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Enhancement of water absorption properties of date palm fibers-based composites via alkaline treatment

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ABSTRACT

This study examined the ability of a biodegradable composite material to absorb water. The composites were created by combining a matrix of polylactic acid (PLA) with date palm wood fibers (DPWF) for reinforcement. Before incorporating the fibers into the polymer, they underwent chemical treatment using alkali solutions of NaOH and KOH. This treatment aimed to modify the surface properties of the natural fibers and enhance their water absorption capabilities. Different composites were fabricated, including PLA combined with untreated fibers (PLAUF), and PLA with treated fibers using both NaOH and KOH (PLAF-NaOH and PLAF-KOH), with varying weight percentages of fibers ranging from 0 to 40%. To evaluate the water absorption capability of the composites, water retention tests were conducted at room temperature (cold) and 50 °C (hot) for 24, 48 h and until the absorption reached equilibrium. The results demonstrated a significant enhancement in water retention, particularly in the case of the NaOH-treated samples, which exhibited approximately a tenfold increase in water retention compared to untreated samples. For example, the 20 wt% NaOH treated filler had a hot-water retention of 20%, compared to 2% for the untreated filler. Additionally, a higher percentage of filler resulted in a higher rate of water absorption. The 40 wt% NaOH treated sample with 20% water retention, absorbed 10 times more than the 10 wt% NaOH treated filler, with only 2% water retention. The developed composites have potential applications in natural gas dehydration processes, offering an alternative to the energy-intensive and environmentally harmful glycol-based dehydration methods currently in use.

1. Introduction

Gas plays a crucial and significant role in various industries as an essential fossil fuel. It serves as a high-quality fuel, an energy source for heating, and a chemical feedstock. Natural gas dehydration is a vital process required to prepare natural gas for different applications. Currently, glycol-based materials like triethylene glycol (TEG) are commonly used as dehydration agents in gas dehydration plants due to their efficient absorbent properties (Ghanbari and Niu, 2018). However, the regeneration process of these glycol solutions consumes a large amount of energy, posing potential harm to the environment. Additionally, the use of glycol solutions in the dehydration process leads to the emission of benzene, toluene, ethylbenzene, and xylene isomers (Bahraminia et al., 2021).

Various studies have explored the water absorption characteristics of biomaterials. For example, the water absorption capability of Kenaf core was investigated for its potential as an animal bedding material, and it exhibited promising results comparable to commercial materials like straw and wood shavings (Lips et al., 2009). Hydrogels, which are three-dimensional cross-linked networks

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made of synthetic or natural materials, have been extensively studied as absorbents. Natural hydrogels can be derived from protein-based polymers such as collagen, elastin, gelatin, fibrin, and silk fibroin, as well as polysaccharide-based materials like chitosan and alginate (Catoira et al., 2019). Hydrogels exhibit exceptional absorption capacities, ranging from 10 to 1000 times their dry weight (Ahmed, 2015). Cellulose, the most abundant organic material, possesses a high affinity for water due to its numerous hydroxyl groups (Luo et al., 2018). Researchers have investigated cellulose-based hydrogels and their properties, including water absorption, degree of polymerization, and water absorption kinetics (Santos et al., 2021; Ghanbari and Niu, 2019). To address the issues with glycol solutions, alternative materials have been explored. Adsorbents such as zeolites, metal-organic frameworks (MOFs), silicas, and silica-alumina are under investigation and being utilized for natural gas dehydration (Santos et al., 2021). Additionally, biomaterials like flax shives, which primarily consist of cellulose and are a byproduct of the flax industry, have been examined as biosorbents as an alternative to glycol in natural gas dehydration processes (Ghanbari and Niu, 2018). In another study, oat hull was examined as biosorbent for natural gas dehydration, and it achieved water absorption capacity that surpasses commercial adsorbents. Oat hull achieved 0.63 (g/g) water absorption capacity, compared with 0.21–0.26, 0.25–0.33, 0.35–0.5, for molecular sieves, alumina, and silica gel, respectively (Ghanbari and Niu, 2019).

