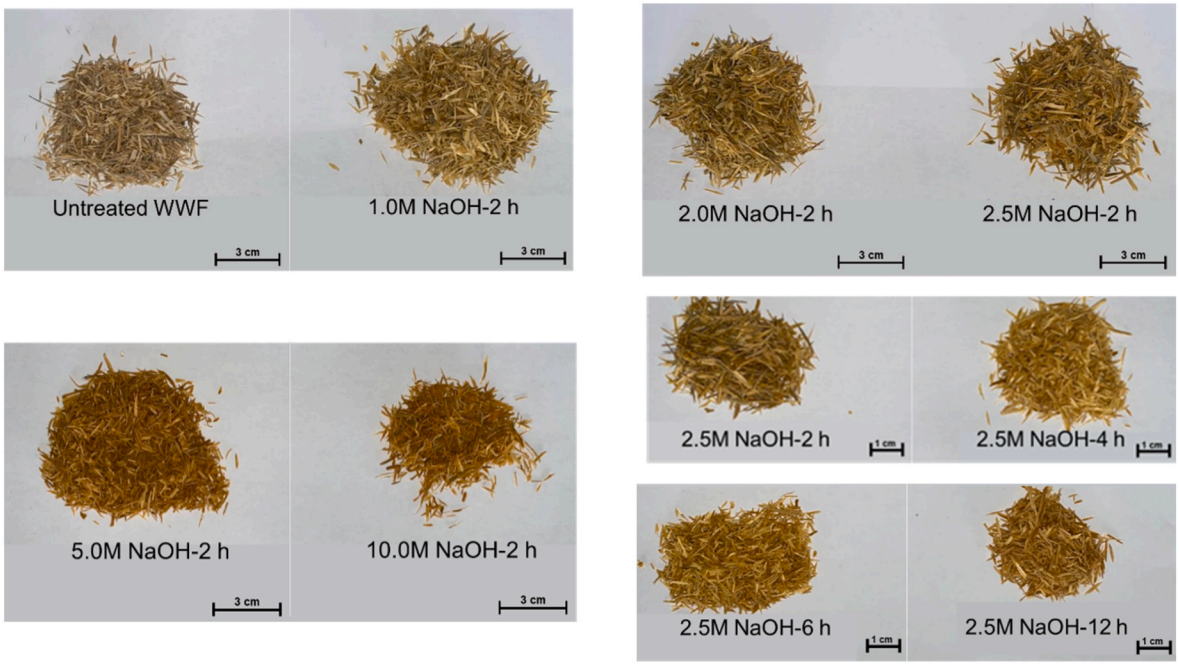

Fig. 1. Untreated WWF and alkali-treated waste wood fibers for different concentrations and durations.

corresponding tests. The bending tensile test was carried out on the prismatic samples with a loading speed of 0.005 mm/s in accordance with ASTM C78 standard [50] in a deformation-controlled electro-mechanical test device (with maximum measuring capacity of~4448 N and load cell accuracy of ±0.1%). The 210 mm span on the samples was divided into three equal sections, and the points on which the loads would be applied were determined. The deformation that will occur at the midpoint of the samples during the experiment was accurately recorded using a deflectometer. The maximum load-carrying capacity and displacement were recorded through four-point bending tests

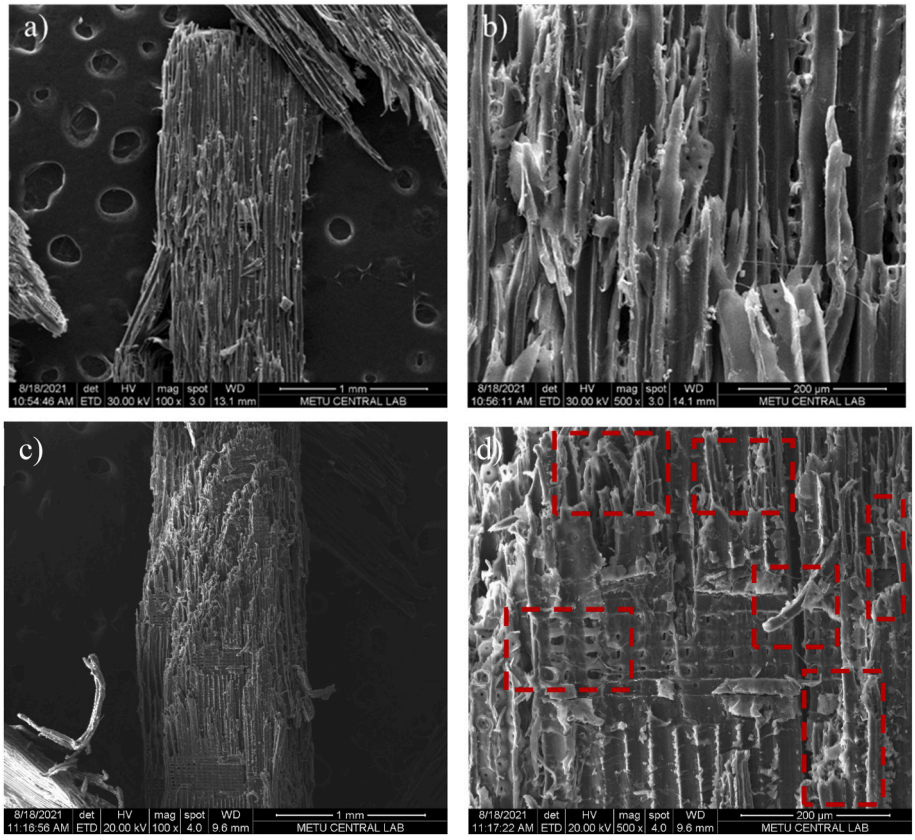

Fig. 2. (a) and (b) Untreated WWF, (c) and (d) Treated WWF with 2.5 M NaOH solution for 12 h (red dashed lines indicate hollows regions and ruptured structures). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

conducted on six prismatic samples at the end of 28-day curing. The compressive strength test was performed on the six replicate samples at the end of curing age using a compression device with a loading speed of 0.9 kN/s according to the ASTM C109 standard [51]. Additionally, water absorption and density measuring tests were conducted on WWFs, 28-day plain mortar, and WWF-included composites.

