Review Article

The Influence of Fiber Processing and Alkaline Treatment on the Properties of Natural Fiber-reinforced Composites: A Review

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Abstract
Throughout generations, research on natural fiber-reinforced composites (NFRCs) has been growing and yielding promising results. The notion of blending natural fibers with polymers comes from the composite's suitable properties, not limited to low density, availability at a low price, biodegradability, and environmental friendliness. The quest for high-performing and marketable NFRCs is driving innovation in the synthesis of such materials. A suitable combination of parameters that optimizes the mechanical and functional properties of the composites without increasing the cost of production is desired. The main objective of this review is to evaluate some of the parameters that influence the behavior and properties of NFRCs. The influence of alkaline modification and natural fiber processing parameters, such as particle size, modification concentration, soaking duration, processing temperature, fiber-to-polymer ratio, and adoption of additives, on composites are discussed. This review summarizes some of the work and provides some directions in the search of an all-around performing economic NFRC.

Keywords: Alkaline, Particle size, Soaking duration, Temperature, Natural fiber-reinforced composites

1 Introduction

The notion of reinforcing polymer composites with fibers dates to 1908, where cellulose fiber in phenolic was used [1]. Moving forward to the 1970s and 1980s, synthetic fibers slowly replaced cellulose fillers due to their performance. The applications of cellulose fillers were limited to the making of strings, ropes, carpets,

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clothing, and decorative products [2]. However, the processing of polymer composites incorporating natural fibers as reinforcements has increasingly grown worldwide [3], [4]. This comes from the desire to obtain advanced materials with good characteristics and functional properties, such as sustainability, biodegradability, durability, and meeting the required needs [1], [5]. For many years' materials such as glass, carbon fiber, and steel have been performing well on aircraft and automotive components. Since then, challenges on developing fuel-efficient components, meeting the demands of the worldwide regulating bodies, and use of light renewable materials have been growing. Hence the development of hybrid composite materials incorporating natural fibers has been of interest [6]. Additionally, government environmental regulations, Greenpeace groups, and non-governmental organizations (NGOs) are compelling the industry to implement processes that are friendly and adhere to the ecosystem. The awareness is stimulating the conservation and preservation of the environment for upcoming generations [7], [8]. In 2015, an increasing production of about 45 million tonnes of grains amounting to 54 million tonnes of rice straw was reported in Vietnam. Meanwhile, burning has been resorted to on most of the postharvest rice straw in open fields resulting in environmental pollution and waste of potential resources. A 2016 survey done by the Food and Agriculture Organisation of the United Nations (F.A.O) reported global paddy production of 761.9 millions tonnes in Asia set to lead the global recovery, with grains production of 680.1 millions tonnes [9]. There was an increase in production in some regions such as Africa and South America [10]. Conversion of agricultural waste such as rice straw to useful materials, as a way of resolving environmental problems and conserving the global environment, is necessary [11], [12]. Recently, governments desire and invest in green composites, these are completely eco-friendly and sustainable, the reinforcement and matrix are fully biodegradable from renewable resources [13].

The main disadvantage arising from using manmade fillers (carbon, glass, and aramid) to reinforce epoxy, polyurethanes, unsaturated polyester resins, and phenolics is the removal of the composites after their end of a lifetime [1]. Natural fiber-reinforced composites (NFRCs) are comprised of blending natural fibers (such as hemp, flax, bamboo, etc.) and polymers to make new materials with improved properties of polymer and fiber [14]. Polymers can be biodegradable and non-biodegradable. Even though, non-biodegradable polymers provide excellent properties, considerations are necessary as they cause ecological imbalance and severe health problems to human beings as they release toxic gases when degrading [15]. Natural fibers as reinforcements to polymers ultimately improve the stiffness and strength properties of the composites [16], [17]. In a subsection, wood polymer composites (WPCs) are those made from embedding wood fibers into the polymer matrix [5], [18]. Figure 1 shows a typical structure of a plastic matrix covered with fibers [19]. The commonly used plastics are thermoplastics and thermosets, such as polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS), low-density polyethylene (LDPE), high-density polyethylene (HDPE), epoxy-resin, etc. [1], [20]–[22]. The choice of natural fibers has proved beneficial due to their availability at a reasonable cost, generation of rural-based economy, non-hazardous during processing, recyclability, their biodegradable nature, and ability to substitute inorganic fibers, such as carbon and glass in reinforcing polymers [23]–[25]. Despite natural fibers being susceptible to moisture absorption, microbiological attacks, low thermal resistance, and undesirable mechanical properties for engineering plastics [1], considerable attempts (by chemical and physical modification) to improve the quality of fiber-polymer interfaces have been made [2], [26]–[28].

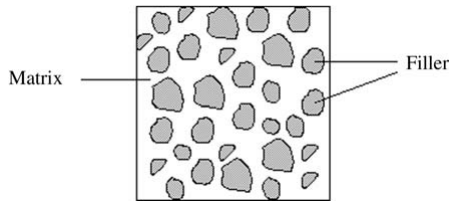

Figure 1: The complete coating of fillers with plastic [19].