The technology for wood-plastic composites is continually advancing, with refinements in manufacturing techniques (such as extrusion, injection, and compression molding), progress in material development involving innovative polymer matrices, treatments, and additives, for use in construction, automotive, and furniture applications. Additionally, there are enhancements in terms of durability against weather, fire, and biological threats, along with the establishment of product standards for use in building construction (Li et al., 2007a). For example, cellulose-epoxy composites were prepared, and their water absorption and mechanical properties have been studied (Alamri and Low, 2012). Moreover, epoxy-bamboo fibers composites were prepared, and the effect of the eco-friendly Na_2CO_3 treatment was studied. It showed that Na_2CO_3 treatment enhanced the mechanical properties of the composite and decreased the water absorption due to better compatibility between the matrix and the filler (Abdul Karim et al., 2023).

Poly (lactic acid) (PLA) is a biopolymer with significant promise in diverse industries thanks to its renewable sourcing, lack of toxicity, and capacity for biodegradation. Additionally, PLA boasts impressive mechanical characteristics, a high production rate, and a relatively hydrophobic nature (Al Abdallah et al., 2022). Meanwhile, date palm wood fibers are abundant natural materials that readily decompose. It is estimated that there are approximately 120 million date palm trees in countries that are major producers of dates (Hanieh et al., 2020). Furthermore, each palm tree generates approximately 20 kg of wood waste annually, and the lifespan of a palm tree ranges from 40 to 50 years (Faiad et al., 2022). Wood fibers possess high hydrophilicity and can absorb significant quantities of water due to their cellulose content. Nonetheless, composites consisting of PLA reinforced with wood fibers are widely studied for different applications, including heat insulation, including buildings and home furnishing, medical material, oil and water separation, and packaging materials (Li et al., 2023). In addition to incorporating wood fibers into PLA matrix, chemical treatments are widely applied to enhance the compatibility between the filler and the polymeric matrix. One of the prevailing chemical methods employed to treat natural fillers is the alkaline treatment, often referred to as mercerization. This treatment is commonly applied to natural fibers before they are added to polymer matrices for reinforcement purposes (Asumani et al., 2012). The alkaline treatment enhances the cellulose content and its accessibility on the surface by partially eliminating lignin, waxes, and oils that coat the outer surface of the fiber cell wall (Li et al., 2007a).

The primary goal of this study is to assess the viability of a biodegradable polymer composite composed of polylactic acid (PLA) and date palm wood fibers (DPWF) as an environmentally friendly water-absorbent material. PLA is a durable biopolymer known for its impressive mechanical properties (Barkhad et al., 2020). However, it has a hydrophobic nature, which can negatively affect the water absorption. Therefore, DPWF is incorporated into the PLA matrix to enhance the water uptake, and to utilize and manage the agricultural wood waste that is generated by date palm trees. Moreover, alkaline treatment is applied with NaOH and KOH to improve the water absorption by removing impurities such as lignin, waxes, and oils. PLA-DPWF composites have not explored so far for the application of biosorbent material in natural gas dehydration.

2. Materials & methods

2.1. Materials

Pellets of PLA were purchased from Zhejiang Zhongfu Industrial Limited in Zhejiang, China, bearing the following specifications: The L-lactide to D-lactide ratio ranged from 24:1 to 32:1, the pellets had a diameter of 3.5 mm, and a molecular weight of 2.41 × 105 g/mol. These pellets were labeled as (4032D). PLA has a specific gravity of 1.24, exhibits semi-crystalline properties, and has a melting point that falls within the range of 155–170 °C. Date palm wood was gathered from the UAE University farm in Al Foah, comprising wood waste sourced from various parts of the palm tree, including leaves, branches, and the base. The NaOH and KOH used in this study were in pellet form and were provided by Sigma Aldrich (St. Louis, MO, USA). Sodium Hydroxide was obtained from Sigma Aldrich, with CAS number 1310-73-2, originating from the Czech Republic. Potassium Hydroxide was purchased from Sigma Aldrich, with CAS Number 1310-58-3, a product of the Czech Republic.