To observe the means and standard deviation values, results of six samples were used in statistical analysis. Results obtained with treated WWF were compared with plain mortar and untreated WWF-composite using one-way ANOVA analysis, with Holm-Sidak method and Fisher's LSD 5% approach.

3. Results and discussion

3.1. Scanning Electron Microscopy (SEM) analysis

To investigate the effects of different molarities and treatment durations on the surface properties of WWF, SEM images of the untreated WWF and treated WWFs by 2.5 M, 5.0 M, and 10.0 M NaOH solution for 2, 4, 6, and 12 h were examined by considering the decays, decomposition, or formation of new products on the surfaces. First, in Fig. 2, SEM images of untreated WWF and treated WWF by 2.5 M NaOH solution for 12 h were compared to the surface morphology of untreated and treated WWF. The cross-section and morphology of WWF have shown a significant transformation through the alkali treatment process with 2.5 M NaOH for 12 h. SEM images show that the WWF surface is rougher than the untreated WWFs (Fig. 2) [52–55]. This could be attributed to reducing surface impurities (non-cellulose substances or cellulose-binding materials), including hemicellulose, lignin, oil, waxes, and other inorganic substances, adversely affecting the adhesion between the WWF and matrix [41,56–59]. The degradation of inorganic materials may also increase the inter-fibrillar region and yield a surface with a rougher texture [60] since non-cellulose substances on the fiber surface are soluble in NaOH solution [41]. Besides, the alkaline treatment caused fiber fibrillation or fiber separation by breaking the structure of bundle fibrils into smaller microfibrils [61–63]. Such fibrillation can enhance the effective surface area (mechanical interlocking adhesion) between cementitious matrix and WWF, leading to better WWF-matrix interface adhesion [8,9]. Furthermore, some hollow/hole regions were observed after alkali treatment which can be evident in removing lignin and other soluble inorganic substance located on the cellulose chain (Fig. 2-d) [41,42]. Additionally, some fiber destruction/ruptures appeared due to the alkali treatment, which means that exposed cellulose is degraded by tested NaOH concentration and treatment duration.

The effects of NaOH concentration on the WWF treatment regarding surface morphologies were monitored on the treated WWF with 2.5 M, 5.0 M, and 10.0 M NaOH for 12hr. In Fig. 3, the morphological changes detected when NaOH concentration increased from 2.5 M to 5.0 M can be attributed to decreasing impurities on the WWF surface, as reported in a study by Alawar et al. [56]. In each molarity, the hole regions were observed, representing the removal of lignin and other soluble inorganic substances [64,65]. On the other hand, a high degree of fragmentation occurred on the surface of the WWFs treated with high concentrations, and the form of the microfibers was destructed (Fig. 3. b, 3. c). The disintegration of WWF began with the increasing NaOH concentration from 2.5 M to 5.0 M, and the situation was more pronounced at 10.0 M. This is attributed to those inorganic impurities being removed in high concentration, and the cellulose chain was become unprotected against the alkaline activity [45], hence the destruction of the crystalline structure of cellulose [42,66]. SEM images showed that beyond the 2.5 M NaOH concentration, although impurities mainly were cleaned, the microstructure of WWFs was started to be broken, which may lead to strength reduction of fiber.

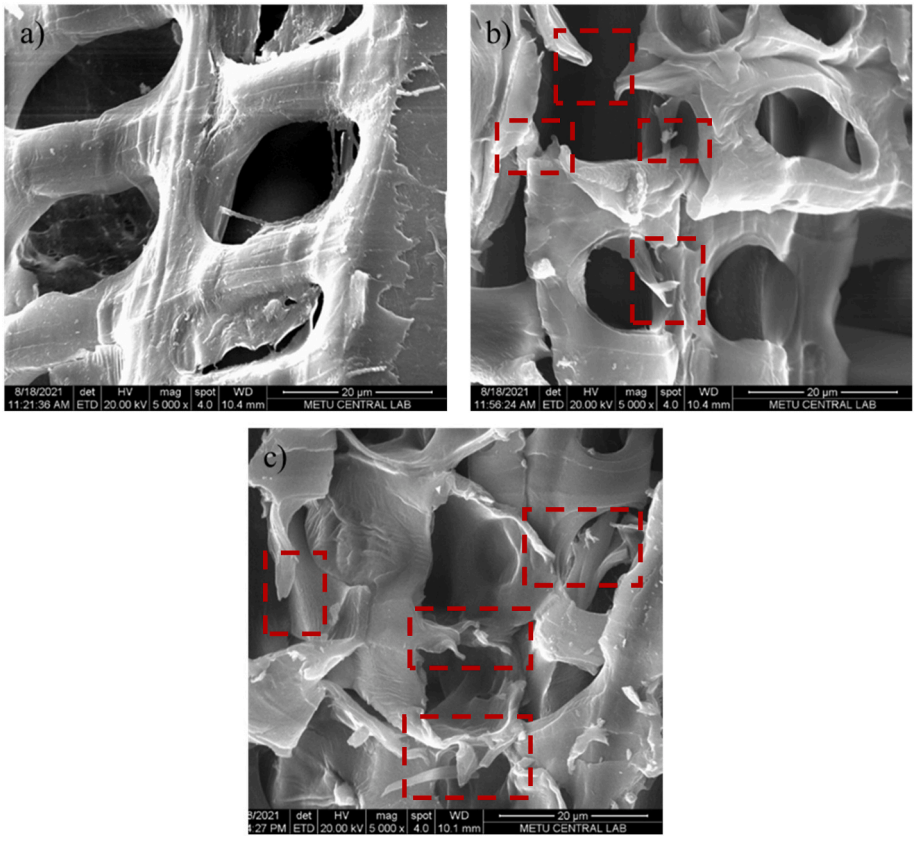

Fig. 3. SEM images of treated WWFs by (a) 2.5 M, (b) 5.0 M, (c) 10.0 M NaOH solution for 12 h (red dashed lines indicate destructed structures). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