Different forms of chemical modification, such as alkaline [29]–[31], silane [32]–[35], enzymes [36]–[38], benzoylation [39], [40] and acetylation [41], [42] have improved the physical and mechanical properties of natural fibers. The interaction of fibers and polymers was improved, producing better functional properties of the composites [24], [43]. The quantity of cellulose, which is narrowly related to the crystallinity of

the fiber and the micro-fibril angle, influences the mechanical properties of plant fibers [26], [44]. Superior mechanical properties have been observed on fibers possessing high cellulose content or high crystallinity [26]. As environmental concerns grow worldwide, it is believed that natural fibers are on the verge of becoming major components of cars. In attempting to reduce vehicle weight, a shift away from steel alloys to aluminium, composites, and plastics has been predicted. Shortly, polymer composites and polymers will make up to ~15% of the aggregate car weight [45]. WPCs apply to decking, fencing, automotive, outdoor furniture, and siding [46], with the fastest growth seen in exterior domestic construction [47]. Natural fiber polymer composites are also applicable to spacecrafts and many German automotive companies such as Ford, BMW, Opel, Audi, Volkswagen, and Mercedes [48], [49]. Bio-based nanomaterials have the potential of delivering environmentally friendly high-performing materials that can replace some of the man-made materials [50].

2 Natural Fiber Polymer Composites

Cellulose, the main structural component of cell walls for natural fibers has attracted the paper and cardboard industry. In the pursuit of high-performance environmentally friendly packaging components, natural plant bodies that are rich in crystalline cellulose nanofibers are considered. They can make transparent and extremely strong films [50]. Research on plant-based natural fibers is growing at an alarming rate for the stated reasons. The specific modulus of hemp, flax, and softwood is comparable to that of E-glass and S-glass due to their low density (1.5 g/cm^3). The density of natural fibers could be beneficial for components where weight reductions are a priority [51]. The potential displayed by nanomaterials from natural fibers promises green materials, high-performance bio-based materials that can substitute some of the synthetic fiber [50]. Furthermore, natural fibers control the environmental problems by restricting the emission of the greenhouse effect from several gases [52].

Wood polymer composites (WPCs) are multiphase structural materials. The WPCs market has seen increased growth especially for decking applications and the demands will increase radically because of Environment Protection Authority (EPA) regulations that ban arsenic-containing treatments of construction materials. According to EPA, the chromate copper arsenic usually used as wood pesticide and preservative in the pressure treatment process has the potential of leaching out of the wood contaminating the water table [53]. The adoption of wood-polymer composites could eliminate such problems [54]. The growth of WPC in the market is predicted to be worth around US$5.8 billion globally by 2021. Wood flour as the most frequently used filler retains high specific strength and modulus, it is eco-friendly and readily available at a reasonably low cost [55], [56]. Wood is rather a filler for plastic and increases the stiffness of the composite without improving its strength. Recovery of wood flour caloric value through furnace and composting can be used for disposal at the end of its useful life. This is not possible with inorganic fillers such as glass fiber, non-ferrous metals, talc, carbon, and calcium carbonate. However, wood flour has unattractive characteristics like low thermal resistance, high moisture absorption and because they are hydrophilic, they result in poor interfacial interaction with hydrophobic polymer matrix during manufacturing [57]. The high hydroxyl group (-OH) contents of cellulose play a major role in the poor compatibility of the fibers and polymer matrices [2]. Figure 2 shows the forms of wood-matrix interface bonding mechanisms responsible for the interfacial adhesion [46]. The properties of fiber-reinforced composites depend on the interface of fiber and matrix. Fiber breakage, fiber debonding, and fiber pullout are the main causes of mechanical failures of fiber-reinforced composites [58]. The ability to transfer stresses from the matrix to the fiber, which ultimately determines the strength of the composite is dependent upon the quality of this interface.

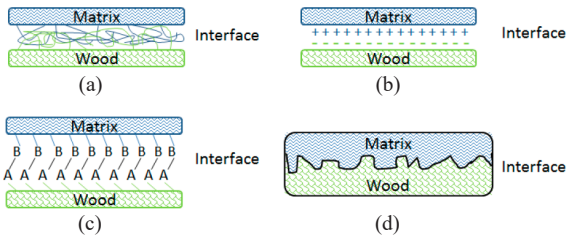

Figure 2: Wood-matrix interfacial bonding mechanisms: (a) molecular entanglement following interdiffusion, (b) electrostatic adhesion, (c) chemical bonding, and (d) mechanical interlocking [46].