2.2. Methods

The date palm wood fibers were initially ground to reduce their size to 212 μm. Simultaneously, alkali solutions were prepared by dissolving 2 wt% of NaOH and KOH pellets in water. The fibers were then immersed in the alkali solutions at a ratio of 10 g of filler per 100 mL of solution (10% w/v) for a duration of 2 h at room temperature. Subsequently, the filler particles were filtered and dried at a temperature of 95 °C for 2 h. The mixing of the filler and polymer was achieved through melt extrusion using the HAAKE Mini Lab II by Thermo Scientific. The extrusion process was carried out under the conditions of a temperature of 190 °C, a torque of 140

N.m., and a retention time of 3 min inside the extruder. The resulting extruded material was then placed into cylindrical molds with a height of 25.7 mm and a diameter of 12.8 mm. To shape and smoothen the samples correctly, the cylindrical molds were placed in a hot press machine (Carver's AUTOFOUR/3015) in three consecutive steps. In the first stage, a pressure of 0.50 tons was applied for 5 min and 20 s at a temperature of 180 ˚C. The pressure was increased to 0.52 tons in the second stage, which lasted for 4 min, while the temperature was raised to 185 ˚C. Finally, in the third stage, the pressure was increased to 3 tons, and the temperature was lowered to 100 ˚C for a duration of 3 min and 30 s. Raw materials and the prepared composites are presented in Fig. 1. The composites are characterized in this work using Fourier Transform Infrared spectrometry (FTIR), Scanning electron microscope (SEM), X Ray Diffraction (XRD), and water retention in hot (50 ˚C) and cold (room temperature) water, over 48 h and over long term. In addition, other properties such as density and mechanical properties for the same composites were determined and measured in our previous work ("IR Spectrum Table).

3. Results and discussion

3.1. Fourier Transform Infrared spectrometry (FTIR)

The FTIR spectrometry technique was employed to examine the functional groups present in untreated date palm wood fibers, as well as fibers treated with NaOH and KOH. Additionally, the FTIR analysis was performed on composites containing untreated fibers (PLAUF), NaOH-treated fibers (PLAF-NaOH), and KOH-treated fibers (PLAF-KOH) with a filler particle content of 20 wt%. The FTIR results of untreated date palm wood fibers, as shown in Fig. 2a, revealed peaks in the range of 1000–1250 cm-1, which corresponded to the ester group (C–O). Peaks observed at 1750 cm-1 and 3000 cm-1 represented C = O and C–H bonding, respectively. Furthermore, the presence of O–H groups was observed at approximately 3300 cm-1 ("IR Spectrum Table). The FTIR analysis of treated fibers indi-

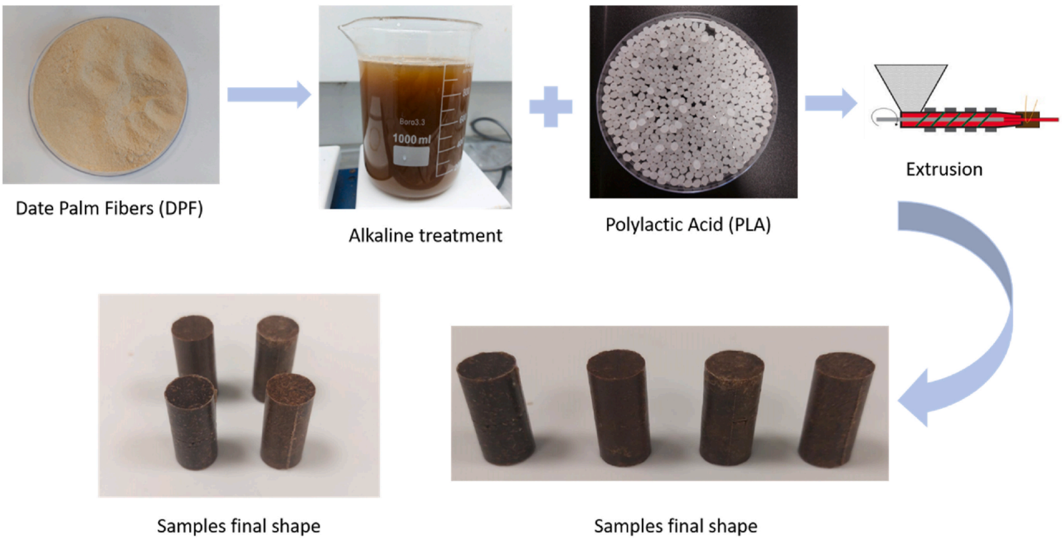

Fig. 1. Raw materials, production methods, and final composites.