SEM images of treated WWFs by 2.5 M NaOH for 2 h, 4 h, and 6 h were given in Fig. 4 to investigate the effects of treatment duration on surface morphology. The formation of holes, fibrillation, and rougher structure was observed on the surface of treated WWFs by 2.5 M NaOH, irrespective of treatment durations. However, treated WWFs with 2.5 M NaOH for 6 h and 12 h showed regional WWF destruction and ruptures. On the other hand, a more consistent fiber arrangement was seen in treated WWFs for 2 h and 4 h. When the alkaline treatment duration changed from 2 h to 4 h, the number of pores increased, and more fibrillation and decomposition of inorganic materials were observed. SEM images also showed that an increment of treatment duration increases the degradation of inorganic substances, but after a particular treatment duration, the microstructure of fiber also started to be degraded.

Consequently, SEM analysis results of WWFs proved that the alkali (NaOH) treatment method is capable of enhancing WWF regarding surface morphology, and NaOH concentration and treatment duration has a significant impact on the treatment of WWF. After alkaline treatment, inorganic substances were removed from the surface of WWFs, and new fibrillation and holes appeared on the WWF, which are beneficial for better mechanical interlocking adhesion. As the 2.5 M NaOH treatment of WWFs for 2 h and 4 h has been successfully performed regarding removing impurities on the fiber surface, lower NaOH molarities of 2.5 M were also tested for further experiment by using FTIR analysis, compressive strength, and flexural strength test.

3.2. Fourier Transform Infrared Spectrophotometer (FTIR) analysis

Fourier Transform Infrared Spectrophotometer (FTIR) analysis characterized the shifts in molecular interactions caused by alkali treatment on the WWF. Changes in the material composition are indicated by changes in the absorption band pattern [68]. In the FTIR spectrum of lignocellulosic fiber, several absorption bands of various chemical groups such as cellulose, hemicellulose, and lignin can be observed. Alkenes, phenolic hydroxyl groups, aromatic groups, b-glucosi linkages, and other functional groups containing oxygen can also be considered other components [67]. To characterize the effect of NaOH concentration and treatment duration on the shifts in molecular interactions, FTIR analysis was applied to the untreated WWF and treated WWF by 2.5 M NaOH for various treatment durations (*i.e.,* 2 h, 4, 6 h, 12 h). Additionally, treated WWF for 2 h duration in different NaOH concentrations (*i.e.,* 1.0 M, 2.0 M, 2.5 M, 5.0 M, 10.0 M) was also investigated. FTIR analysis results of the duration-depending and NaOH concentration-depending parameters were presented in Fig. 5a and Fig. 5b, respectively. It can be stated from the figures that regardless of whether the WWF was either untreated or alkali-treated, it presented typical absorption bands of the components mainly corresponding to cellulose, hemicellulose, and lignin [68].

According to the results, a broad absorption band in the range of 3640–3010 cm^{-1} corresponds to the intermolecular bonded hydroxyl (-OH) groups of the WWF samples [69,70]. Although a noticeable change at this absorption band was not registered in general, minor increases were attributed to the further formation of –OH groups as a result of debonding of crosslinks between cellulose with lignin and hemicellulose, which can be observed as treatment duration increased [68]. Another point that should be noted is the absorption value in this wavelength range of WWFs treated with 10 M NaOH for 2 h decreased significantly, which may be related to the deterioration of WWF integrity in the high alkalinity medium [71]. A small absorption band was also detected in the wavenumbers range of 2960–2830 cm^{-1}, which indicates the presence of C–H stretching groups of cellulose. There was no remarkable change up to 5.0 M NaOH treatment [72,73]. However,

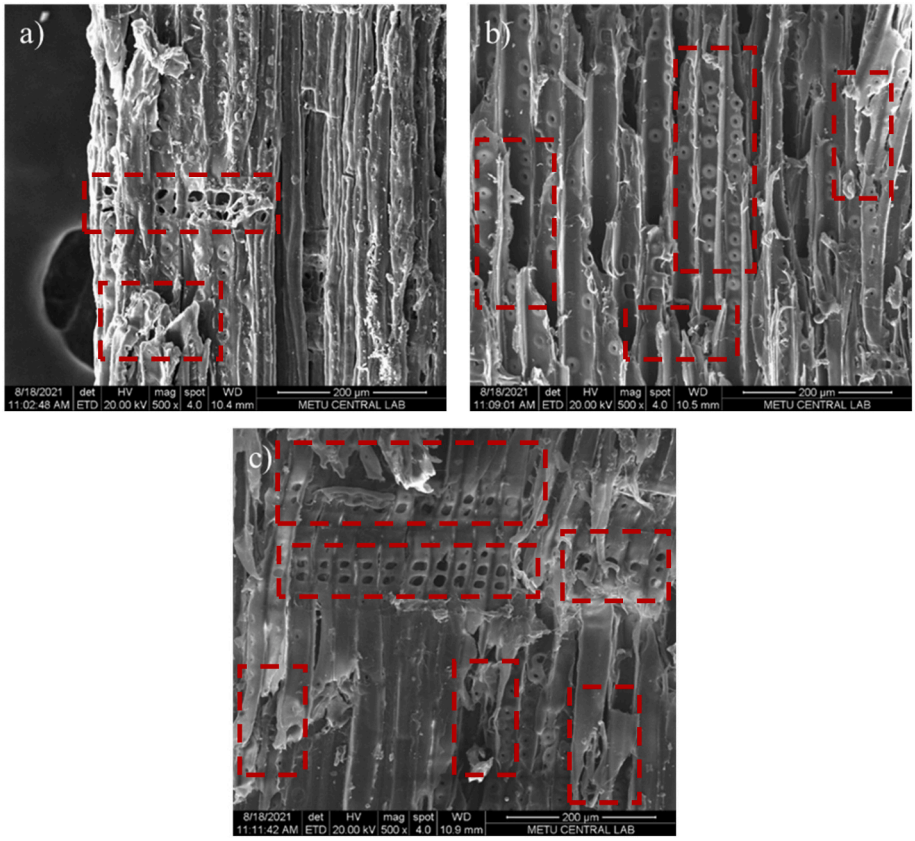

Fig. 4. Effect of increasing treatment duration under constant NaOH concentration: SEM images of WWFs treated in 2.5 M NaOH solution for (a) 2 h, (b) 4 h, (c) 6 h (red dashed lines indicate hollows regions and ruptured structures). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