The production of WPCs and recyclability consumes less energy in comparison to conventional building materials like cementitious products and metals. For instance, cement is sintered at 1200°C which is 1020°C higher than the compounding of WPCs. The cost of virgin polypropylene is about 1.04–1.43 €/kg while wood flour costs around 0.09–0.18 €/kg. When considering performance and processing, the final cost of WPCs is considerably lower than that of laminated or solid wood decking. The market price of outdoor solid teak wood decking is above 80 €/m^2 whereas that of WPC decking is around 45–50 €/m^2. P. elongate/PP composites reduce thermoplastic composite manufacturer's exposure to rising petroleum prices and produce end-products having improved structural rigidity and marketable performance capabilities [59]. The cost of glass fiber ($2900/ton) or mica ($700/ton) is significantly higher than that of producing grafted aspen wood fiber which is $250 to $350/ton [60]. WPCs are showing interesting prospects [5]. WPCs are gaining popularity as they are low maintenance, durable while possessing acceptable stiffness and strength at lower prices relative to other competing materials. WPCs have the potential of becoming fully natural materials when the matrix is based on natural polymers. WPCs are resistant to natural conditions such as biological deterioration for applications where the untreated timer is not recommended. They are cost-effective as they do not require painting, polishing, and surface treatments as wood products [61]. They are advantageous as their properties are controllable [62].

3 Fiber Processing

The processing of natural fibers has a strong relationship with the functional properties of the resulting fiber-reinforced composites [63]. Fabrication of superior bio-composites is faced with challenges such as efficient biofiber surface treatment, bio-composite processing, bio-composite formulation, and matrix polymer modification [64]. Adhering to such challenges, efficient and effective surface treatment, processing, and ratios are some of the engineering concepts for making engineered bio fibers ready for composite fabrications [45], [64]. Some of the processing parameters that are influential to the performance of natural fibers and their respective fiber-polymer composites have been discussed below.

3.1 *Alkaline modification*

The alkaline modification also known as mercerization, is the commonly used surface modification of natural fibers, most economic and effective [65], [66]. The modification changes the fiber composition and responds to its structure [67]. It involves immersing fibers in an aqueous solution of sodium hydroxide (NaOH) of different concentrations, Equation (1) [68], [69]. Numerous researchers have subjected different fibers to alkaline concentrations ranging from 0–50% [70]–[78]. They have reported improvements in the quality of fibers and the mechanical properties, due to good adhesion between the fibers and polymers. Alkalization increases the surface roughness of fibers and disrupts the hydrogen bonding in the network structure. It also changes the micro-fibril aggregation in the cell wall creating a strong interaction between the matrix and fiber [3], [79], [80]. The numbers of reactive functional groups are increased following the removal of waxes and natural fats from cellulose fiber surfaces [81], [82]. Swelling induces fiber deconvolution which influences the orientation of the crystallites [24], [83]. Swelling is beneficial in relieving the internal stresses in fibers [84]. Alkaline treatment increases the luster and improves chemical reactivity [83]. Depending on the concentration, the modification extracts a certain portion of non-cellulosic components such as oils, wax, and binding hemicellulose and lignin covering the fiber cell wall promoting the establishment of new hydrogen bonds between the cellulose chains [85]. Immersing natural fibers in sodium hydroxide produces alkoxide as a by-product of ionizing the hydroxyl group in the fiber [3]. However, the literature suggests that high concentrations (more than 10 wt%) are destructive to natural fibers [21], [58], [86].

Alkaline modification improves the order of cellulose crystals and is usually reflected by high crystallinity index. The partial removal of binding non-cellulosic components such as lignin yields better packing of cellulose chains, and transformation of cellulosic form from I to II. High crystallinity index is a characteristic of strong and stiff fibers, desirable for producing better composites [26], [58], [87], [88]. Improvements in tensile properties following alkaline

treatment are credited to interacting factors like the rupture of alkali-sensitive bonds present amid the hemicellulose and cellulose, owing to the extraction of hemicellulose promoting homogeneity of fibers, and transferring of stress between interfibrillar regions [89]. A disadvantage of using alkaline treatment is the deterioration of fiber strength during modification [30].

3.1.1 The effect of particle size

Particle size and distribution play an important role in the performance of natural fiber-reinforced composites. The effect of particle size and distribution has been investigated, and reports have shown its influence on the mechanical and functional properties of the composites [90]–[96]. Olakanmi *et al.* [97], examined the effects of particle size/distribution (–75, +75–150, +150–210, +210–300 and +300–425) μm on the functional properties of WPCs. Agglomeration, segregation, and incomplete wetting of wood flour particles during processing could be eliminated by the selection of suitable wood particle size. This improves the interaction between polymer and wood flour which ultimately enhances the thermal and mechanical properties of the composite. They reported optimum properties of WPCs at particle size/distribution of +210–300 μm. Poor dispersion from uneven mixing of wood fibers in the polymer matrix due to wood fiber size increased thickness swelling. Clusters form when the wood fibers are uncovered by the polymer matrix. Micro cracking and more gaps occur from the swelling of wood fiber [98]. Zaini *et al.* [90] studied particle size (63–250) μm and revealed increases in flexural modulus (stiffness), tensile, and impact strength with an increase in particle size. They also found that particle size of 250 μm produced optimum mechanical properties. Siwek *et al.* [99], reported optimum mechanical properties on the particle size of 255 μm.