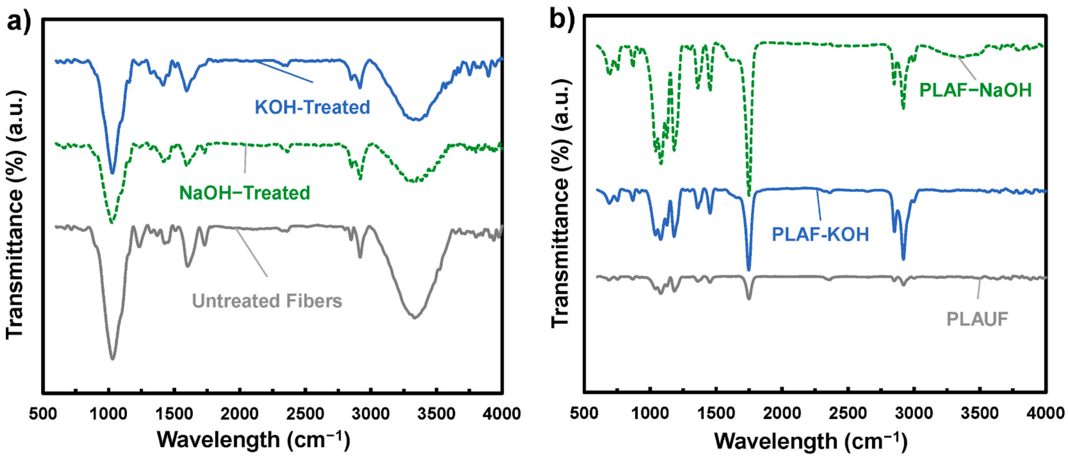

Fig. 2. FTIR results for Untreated, NaOH, and KOH treated wood fibers (a), PLAUF, PLAF-KOH, and PLAF-NaOH composites (b).

cated changes resulting from the removal of wax, lignin, and hemicellulose through alkaline treatment. In both NaOH and KOH treated fibers, there was a reduction in the peaks around 1250 cm-1 and 1750 cm-1, which can be attributed to the removal of hemicellulose (Rout et al., 2016). These peaks correspond to the stretching of C–O and C = O in hemicellulose, respectively. Moreover, a reduction in the peak around 1030 cm-1 was observed, indicating the removal of lignin content (Rout et al., 2016). Regarding the composites, Fig. 2b demonstrates a significant increase in the carboxylic acid group (O–H) at a wavelength of approximately 3000 cm-1, resulting from the high moisture absorption by the composites. The removal of lignin and wax from the filler particles enhanced the moisture intake by cellulose in the prepared composites, leading to increased water absorption, as will be further elucidated in the water retention results. The sharpening of the carboxylic acid group around 1750 cm-1 (C = O) in NaOH and KOH-treated composites mostly occurred due to the reactivity between cellulose and polylactic acid. In the study of Altun et al. on the effect of alkaline treatment of PLA and pine wood composites, FTIR results specified similar peaks to the obtained peaks in our work, with peaks at 1750, 3000, and 1043 cm-1, corresponding to carbonyl stretching, C–H stretching, and C–O stretching, respectively (Altun et al., 2013). In a review work on cellulose extraction from different biomass, it is highlighted that FTIR results for biomass showed peaks at 3297 cm^{-1} that indicated O–H stretching in cellulose and hemi-cellulose, 2927 cm^{-1} which represented C ≡ C stretching in wax, 1607 cm^{-1} denoted C–O stretching in lignin, and 1022 cm^{-1} revealed CO stretching in lignin (Jaiswal et al., 2022). This can explain why the peaks at 1022 and 1607 are sharper in the untreated fibers, where lignin is present in high proportion.