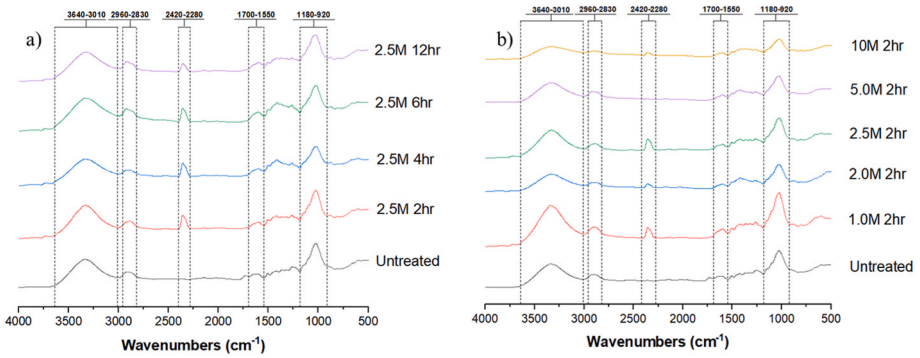

Fig. 5. FTIR spectra of treated WWF by a) 2.5 M NaOH for various durations, b) various molarities of NaOH for 2 h.

in this band, the observed decrease can be related to cellulose structure change for WWF treated with 10.0 M NaOH [74]. Although the formation of the absorption band in the wavenumber range of 2420–2280 cm^{-1} in the alkali-treated natural fibers cannot be generally associated with the studies in the literature, it was thought that such circumstances observed after alkali-treatment are due to modifications resulting from the WWFs-alkali interaction [75]. C–O stretching of carbonyl groups in the structure of hemicellulose, which occurred in the vibration band at a wavelength of 1735 cm^{-1}, was detected in the untreated WWF and completely disappeared after alkali treatment regardless of the treatment duration and NaOH concentration. This can be explained by the fact that the ester type in hemicellulose structures of the WWFs is more prone to be completely eliminated after alkali treatment [68,76,77]. Also, it is known that components such as hemicellulose are more sensitive to reacting with NaOH due to their lower molecular weight. Thus, its amorphous content is more prone to remove compared to cellulose [78]. At last, a broad absorption band was detected in all WWF in the wavelength range of 1180–920 cm^{-1}, independent of treatment duration and NaOH concentration. In the literature, it has been reported that Si–O–Si asymmetric stretching and/or Si–O–C band formation is frequently encountered in this band range. The treatment process causes small increases in the absorption band due to condensation reactions between fiber and agents [32,74].

As a result, it can be stated that optimum alkali concentration and treatment duration cannot be determined via FTIR analysis due to only minor changes detected in band absorptions between different alkali treatment conditions. However, in general, absorption band changes corresponding to the degradation of impurities such as lignin and hemicellulose after alkali treatment and decomposition of cellulose chains at higher alkali concentrations were in line with the literature studies.

3.3. Four-point bending test

Four-point bending test was conducted on composites with untreated and alkali-treated WWF to measure load-carrying capacity and displacement values. Obtained results from plain and WWF composites at the end of 28-day curing were shown in Fig. 6. Each data in Fig. 6 shows the representative curve of the samples that is the closest to the mean values among six four-point bending test results. Fig. 6. a demonstrated that as the alkali concentration increased up to 2.5 M, the load-carrying capacity of the composites with treated WWF increased gradually, while beyond 2.5 M alkali concentration, the load-carrying capacity of the composites with WWF decreased slightly for the constant treatment duration of 2 h. The increase in load-carrying capacity is associated with the enhancement of the WWF surface area and the strengthening of the WWF-matrix interface adhesion as a result of the removal of impurities such as lignin hemicellulose and wax oil at a given

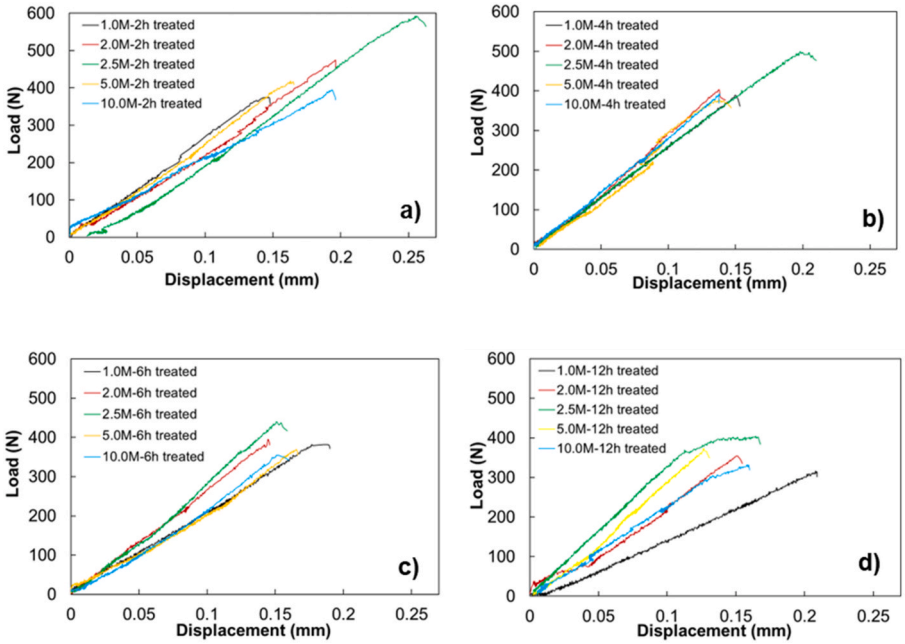

Fig. 6. Load-displacement curves of composites with treated WWF by 1.0 M, 2.0 M, 2.5 M, 5.0 M, and 10.0 M concentrations for a) 2 h, b) 4 h, c) 6 h, d) 12 h.