Composites of particle sizes 150 and 300 μm were investigated [100]. The flexural and tensile properties of the composites were better on the composite with particle size 150 μm. This was attributed to better compaction and reduced porosity giving effective stress transfer between the fiber and matrix. A recent study on the effect of grain size (between 250 and 950 μm) on the structure and properties of composites has been reported. They found optimal flexural strength on composites of grain size 950 μm, while the impact strength improved by 92% for composites with grain size 250 μm [101]. Optimum tensile, elastic modulus and the least water absorption were produced by composites reinforced with date palm seeds of particle size 0.5 mm in comparison to those of 2 mm and 2.8 mm [102].

Sälzer *et al.* [103] evaluated particle shape and size in correlation with the mechanical properties. The average particle sizes; 500–1000, 200–400, 40–120, and 20–40 μm were denoted as "very coarse, coarse, fine and very fine" respectively. They concluded that medium-sized particles (coarse and fine) produced better tensile and flexural properties. However, high stiffness correlated with the aspect ratio as longer particles produced superior stiffness. Composites fabricated from larger-sized wood particles were characterized with more and larger pores while those from small wood particles possessed a higher compaction ratio. Thickness swelling, and water absorption rates were lower on particle size < 0.59 mm and < 590 μm. Similarly, Kaboorani [104], associated less absorption rate with smaller particles, stating that larger particles in WPCs led to higher water absorption rate because of larger surface area exposed to reaction with water. However, the bending properties were found to be higher on composites made from larger particle sizes [105]. A similar observation has been reported [106]. Decreases in mechanical properties despite high aspect ratio could be attributed to the breakage of too-large particles during processing [106]. Composites from large particles have been associated with high thickness swelling, diffusion coefficient, and poor water absorption properties [104]. Rimdusit *et al.* [107], showed increases in tensile and flexural strengths with an increase in particle size up to 275 μm, where decreases were observed. Optimum properties were reported at a particle size of 275 μm. Poor mechanical properties linked with small particle size are attributed to difficulties in dispersion from particle-particle interactions.

The authors in [93], [108] reported marginal improvements in the mechanical properties following variation of particle size. Meanwhile, Stark & Rowlands [108], argued that aspect ratio, not particle size has the supreme effect on the stiffness and strength of wood/PP composites. Enhanced stress transfer from the polymer matrix to the fiber has been observed on fibers with a high aspect ratio, eventually improving the mechanical properties of the composite [106], [109], [110]. However,

Gozdecki *et al.* [111], did not report any statistical differences between mechanical properties of WPCs despite virgin wood particles (VWPs) having a higher aspect ratio than recycled wood particles (RWPs). Though mechanical properties increased with increasing aspect ratio, the highest aspect ratio on analysis by Reddy *et al.* [69], did not produce the best mechanical properties. Differences are rather difficult to explain but, they could be attributed to factors of incorporating coupling agent type and content, wood content, processing method, the polymer used, and wood particle geometry. Gozdecki *et al.* [112], concluded that an increase in particle size to some extent leads to improvements in the mechanical properties.

3.1.2 The effect of alkaline concentration

The concentration of alkaline treatment has been reported to be of great influence on the performance of natural fibers and the resulting composites [30], [72], [74], [113]–[116]. Researchers are on the verge of determining the best alkaline modification concentration as the behaviour on the same concentration differs with respect to natural fibers. Kumar *et al.* [31] investigated alkaline concentrations of 3, 6, and 9% on kenaf/epoxy composites and reported optimized properties at a concentration of 6%. Cai *et al.* [21], subjected abaca fibers to 5, 10, and 15% aqueous NaOH solution whereas Oushabi *et al.* [58] treated date palm fibers with 2, 5, and 10% NaOH at 25°C for 2 h and 1 h respectively. The concentration of 4% NaOH was examined by Olakanmi *et al.* [97], on waste Daniella Oliveri wood flour at ambient temperature. These researchers found the alkaline concentration of 4 and 5% to be optimizing the crystallinity of cellulose, tensile strength, and Young's modulus. The optimal alkaline concentration condition has been reported to be at 4 wt% NaOH in the presence of 1 wt% dopamine for bamboo/PLA composites [117]. The concentrations of 4, 6, and 8% were examined [118], and found optimum thermal and mechanical properties at 6% NaOH and deteriorated as concentration increased. The interfacial shear stress (IFSS) indicated a weak interlocking bond between fiber and matrix after the 6% NaOH concentration. These studies [75], [119] reported optimum impact, hardness, tensile and flexural strengths following 6% NaOH treatment. The 6% NaOH surface modification on agave americana fibers was reported to improve the thermal behavior, tensile strength, and wettability due to extraction of the amorphous materials [120].