3.2. SEM

To investigate the impact of alkaline treatment, particularly NaOH, on the filler's morphology and the removal of particles from the fiber surface, SEM (Scanning Electron Microscope) tests were conducted. Both pre-treatment and post-treatment wood fiber samples were examined using a NeoScope Scanning Electron Microscope provided by JOEL. Prior to imaging, the samples were coated with a layer of gold. The images were captured at an accelerating voltage of 10 kV and a scale of 200 μm. In Fig. 3a, the SEM image clearly depicts the presence of impurities on the surface of the fibers prior to treatment. Conversely, Fig. 3b displays a smooth surface resulting from the NaOH treatment, demonstrating the removal of wax and impurities from the surface layer. In the work of Jaiswal et al., similar behaviors were observed for works on treated fibers, where wax and other impurities were removed (Jaiswal et al., 2022).

3.3. X Ray Diffraction

The impact of the treatment on the crystallographic structure was examined using X-Ray Diffraction (XRD), comparing untreated fibers with those treated with NaOH and KOH. The experiment utilized a Malvern Panalytical X-ray diffractometer. The results of the XRD analysis are depicted in Fig. 4. In both treated and untreated fibers, there are three clear peaks observed between 20 and 30°, as illustrated in the figure. These peaks correspond to the cellulose component of the wood, as stated in previous research (Wang et al., 2016). The alkaline treatment, based on previous studies, removes lignin, as well as wax and other contaminants from the fiber surface. This leads to a higher proportion of cellulose in the fibers (Li et al., 2007b). Additionally, the alkaline treatment significantly reduces the amount of hemicellulose, a major component of biomass (Rout et al., 2016). Consequently, the peaks in the NaOH and KOH treated sample exhibit greater sharpness compared to the untreated fibers (UF), indicating increased crystallinity and a higher proportion of cellulose due to the removal of lignin and hemicellulose. Similarly, in the work of Haddis et al., where the treatment of poplar wood by steam explosion to obtain cellulose nanocrystals (CNC) were applied, the treated biomass yielded higher crystallinity than raw polar wood (Haddis et al., 2024).

3.4. Water retention

To examine the absorption capability of three types of composites, namely PLAUF, PLAF-NaOH, and PLAF-KOH, a water retention test was conducted following ASTM-D570. Specimens with various filler loadings were used for the test. Cylindrical samples were submerged in water baths at room temperature and at 50 °C. The water absorption was determined using Equation (1), where W_I represents the initial weight of the samples before immersion and W_f represents the final weight after 24 or 48 h. The average results of 3–5 samples for each composite are presented in Fig. 4.

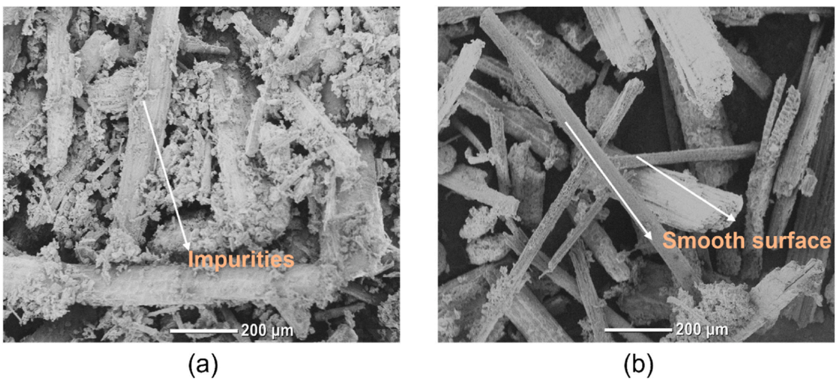

Fig. 3. SEM of Untreated wood fibers (a), and NaOH-treated wood fibers (b).