concentration of NaOH [79,80]. Moreover, the transition of fibers from bundle form to fibrillation due to the alkali-treatment process (Fig. 2) may increase the surface area of the fibers and thus an improvement in mechanical properties [80]. Fibrillation of the fibers yielded an increase in the aspect ratio due to reducing the diameter of the fibers and causing a rough surface, leading to the stronger matrix-fiber interface [81]. Rougher surface observed in SEM image (Fig. 4) and decreases in absorption bands in FTIR analysis (Fig. 5b) that correspond to the elimination of impurities could be evidence of increasing load-carrying capacity of 2.5M-2 h-WWF-composite. In that context, the flexural strength capacity/modulus of rupture (MOR) was recorded in 2.5M-2 h-WWF-composite as 7.9 MPa, while the flexural strength capacity of plain mortar and untreated fiber composite was found as 4.8 MPa and 5.2 MPa, respectively. Beyond 2.5 M alkali concentration, composite with treated WWF by 5.0M-2 h and 10.0M-2 h were presented 24% and 30% lower load-carrying capacity compared to 2.5M-2 h-WWF-composite, respectively ($p < 0.001$). This outcome can be related to the degradation of lignocelluloses at the higher NaOH concentrations, causing the WWFs to lose their stiffness, resulting in a decrease in mechanical properties [82,83]. Furthermore, the literature studies also presented that the high alkali concentration reduces the load-carrying capacity of the fibers by disrupting the long cellulose chain structure on the fiber surface, which has been supported by color changes on fiber images and SEM images (Figs. 1–3) [84]. As also seen in Fig. 6 b-*c*-d, similar trends obtained from alkali concentration changes proceeded on 2 h of treatment duration were also recorded for different treatment durations. However, it can be referred that changes in load-carrying capacities were found to be less pronounced as alkali treatment duration increases due to the deterioration of cellulosic structure by longer alkali treatment durations, even at low alkali concentrations.

Similar to the effect of NaOH concentration, it was observed that the treatment duration had a significant effect on the behaviour of the WWF-included composites. Based on the composite with treated WWF by 2.5 M alkali solution, which demonstrated the highest performance irrespective of treatment duration, a decrease in load-carrying capacity from 2 h to 4 h by 15.7% ($p < 0.001$), from 4 h to 6 h by 12.1% ($p < 0.001$), and from 6 h to 12 h by 8.1% ($p < 0.001$) was obtained. A similar down-trend was also recorded for other composites in the load-carrying capacities with increasing treatment duration. A similar trend has also been reported in some literature studies conducted on the treatment of natural fibers [82,83]. Such a trend can be associated with the closure of the voids and the decrease in the diameter of the WWFs in the first periods during alkali treatment, which increases strength. However, in the long-term treatment duration periods, the WWF becomes more unstable due to the degradation of the cells and thus leading to decreases in strength [85]. These findings can also be associated with SEM images (Figs. 2 and 3) which demonstrate the degradation of the WWFs structure with the increasing treatment duration.

Considering the displacement values of the composites under flexural load, unlike traditional synthetic fibers included composites, no deflection-hardening or deflection-softening behaviour was observed regardless of NaOH concentrations and treatment durations. Some literature studies on natural fiber composites exhibited a brittle behaviour by showing a sudden decrease after the ultimate load, as observed in this study [31,46,80]. This can be attributed that the low aspect ratio of WWFs compared to synthetic fiber, the percentages of the utilized fibers, the off-axis orientation of WWFs, and brittle nature of WWF, resulting in lower bridging behaviour and high toughness of matrix used in this study [41,86,87]. On the other hand, even though the composites did not exhibit any ductile behaviour as a result of the WWF inclusion, it caused an improvement in the rupture deformation as a consequence of the increasing load-carrying capacity. This increase in rupture deformation can also be associated with an increase in the aspect ratio of WWF [65]. This was more pronounced in the 2.5M-2 h-WWF-composite reaching a displacement value of 0.26 mm, while the displacement values of the other composites produced with different alkali molarity with 2 h duration were in the range of 0.15–0.20 mm.

To examine the effects of WWFs-inclusion and alkali treatment of WWFs, the representative curve of the samples that the closest to the mean values of the load-carrying capacities of plain-mortar (matrix), untreated-WWF-composite, and the highest performance composite (2.5M-2 h-WWF-composite) were shown in Fig. 7. Results revealed (Fig. 7) that the load-carrying capacity of composites was enhanced with the inclusion of 2.0% WWF irrespective of treatment condition compared to plain mortars/composites [27,46,88]. Besides, the load-carrying capacity of composite with treated WWF by 2.5M-2 h increased by approximately 53% compared to untreated WWF-incorporated composite ($p < 0.001$). This increment can be explained by the fact that alkali treatment would have altered the morphology of the WWF by removing impurities and roughening the WWF surface, improving WWF-matrix adhesion, and increasing the load-carrying capacity of the composites [89].