Cai *et al.* [21], associated decreases of tensile strength and Young's moduli following fibers treated with high alkaline concentrations of 10 and 15%, to fiber breakage and complete removal of fiber binding materials under strong modification conditions. High alkaline concentrations of 10 and 15% contract and twists the microfibrils along the longitudinal axis, accounting for higher strain at break [21], [121], [122]. The 5% NaOH concentration demonstrated better interfacial bonding between the treated fibers and epoxy matrix producing composites that possessed superior interfacial shear stress (IFSS). Not only does the 5% alkaline modification induce less damage to the fibers, but it has also been suggested through SEM to achieve better fiber surface cleaning, roughness, and rigid surfaces that promote good mechanical interlocking adhesion with matrix [21], [58]. The effect of NaOH concentrations of 1, 3, 5, 7, and 9% were studied, and the flexural and tensile strengths increased with the increase of NaOH concentration up to 5% thereafter a decrease was reported. However, the impact strength increased up to 7% NaOH concentration. They recommended the 5% NaOH to be the optimal concentration for the treatment of natural fibers [123].

These studies [23], [86] suggested that 5 wt% NaOH modification produced superior tensile properties. The fibers were twisted while the lumen of abaca fibers collapsed as a result of swelling of the cell wall following subjection to strong concentrations of 10 and 15 wt% [86]. Absorption of sodium ions into the cellulose structure breaking bonds between cellulose sheets could lead to swelling [21], [72]. Alkaline treatment extracted some artificial and natural impurities [23]. An alkaline concentration (1, 2, 3, 4, 5, 6, 7, and 8%) study by [124], on kenaf/PP composites, suggested increases in flexural and tensile properties with an increase in concentration up to 6% thereafter decreases were reported. Optimum kenaf/PP properties were found to be at 5–6% NaOH concentration and the mechanical properties approached those of glass fiber reinforcement, with specific properties exceeding. Meanwhile, concentrations less than 1% NaOH were reckoned insufficient due to the presence of impurities preventing good adhesion. Fibers from sugarcane bagasse were immersed in NaOH concentrations of

1, 2, 4, 8, 15, 20, and 40 wt%. The stiffness of the fibers optimized at concentrations of 2–4 wt%, while the tensile was more pronounced at 5–8 wt%. They concluded that 5 wt% NaOH was the optimal treatment concentration at 1h soaking duration [125].

However, better degradation resistance and mechanical properties were reported on composites treated with 3% NaOH, the water absorption reduced with an increase in alkaline concentration, and better hydrophobicity of composites was reported at 5% NaOH treatment [126]. The weight and diameter of sisal and oil palm fibers reduced following alkaline concentrations of 0.5, 1, 2, 4, and 10% at room temperature. However, the maximum tensile strength of the composite was found to be produced by fibers treated at 4% NaOH [127]. Though it is suggested that surface modification improves surface tension, wetting, porosity, and mechanical properties [23], [97], sisal fibers were subjected to 4% NaOH and reduced mechanical properties when compared to untreated [128]. These reductions could be characterized by an insufficient soaking duration of 1h since the concentration of 4% NaOH has been associated with optimizing the functional properties of natural fiber-reinforced composites [97]. The effect of alkaline concentrations (1–9%) was studied by the current authors [68] and [65], they concluded that concentrations of 1–3% were insufficient in cleaning the fiber surfaces as depicted by the topography of the fibers. The concentrations of 5% and 6% were reported to improve the overall weight, the crystallinity of the fibers [68], and the tensile strength recorded the highest average unit break of 243.7 N/mm^2 respectively. The low average unit break recorded at 9% NaOH was attributed to fiber damage and fibrillation [65]. A similar observation has been reported about concentrations of 15% and 10% [129]. Despite decreased tensile strength and crystallinity of cellulose following high alkaline concentrations, better weight losses, diameter, cross-sectional shrinkage, and strain at break have been reported [21], [129]. It was characterized by the stripping of the hemicellulose and lignin from the fibers, inducing twisting of fibers. Composites fabricated from 5 wt% NaOH treated fibers demonstrated superior interfacial shear strength (IFSS), reflecting excellent adhesion with the epoxy when compared to those of 10 and 15 wt% NaOH [21]. Higher concentrations do not only cause fibrillation, but cellulose chains are also degraded lowering the density of fibers [74], [85]. However, Alawar, Hamed, and Al-Kaabi [130] concluded that 1% NaOH optimized the mechanical properties among alkaline concentrations of 0.5, 1.0, 1.5, 2.5, and 5%. They reported increased porosity and further weakening of fibers following an increase in alkaline concentration, yielding poor tensile properties of the composites. An observation made is that soaking duration and treatment temperature highly influence optimum alkaline concentration. For instance, prolonging fiber treatment at high temperatures on 5% NaOH would eventually damage fibers yielding poor mechanical properties. In essence, high temperatures and prolonged dipping time are suitable for lower sodium hydroxide concentrations.