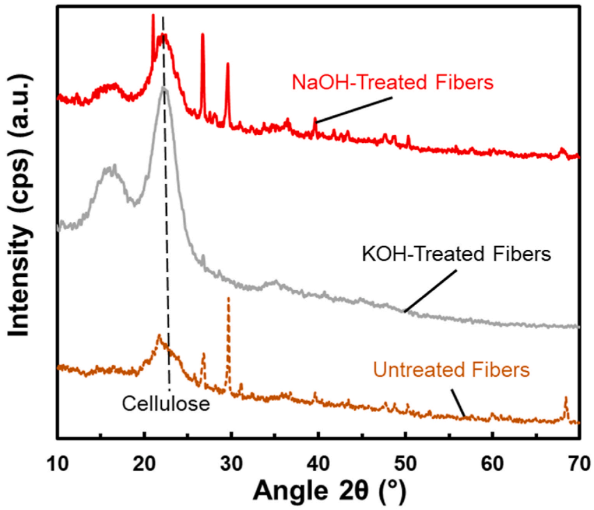

Fig. 4. XRD for untreated, and NaOH-treated palm trees wood fibers.

$$\text{Water Retention } (\%) = \frac{W_f - W_I}{W_I} \text{ x } 100 \tag{1}$$

The filler in the composites possesses a hydrophilic nature, resulting in a direct relationship between water retention and the weight percentage of the filler in the composites. Both in treated and untreated composites, for both cold and hot water retention, the trend observed is similar, with absorption increasing as the fiber weight percentage rises. This behavior made the 40 wt% samples absorb the most water, while the 10 wt% absorbed the least amount. For cold water retention, the PLAUF composites exhibited the lowest retention levels, ranging from 0.23% to 2% for filler weight percentages of 10% and 40%, respectively, as shown in Fig. 5a and b. After 48 h, the PLAF-KOH samples absorbed water ranging from 0.5% to 4.5%, for filler weight percentages of 10% and 40% respectively. Comparing the water intake of PLAF-NaOH composites to that of PLAUF and PLAF-KOH composites reveals significantly higher absorption. After 48 h, the cold-water absorption ranged from 2.35% to 22.2% for filler weight percentages of 10% and 40%, respectively. This indicates that the water retention for PLA-NaOH was ten times higher than untreated PLAF, while PLAF-KOH was around two times higher than untreated PLAF. In contrast, hot water exhibited even higher absorption in all samples. After 48 h, PLAUF had an intake ranging from 1.5% to 5% for filler weight percentages of 10% and 40%, respectively. Fig. 5d illustrates that PLAF-KOH exhibited higher absorption, ranging from 2.5% to 12.5% for the 10% and 40% filler weight percentages, respectively.

Furthermore, PLAF-NaOH composites displayed more pronounced hot-water retention, with water absorption ranging from approximately 14% for the 10% fiber to 42% for the 40% fiber. It is evident that the NaOH treatment of the filler resulted in the most significant removal of lignin, as well as impurities and wax from the fiber walls, resulting in greater exposure of the cellulose content. As a result, PLAF-NaOH composites exhibited the highest water absorption compared to all other composites. The 40 wt% samples demonstrated the highest water absorption due to the hydrophobic nature of the filler, making them the optimal choice within the PLAF-NaOH composite range. The performance of these developed materials is comparable to traditional water absorbers used in the gas industry, such as molecular sieves and silica gels, which can absorb up to 22% and 32% of moisture relative to their own weight, respectively (Chen, 2017). Hafidz et al. (Binti Mohd Hafidz et al., 2021) conducted a study on the effect of NaOH treatment on water retention in natural fiber-reinforced unsaturated polyester composites. The composites were prepared using polyester/palm oil fibers and polyester/kenaf fibers, with varying filler percentages. The results indicated that the polyester composite filled with 60% palm oil fibers and treated with 5% NaOH achieved the highest water absorption, reaching 40% of the initial dry weight. Generally, the treated samples exhibited higher water retention compared to the untreated ones, and samples treated with 5% NaOH absorbed more water than those treated with 1% NaOH. Furthermore, a higher fiber content resulted in increased water retention. Moreover, another study examined the effect of lignin removal on water retention of hemp fibers, in which the alkaline treatment using 17.5% NaOH led to an increase in the water absorption (Pejic et al., 2008). In addition, in a study was done by Benyettou et al. on modelling and optimization of the absorption rate of date palm fiber reinforced composite using response surface methodology (Benyettou et al., 2023). It was found out that, as expected, composites containing a higher concentration of fibers tend to absorb more water than those with a lower fiber content. The study also noted that bio-composites with fibers aligned in a unidirectional stacking pattern exhibit greater water absorption compared to those with fibers arranged in crisscross layers. This can be attributed to the presence of polar hydroxyl groups within the cellulose fibers, hemicellulose, and lignin structures, which have the capability to form hydrogen bonds with water. Other factors affecting the water absorption, according to the study, are time, fiber content, and types of water.