3.4. Compressive strength

Average compressive strength results of plain mortar, the composite with untreated WWF, and with treated WWF by various alkali concentrations and durations were shown in Fig. 8. It was seen that plain mortar exhibited a 43.5 MPa compressive strength result, while the composite with 2% untreated WWF addition showed 38.1 MPa compressive strength after 28 days of curing. A decrease in compressive strength of composite of approximately 12.4% ($p < 0.001$) with the untreated WWF inclusion can be related to the fact that impurities such as lignin, hemicellulose, and wax oil on the surface of WWF caused poor adhesion between WWF and cementitious matrix. It was expected since the incorporation of untreated WWF, which contains a high amount of impurities and has poor adhesion ability, caused more porous and heterogenous microstructure, thus yielding a lower compressive strength [90–92]. Similar to the results under flexural behaviour, it was observed that the composites with treated WWF addition caused an increase in compressive strength up to a specific NaOH concentration and treatment duration. In this context, the compressive strength of the composite with treated WWF by 2.5M-2 h was found to be 6.4% higher than plain mortar ($p < 0.001$) and 21.5% higher than composite with untreated WWF ($p < 0.001$) by reaching the highest compressive strength in the series with 46.3 MPa.

In addition, a slight increase was observed in compressive strength of the composites with treated WWF by 2.0M-2 h ($p = 0.022$), 2.0M-4 h ($p = 0.001$), 2.0M-6 h ($p = 0.038$), 2.5M-4 h ($p < 0.001$), 2.5M-6 h ($p = 0.005$), 5.0M-2 h ($p = 0.008$), 5.0M-4 h ($p = 0.026$) and 10.0M-2 h ($p = 0.062$) compared to plain mortar. The increases in compressive strength are associated with the fact that alkali treatment improves the adhesion

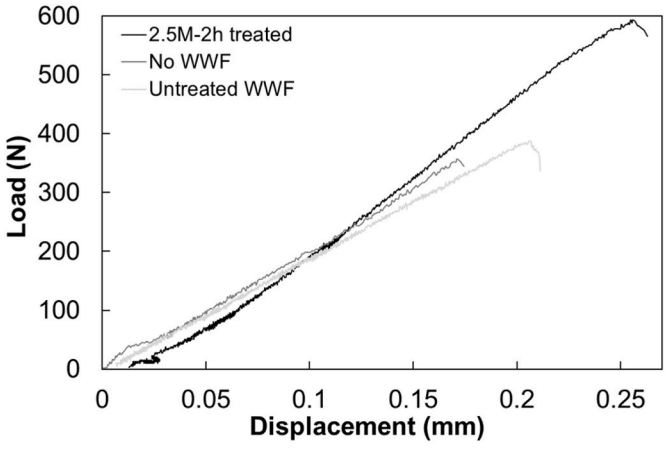

Fig. 7. The load-displacement curves of matrix (composite without WWF), and with untreated and treated WWF.

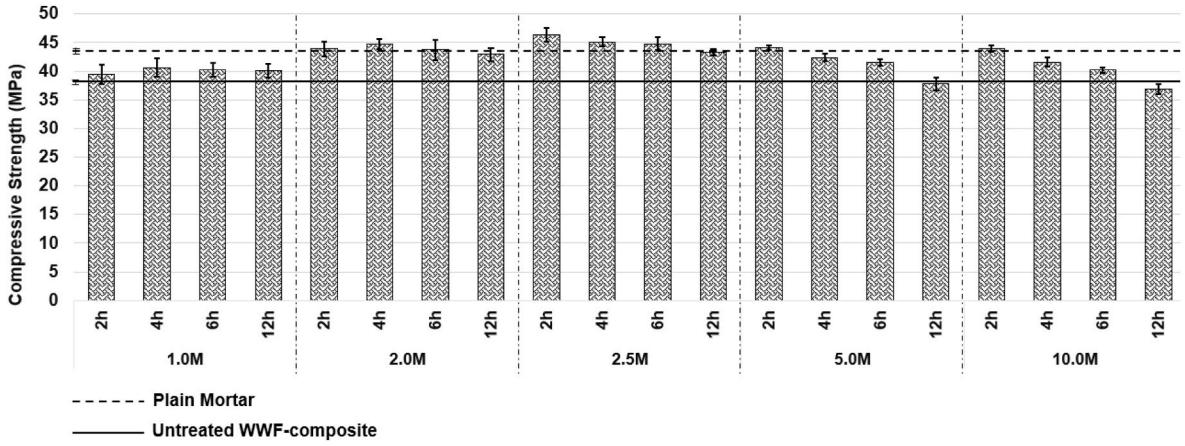

Fig. 8. Compressive strength results from plain mortar, untreated WWF, and treated WWF composites.