3.1.3 The effect of soaking duration

Fiber soaking duration has proved imperative on the performance of natural-fiber reinforced composites. Literature [31], [69], [87] has shown the significance of considering the duration of treatment. The structural and functional properties of natural fibers have shown noticeable differences following subjection to various soaking duration. Prolonged soaking durations at high alkaline concentrations were reported to increase fiber shrinkage which proved detrimental to jute fibers, ultimately affecting the mechanical properties as crystallinity ratio reduced. The DP was also reduced due to fiber shrinkage [73]. Short soaking duration is insufficient in cleaning the fibers as fiber surface impurities were observed [42]. The crystallinity of fibers, the orientation of crystallites, and the length of crystallites with fiber axis are influential to fiber strength [84]. Reddy *et al.* [69] immersed Borassus fibers in 5% NaOH solution for 1, 4, 8, and 12 h. They reported increased tensile strength, modulus, and elongation from 0 h up to 8 h hereafter a decrease was observed. Optimum tensile strength (121.3 MPa), modulus (35.2 GPa), elongation (58.1%), crystallinity index (58.4%), and cellulose contents (63%) were recorded at 8 h soaking period. A similar treatment was done by Rout *et al.* [131], maximum cellulose content was produced by a soaking duration of 6 h and reduced with prolonged treatment of 12 h. The crystallinity index increased up to 24% for 6 h treatment period where a decrease was observed for 12 h duration. This explains the increase in tensile properties with treatment time up to 6 h where a decrease was observed.

However, the density increased with soaking time. They suggested longer treatment time partially removed crystalline cellulose content. Soaking duration of 8 h was also reported to optimize the tensile strength of the composites [31].

Alkaline treatment durations of 0, 2, 4, 6, and 8 h were investigated [132]. They reported a correlation between crystallinity and brittleness at the soaking time of 8 h. The highest modulus was recorded at 8 h of treatment, fibers were separated from large extractions of hemicellulose. However, drastic changes in the rate of flexural, tensile strength, modulus, tenacity, and percent strain occurred within 0.5–4 h of soaking time, with a maximum at 4 h. Prominent losses of cementing hemicellulose component were observed at a soaking time of 4 h. This improved the closer packing of cellulose chains, giving fibrils leverage to reorientation along the direction of tensile forces [89], [133]. Similarly, Mohan, Reddy, and Gowda [134] concluded that 4 h of alkaline treatment was better than 20 h of treatment. Zin *et al.* [118], immersed pineapple leaf fibers for periods of 1 and 3 h. They found optimum tensile strength following 3 h of alkaline treatment while the 1 hour produced the highest interfacial shear stress and thermal stability of fibers. A comparable observation was reported on kenaf fibers [75]. These researchers [21], [86], subjected abaca fibers to alkaline treatment for durations of 30 min and 2 h respectively. Fibers immersed for 30 min produced superior tensile strength of 847 MPa in comparison to 773 MPa of 2 h. The young's modulus was found to be better for fibers treated for 2 h than those of 30 min. Borysiak and Doczekalska [72] immersed wood fibers in aqueous NaOH for intervals of 15, 30, 45, 60, and 90 min respectively. They reported a decrease in the degree of crystallinity as soaking time increased. However, effective conversion of cellulose I to cellulose II was ideal at prolonged soaking duration. Fiore *et al.* [135] reported that 6 wt% NaOH for 48 h was more successful on cleaning fibers whereas 144 h damaged fibers as the propagation of cracks along the axis of the fibers was evident. Such fibers usually yield poor mechanical or lower tensile strength [136].

Rokbi *et al.* [137] suggested that soaking duration of 24 h in 10% NaOH optimized flexural strength and modulus by 60% and 62% respectively. They concluded that a prolonged duration of 48 h made fibers stiffer and brittle. Mokaloba and Batane [42] examined soaking fibers for 45 s, 15, and 45 min in 6% NaOH respectively. They reported better crystallinity, tensile strength, and IFSS at 45 min soaking period. Soaking duration of 45 s and 15 min were reported insufficient as surface impurities were present under microscopy study. Salah *et al.* [113] reported optimum crystallinity and mechanical properties at 2 h of 3% NaOH treatment.

3.1.4 The effect of temperature

Limited investigations have been reported on the effect of temperature on the mechanical properties of natural fibers and the respective natural fiber-reinforced composites. Gassan and Bledzki [73] conducted alkaline treatment at 20 and 50°C. They reported that 20°C improved shrinkage of fibers which enhanced the degree of polymerization (DP), crystallinity ratio, Harmans factor (orientation), and the tensile properties. Significant improvements in the tensile strength and crystallinity of fibers increased following heat treatment of up to 140°C after a decrease [129]. However, they decided to adopt 25°C for alkaline treatment. The tensile strength was optimized at the temperature of 30°C [31]. Edeerozey *et al.* [65] considered alkaline treatment at room temperature at 95°C. They reported better fiber cleaning at 95°C as the highest value of fiber unit break was recorded. Saha *et al.* [138] examined the effect of alkaline treatment at ambient (30 ± 2°C), elevated (90 ± 2°C) and steam (125 ± 2°C) temperatures. They reported the highest mechanical properties, weight loss and chemical composition at 125 ± 2°C, then at ambient and 90 ± 2°C respectively. As treatment temperature increased, alkali concentration and dipping/soaking time reduced. Alkaline treatment of natural fibers at elevated temperatures promotes better fibril separation through heat energy, aiding the breakage of hydrogen bonds holding fibrils together [65], [138].