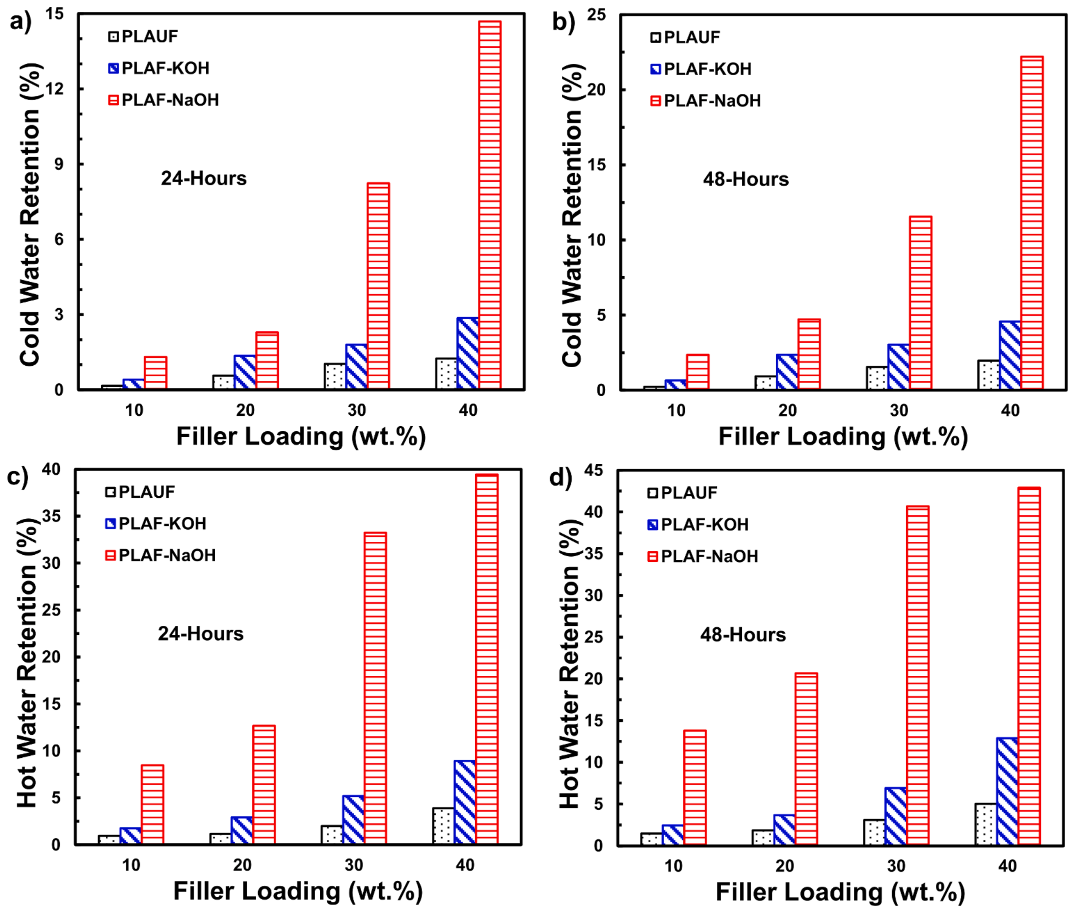

Fig. 5. Cold Water Retention after 24 h (a) 48 h (b), and hot water retention after 24 h (c) and 48 h (d).

3.5. Long term water retention and sample regeneration

Long term water retention was also implemented on the NaOH samples, to test the maximum capacity for water uptake of the samples, and the results are presented in Fig. 6. It was observed that the maximum water uptake for the 10% treated samples stood at about 7% after 192 h (8 days), with no noticeable increase from the reading after 168 h. Therefore, it can be concluded that the equilibrium was reached after 168 h (7 days). While for the 20% treated samples, it reached saturation at around 17% during the same period. On the other hand, after a period of 168 h (7 days), 30 and 40% samples were torn apart due to large water uptake, at around 20% and 22%, respectively.