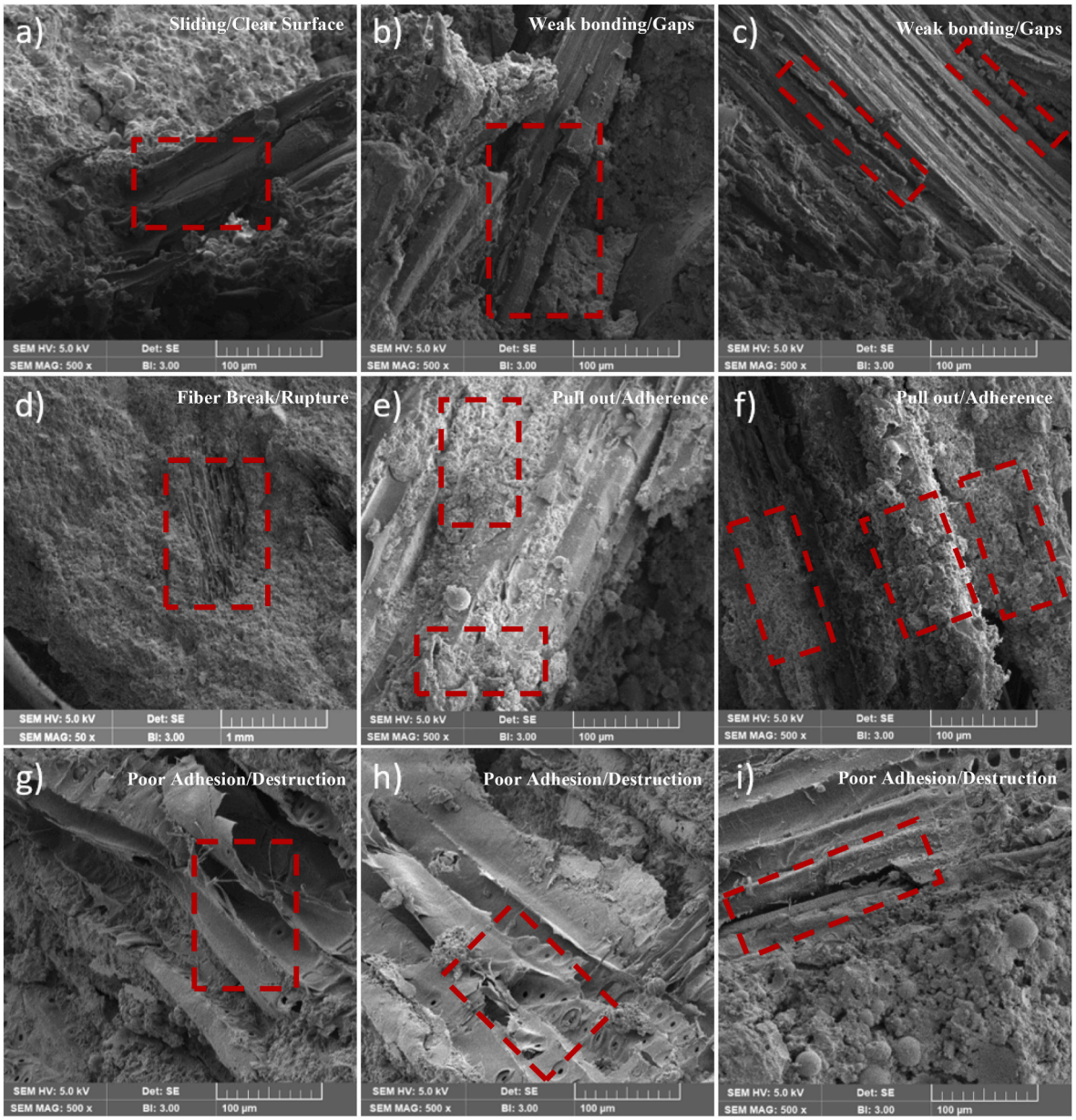

Fig. 9. SEM observations of a-c) untreated WWF, d-f) treated WWF by 2.5M-2 h, and g-i) treated WWF by 10.0M-12 h-included composites (Red dashed lines indicate the corresponding failures). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

capacity of WWF by eliminating impurities on the surface of WWF at optimum alkali treatment concentration and duration, thus causing a strengthened matrix-WWF interface bond [47,80]. Strong interlocking between WWF and matrix due to the formation of additional sites (Fig. 4) with enhanced interface properties leads to improved mechanical performance [80]. On the other hand, decreases were observed in the compressive strength of the composites compared to plain mortar at the rest of the alkali concentrations and durations. It can be associated with inadequate development of the WWF-matrix interface, as a relatively low alkaline concentration is insufficient to remove the impurities on the WWF surface. In addition, at high alkali concentrations, the deterioration and fragmentation of the WWFs, and a weak WWF-matrix interface may cause a weak microstructure with the formation of a more heterogeneous structure [31,80,84,93]. In this context, insufficient or high alkali concentrations and longer treatment duration resulted in a slight decrease in the compressive strength of composites.

3.5. SEM observation of WWF reinforced matrix

Interfacial characteristics of WWF with cementitious matrix were investigated through corresponding SEM images shown in Fig. 9, which demonstrates the failure mode of WWFs and fiber/matrix interaction. To visualize representative WWF interaction in matrix considering treatment condition, composites with untreated WWF, treated WWF by 2.5M-2 h, and treated WWF by 10M-12 h were scanned via SEM analysis. SEM images showed that mechanical failures of WWF incorporated matrices were mostly due to fiber pull-out, fiber breakage, and debonding/gap failure. From Fig. 9. a-b-c, it can be seen that surface of the untreated WWF was smooth and free from the attached matrix/mortar, which was an indication of poor interfacial bonding [41,94–97]. WWF is well known to be hydrophilic while the matrix behaves hydrophobic. This adhesive character difference results in detachment. Non-cellulose impurities existing on the WWF surface, which could inhibit the hydration reaction also contribute to this incompatibility [41]. Fig. 9. a illustrates that the untreated WWF was sliding along with the surface until fiber pull-out, without showing strong mechanical locking between the fiber and matrix as there was no damage/defects or attached matrix observed. In Fig. 9b-c, the gaps between the fiber and the matrix were observed due to the weak bonding of fibers and matrix [98]. SEM images of matrix containing treated WWF by 2.5M-2 h demonstrated that fiber surfaces were rougher than untreated ones, and some matrix residuals are observed on the surface of treated WWFs (Fig. 9. d-e-f). These observations indicated adequate bonding/adhesion between matrix and fiber [80,86,95,98]. High delamination and removal of impurities on the surface of treated WWF due to chemical modification by NaOH treatment may lead to better adhesion between matrix and fiber [96,99]. From Fig. 9. d, breakage/rupture of fiber was observed instead of fiber sliding because of the enhanced interfacial characteristics. Excellent bonding may result in WWF breakage with the matrix cracking, which means adhesion between the matrix and fiber was not disappeared, and the failure was dominated by the matrix itself [80,100]. However, this breakage may be attributed to the off-axis orientation of fibers in the matrix that prevents the fiber pull-out during load-carrying [86]. Pull-out of fiber with attached matrix on its surface was observed from Fig. 9e-f, which verifies good interfacial adhesion between matrix and fibers [41]. To represent matrix interaction with treated WWF in severe conditions (high alkalinity and long treatment duration), SEM images of a matrix with treated WWF by 10M-12 h were investigated. From Fig. 9. g-h-i, gaps/debonding, unfilled holes formed via removal of impurities (Figs. 3 and 4), destructed structure of fibers, and less amount of attached matrix on the fiber surface were observed; indicating poor adhesion between matrix and fiber. Poor interaction characteristics of treated WWF with the severe condition could be due to excess removal of covering materials, including cellulose-based substance, excessive delignification and destruction of the crystalline structure of cellulose, and increasing brittleness of WWF due to high alkalinity [80,95,96,101].