3.2 *The effect of fiber to polymer ratio*

The ratio of fiber to polymer plays a crucial role in determining the mechanical properties of the natural fiber-reinforced composites [7], [93], [139]. Properties such as thickness swelling, and water absorption affect the durability of the WPCs, and they highly depend on the contents of fiber and plastic. Plastic and wood contents highly influence the thickness swelling and

water absorption properties of the WPCs [98]. It is important to determine a good fiber to plastic ratio as increasing fiber content yields poor water absorption and thickness swelling properties with decreasing plastic contents. The presence of voids and poor adhesion between wood fiber and polymer matrix as a result of reduced plastic content encourages hydrophilicity of the composites [92], [98]. Considerable amounts of filler (30% PS, 30–40% for PE, and 20–40% PP) increased the strength of the composites in comparison to the unfilled composites. As fiber contents increase, the mechanical properties decrease [97]. The differences are attributed to the nature of pre-treatment, the species, and or processing technique [140].

A study done by Raj *et al.* [141] showed an increased tensile modulus of 800 MPa and the stress at yield also increased from 10.9 MPa to 17.3 MPa at 30.0 wt% fiber content. Mishra *et al.* [23] also reported increases in tensile strength, impact strength, and flexural strength up to fiber content of 30 wt% where a decrease was observed. Maximum tensile strength, impact, and flexural strength of about 43 MPa, 80.29 J/m, and 87 MPa respectively were recorded at the fiber content of 30 wt%. They found the high toughness of the fabricated natural fiber polymer composite to be in the classification of tough engineering materials. A similar observation was reported [142]. Poor water uptake properties were reported at 70% sisal fiber content whereas high impact strength was reported at 50% fiber content while 30% produced excellent hardness, water absorption, wear resistance, and flexural properties. A review done by Akil *et al.* [139] revealed optimum tensile properties around kenaf fiber content of 70% on kenaf/PLLA composites. However, for kenaf/PP-MAPP, optimum mechanical properties were reported at the kenaf fiber content of 40%. They concluded that the nature of polymer and additives used to play a major role in determining the optimum fiber to polymer ratio.

Studies conducted [59], [98], [104] revealed increased thickness swelling and water absorption properties with an increase in wood content. This was attributed to increased hydrogen bonding sites as wood content increased. Wood being hydrophilic and rich in a hydroxyl group, readily reacted with water molecules by hydrogen bonding. The modulus of rupture (MOR) was optimum at 40% fiber content while the tensile and notched impact strengths were highest at 30% fiber content. They recommended a ratio of 50/47/3 of P. elongata wood flour, PP, and MAPP respectively, for outdoor siding and deck flooring [59]. As moisture content, water absorption and thickness swelling increased, the bending properties were seen to deteriorate with an increase in fiber content [105]. The functional properties of Daniella oliveri/vHDPE composite were optimized at a wood flour content of 35 wt%. Undesirable mechanical properties were produced at low wood flour contents of < 35 wt%, attributed to low load transfer from localized segregation of wood particles. Non-uniform stress transfers due to the occurrence of agglomeration on high wood flour content (> 35 wt%) were developed, and microcracking at the interface as a factor of high wood flour content led to poor mechanical properties [97]. The uniform distribution of stress is thought to be dependent upon the orientation and population of fibers. Therefore, appropriate fiber loading is crucial for the functional properties of composites. The tensile and tear strength of sisal/oil palm reinforced composites were optimum at 30 phr fiber loading [127], [143]. Fiber loading of 30 phr is appropriate for maximum orientation and the right population of fibers to actively take part in transferring stress from the matrix along the fibers [127].

Somashekar and Shanthakumar [144], reported the highest mechanical properties at 20 wt% fiber content. Though they did not exceed fiber contents of 20%, the mechanical properties increased with increasing fiber content. Hardness, Young's modulus, minimum torque (a measure of viscosity), and maximum torque increased with wood fiber loading of up to 20 phr. The presence of filler in the matric reduced the mobility of macromolecular. Optimum scorch time, rupture, and stress at yield were found to increase with filler content up to 15 phr after a decrease was observed [2]. The mechanical properties declined with an increase in filler content [90]. However, the flexural modulus (stiffness) increased with a steady increase in filler content. Bledzki, Gassan, and Theis [145] reported increased tensile strength and Young's modulus of about 50 to 150% respectively, due to an increase in wood content from 35 to 50 wt% in wood/PVC-soft composite. Wood fiber and flour at 40% filler level produced higher unnotched impact, tensile and flexural strengths with the aid of MAPP [108]. Increasing filler content from 30 to 40% decreased the Charpy properties of WPCs. A significant increase in tensile modulus