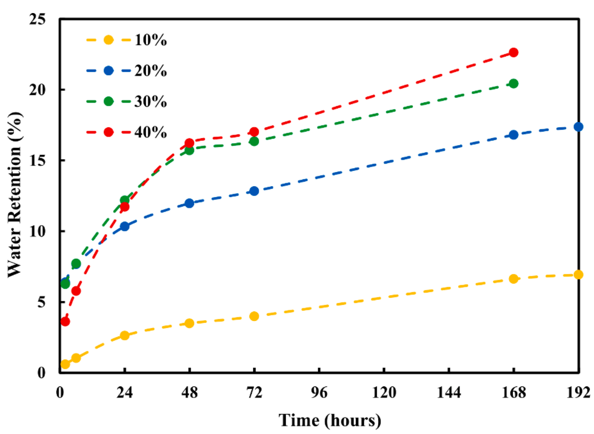

Fig. 6. Long-term cold-water retention for NaOH treated samples.

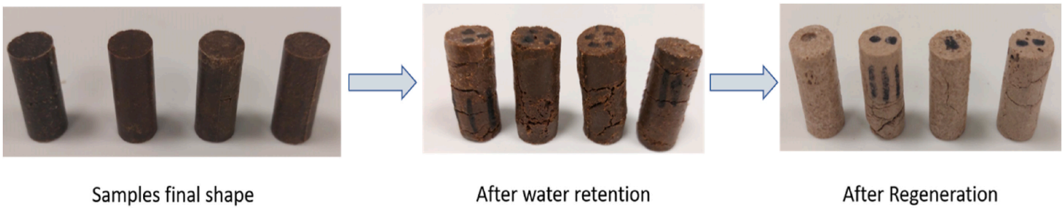

Fig. 7. Final composites before and after water retention, and after regeneration.

Afterwards, A drying test was applied to selected samples, to test the time required for the samples to gain their original weight after the removal of the water they retained. Samples from the 4 different weight percentages were placed in an oven at temperature of 60 °C. Readings were taken after 30 min and 1 h, and it showed a total water evaporation after an hour, where the samples regained their initial weight. Fig. 7 shows the samples before water retention, after water retention, and after regeneration (drying).

4. Conclusion

This study focused on investigating the impact of alkaline treatment on enhancing the water absorption properties of biodegradable composites composed of polylactic acid (PLA) and date palm wood fibers (DPWF) as a filler. The treatment involved using two different alkaline solutions, namely NaOH and KOH, on the DPWFs before combining them with the polymeric matrix. The objective was to develop composite materials with a high capacity for water absorption. The alkaline treatment significantly improved the water retention of the composites. The water absorption after 24 and 48 h, both at room temperature and at 50 °C, was notably higher compared to composites using untreated fibers, regardless of the filler percentages. For instance, in cold water retention which was carried out at room temperature, the water absorption for 40 wt% sample after 48 h increased from 2% to 20%, for untreated composite and NaOH treated composite, respectively. Moreover, NaOH increased the hot water retention at 50 °C for 40 wt% sample, from 5% to 50% after 48 h. Alkaline treatment, also known as Mercerization, is a widely recognized process for removing lignin and wax from natural fibers. Consequently, the enhanced water absorption capability was expected due to increased exposure of cellulose to water. The achieved results were on par with the absorption capacity of traditional materials, indicating the potential of the developed material as a water absorbent in various applications, such as natural gas dehydration. The developed material can be further studied to test their ability for absorption applications, including water retention. Tests such as BET surface area, porosity, and chemical resistance against specific compounds (based on the application) can be performed to further assess the material. Moreover, cost analysis can be done to determine the economic feasibility of the composites for the targeted application. Cost wise, palm trees fibers are affordable since they are agricultural waste. However, the cost of chemical treatment and PLA polymer should be taken into consideration.

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Credit author statement

Hyder Al Abdallah: Data curation, Investigation, Writing analysis; Writing, and Draft Preparation. Basim Abu-Jdayil: Conceptualization; Investigation, Funding acquisition; Supervision; Writing - Review and Editing.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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