3.6. Physical properties of WWFs and WWFs-included composites

The physical properties in terms of density and water absorption of untreated and 2.5M-2 h treated WWF, plain mortar, untreated WWF-included composite, and 2.5M-2 h treated WWF-included composite were demonstrated in Table 2.

After alkali treatment of WWF by 2.5M-2 h, water absorption decreased from 155.2% to 145.7% compared to untreated WWF. This can be associated with the fact that hemicellulose and lignin, which have great influences on the water absorption capacity of WWF, were removed from the surface after the alkali treatment, thus decrement in water absorption was recorded [102].

Obtained results from the cementitious system showed that the inclusion of WWF resulted in a decrement in the density irrespective of the treatment process. However, no significant difference in the density was observed between untreated WWF composite and 2.5M-2 h treated WWF composite. On the other hand, water absorption of untreated WWF-included composite and 2.5M-2 h treated WWF-included composite increased by 8.9% and 5.3% compared to plain mortar, respectively. This could be attributed to the presence of WWF which has a high-water absorption capacity. Additionally, as can be inferred from Table 2, the decrease in the water absorption in 2.5M-2 h treated WWF-included composite compared to untreated WWF-included composite can be associated with removing of hemicellulose and lignin from the surface after the alkali treatment [102].

4. Conclusion

This paper investigated the effect of various alkali-treatment conditions on the microstructural properties of waste wood fiber (WWF) obtained from construction and demolition waste and cementitious matrix-WWF interfacial bonding characteristics by performing SEM, FTIR, compressive, and four-point bending tests. In this context, alkali concentrations of 1.0 M, 2.0 M, 2.5 M, 5.0 M, 10.0 M, and treatment durations of 2 h, 4 h, 6 h, and 12 h were studied comprehensively. The following conclusions have been drawn from the experimental studies performed within the scope of the current research.

- According to the SEM images of alkali-treated WWFs, it was found that alkali treatment is capable of enhancing WWF regarding surface morphology, and alkali concentration and treatment duration have a significant impact on the treatment of WWF. After the alkali treatment, impurities (*i.e.,* painting, lignin, and hemicellulose) located on the WWF surface were successfully removed. However, higher alkalinity (beyond 5.0 M) and longer treatment duration (after 6 h) yielded a high degree of fragmentation of WWF and destruction of the crystalline structure of cellulose. According to the band changes observed in the FTIR analysis after alkali treatment, it was observed

Table 2
Physical properties of WWFs, plain mortar, and WWFs-included composites.

	Untreated WWF	2.5M-2 h treated WWF	
Water absorption (%)	155.2	145.7	
	Plain Mortar	**Untreated WWF Composites**	**2.5M-2h treated WWF Composite**
Density (g/cm^3)	2.341	2.285	2.261
Water absorption (%)	7.813	8.505	8.224

that medium alkalinity (*i.e.,* 2.0 M and 2.5 M) was found to be sufficient to remove lignin and hemicellulose structures of WWF.

- Under flexural loads, composite with WWF treated by 2.5M-2 h reached flexural strength of 7.9 MPa by showing higher performance than untreated WWF composite (5.2 MPa) and plain mortar (4.8 MPa). The increase in load-carrying capacity and a slight increase in rupture deformation (in the comparison of 2.5M-2 h WWF composite, untreated WWF composite [$p = 0.048$], and plain mortar [$p = 0.006$]) are associated with the enhancement of the WWF surface characteristics, increase in aspect ratio, and strengthening of the fiber-matrix interface adhesion as a result of the removal of impurities such as lignin hemicellulose, and other inorganic substances at a given treatment condition.
- According to the compressive strength results, composite with WWF treated by 2.5M-2 h was found to be 6.4% higher than plain mortar and 21.5% higher than composite with untreated WWF by reaching the highest compressive strength in the series with 46.3 MPa. The increases in compressive strength are associated with the fact that alkali treatment improves the adhesion capacity of WWF by eliminating impurities on the surface of WWF at optimum alkali treatment concentration and duration, thus causing a strengthened matrix-WWF interface bond. At high alkali concentrations (beyond 5.0 M), the deterioration and fragmentation of the WWFs, and the weak WWF-matrix interface, caused a weak microstructure with the formation of a more heterogeneous structure.
- SEM images acquired from the matrix-WWF interface zone showed that mechanical failures of untreated WWF incorporated matrices were mostly due to fiber pull-out, fiber breakage, and debonding/gap failure. The surface of the untreated WWF is smooth and free from the attached matrix/mortar, indicating poor interfacial bonding. However, SEM images of matrix containing treated WWF by 2.5M-2 h demonstrated that WWF surfaces are rougher than untreated ones. Some matrix residuals were observed on the surface of treated WWF, indicating proper adhesion between the matrix and fiber. At higher concentrations, unfilled holes formed via removal of impurities, destructed structure of WWF, and less amount of attached matrix on the WWF surface were observed, indicating poor adhesion between matrix and WWF.

The primary purpose of the current study was to examine parameters regarding the alkali-treatment process for WWF. By obtaining optimum process parameters, valorization of waste wood from construction and demolition waste in the building materials can become widespread in different areas with high compatibility in cementitious composite. For further studies, the effects of enhanced WWF via alkali treatment on durability, heat insulation, and noise reduction will be attractive topics for new research.

Author statement

Merve Sonmez Tugluca Visualization, Investigation, Writing-Original Draft.

Emine Ozdogru Visualization, Investigation, Writing-Original Draft.

Huseyin Ilcan Investigation, Writing-Original Draft.

Emircan Ozcelikci Investigation, Writing-Original Draft.

Huseyin Ulugol Writing-Original Draft, Writing-Review & Editing.

Mustafa Sahmaran Conceptualization, Methodology, Resources, Writing-Review & Editing, Supervision, Project administration, Funding acquisition.

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.

Data availability

No data was used for the research described in the article.

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