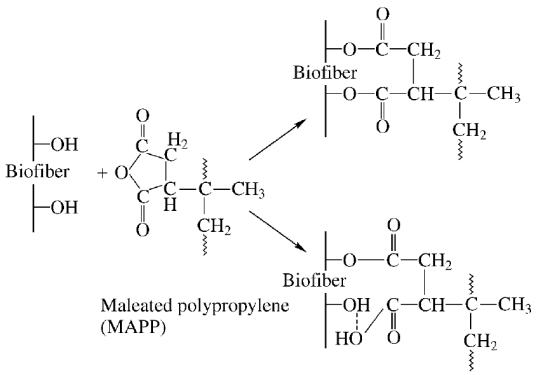

Figure 3: Reaction of natural/biofiber with MAPP [45].

corresponds with increasing filler content [103].

3.3 *The effect of additives*

Additives and or coupling agents improve the interface bonding between hydrophilic natural fibers and hydrophobic polymers by avoiding phase separation, yielding stronger materials [60]. MAPP is commonly used to improve the mechanical interaction of polymer and fiber,

Figure 3 [45], [64]. Literature reports a variety of commonly adopted additives and the advancements brought by coupling agents to the mechanical properties of natural fiber-reinforced composites [19], [59], [78], [146], [147]. Improved adhesion between fiber and polymer due to coupling agent reduces the water absorption and thickness swelling tendencies. WPCs without coupling agents have been characterized with high levels of porosity allowing faster penetration of water molecules [104]. Coupling agents officiate transferring stresses from matrix to filler [2]. Coupling agents such as maleic anhydride (MAH) improve the wettability of natural fiber in composites [148]. The addition of talc improved the tensile moduli of PLA/WF composites [149].

Stark and Rowlands [108] suggested that the addition of MAPP produced at least three times stiff and twice strong composites in comparison to those without 3% MAPP. MAPP content of 4 wt% has been found to optimize the tensile properties. Fiber debonding and tensile failure of the matrix were reported on composites without MAPP whereas those with MAPP failed due to local shear yielding of the matrix around the fiber [150]. MAPP provided better filler-matrix interaction, improving the tensile, impact, and flexural properties of the composites. The effect of filler content became insignificant in the presence of MAPP [103]. Incorporating MAPP enhanced the thermal stability, better than composites without MAPP and polypropylene on their own [93]. Government and Onukwuli [119], investigated the effect of maleated polyethylene (MAPE) on HDPE/avocado composites. They reported the highest hardness, impact, tensile and flexural properties and the least water absorption on composites incorporating MAPE.

$$Fiber-OH + NaOH \rightarrow Fiber-O-Na^{+} + H_2O + impurities \quad (1)$$

4 Conclusions

There are many factors that influence the properties of NFRCs and they are not limited to surface modification, particle size, concentration, soaking duration, temperature, and fiber-polymer ratio. The NFRC's functional and mechanical properties are also dependent on other factors, such as the nature of the natural fiber, climate conditions, and growth medium. According to the findings of this review, appropriated particle size/distribution is important in the processing of fibers for the manufacturing of fiber-reinforced polymeric composites. Very coarse particles (> 400 μm) and very fine particles (< 40 μm) are not recommended. Particle size around 250 μm has been found to optimize composite's properties and aspect ratio (length to thickness) is highly influential on fiber-reinforced polymer composites.

Alkaline concentrations of 5 and 6% are ideal for modification of natural fibers at room temperature. However, few studies have been done to investigate the involvement of alkaline concentration and temperature to optimize the performance of the composites. Optimal alkaline concentration still demands further investigation without the influence of other external parameters. Soaking periods of 2–4 h act rapidly on improving the quality of fibers by loosening and separating the fibers through the loss of hemicellulose. The 8 h soaking time has been reported to optimize the functional properties. Conflicting views have been reported on soaking duration as the times and concentrations are not consistent. Sufficient tests have not been done for an clear comparison of soaking durations to identify best condition to improve mechanical properties.

Although the influence of temperature has been overlooked, many reports have shown improvements in the functional properties following treatment at room temperatures and elevated temperatures. Inconsistent results from different research has been observed, raising the further demands for investigations. Some studies have reported improvements in mechanical properties in consideration of temperature. The fiber/matrix ratio influences the performance of NFRCs. Fiber separation and agglomeration are highly associated with low and high fiber contents respectively. This reduces the stress transfer capabilities across the matrix to fiber, leading to poor mechanical properties, high thickness swelling, and water absorption properties. Numerous reports showed fiber contents between 30 and 40% to be ideal. The mechanical properties improve significantly following the addition of a couple of agents, such as MAPP.

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