Microwave Treatment on Wood Waste Product-A Review

Mohammad Farsi, Mohammad Jawaid, Amir Amini, Masoud Ebadi, and Majid Shahbabaei

Abstract This chapter presents an extensive review of the scientific literature associated with various microwave treatments on wood waste products. First, the basic concepts of microwave radiation and its applications in wood waste product fabrication are reviewed. Then, an extensive literature review of the most significant experimental research papers is provided, divided into two microwave heating treatment uses: wood drying and wood waste products performance improvement. Next, the post-treatment of wood-plastic composites (WPCs) by microwave irradiation as a case study was reviewed and a real example of WPCs samples was discussed. Finally, the chapter concludes with a proposal of doing future research studies concerning the impact of microwave technology on some important properties of wood waste products, i.e., resistance to biological agents, fire, environmental conditions, and so on.

1 Introduction

Wood is a complex natural composite material made of cellulose, hemicelluloses, lignin, and extractives [1]. Each of these main ingredients has its unique structure. For example, cellulose is a natural linear homopolymer (polysaccharide), in which D-glucopyranose rings are connected with β-(1–4) glycosidic linkages. Different from

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Table 1 Global production of forest products in 2020 [6]

Product	Unit	2020	Production		
			Change (%) compared to		
			2019 (%)	2000	1980
Roundwood	million m^3	3 912	−1	12%	25%
Wood fuel	million m^3	1 928	−1	7%	15%
Industrial roundwood	million m^3	1 984	−2	17%	37%
Wood pellets and other agglomerates	million tons	50	3		
Sawnwood	million m^3	473	−3	23%	12%
Wood-based panels	million m^3	367	−1	107%	280%
Plywood	million m^3	118	2	103%	200%
Particle board, OSB, and fiberboard	million m^3	250	−2	109%	335%

cellulose with several sugar moieties, hemicelluloses are mostly branched polysaccharides with lower molecular mass with a degree of polymerization (DP) of 50–200. Lignin as a randomly branched polyphenol is the most complex polymer among naturally occurring high molecular weight materials made up of phenyl propane (C9) units. All of these materials characterizes as polar polymers witnessed by dipole polarization.

As an important renewable resource, Wood has a broad range of material properties. Relatively high strength and stiffness, natural appearance with interesting texture, good insulation properties, easy machinability, low density, flexibility during the processing with no harm to equipment, good mechanical properties, sustainability, environmentally friendly, and low cost are some of their unique structural and chemical characteristics [2–4].

Owing to unique physical and chemical properties, wood and wood fibers are used in a diverse range of applications such as construction, furniture, packaging, energy production, etc. Hydrophilicity and poor compatibility of those materials with polymer matrices having hydrophobicity characteristics significantly reduce the final performance of the products for use in industries [5].

According to the statistics published by FAO,[1] worldwide wood consumption reached 4802 million cubic meters in 2020 worth around 244 billion dollars (Table 1), which included sawdust, wood panels, sawn wood, and wood pellets [6]. Global wood products trade increased by 143% up to US$244 billion between 1990 and 2019 [6]. The largest contributor to this trade expansion has been related to economic growth and demand from China to import wood products to China which increased by 760% up to US$49 billion [7].

During the secondary production process, wood suffers a lot of waste in the form of sawdust, wood chips, wood bark, sawmill scraps, and hard chips. Today, wood waste is considered an important economic and environmental issue in the world. A large amount of wood waste is produced every year in the world, which requires

[1] Food and Agriculture Organization (FAO).

disposal, reuse, or other end-of-life management. Dovetail Partners [8] estimated that more than 64 million metric tons of wood waste were generated in 2010 with 28 million metric tons recovered, 27 million metric tons still available for recovery, and 10 million metric tons non-recoverable. Bergeron [9] estimated that 50 million cubic meters of wood waste are generated each year in the EU.[2] Currently, wood waste can be used as fuel or building products. A large part of wood waste can be recycled or turned into usable products through chemical, physical, or mechanical treatments. Wood-plastic composites (WPCs) are one of the most important products made from wood waste.

2 Wood-Plastic Composites (WPCs)

WPC polymer constitutes wood waste, plastic, and chemical additives such as lubricants, coupling agents, nucleating agents, pigments, and UV stabilizers. Compared with pure plastics, the incorporation of wood fiber into the plastics led to improved flexural and tensile stiffness [10].

In 2019, the global WPC market size was worth 5.3 billion dollars. It is estimated that the market will grow up to 11.4% CAGR[3] from 2020 to 2027 [11]. Advances in the development of sustainable construction materials coupled with rising repair and renovation activities are driving the market growth. 51 and 22% of WPCs market growth are related to Europe and North America, respectively, while the rest is attributed to Asian and African countries [12].

Forecasts for the turnover of WPCs in the world may reach 13.47 billion dollars by 2026. In Asia, China exclusively possesses 61.8% of the market for WPCs [12]. Reportedly, Asia, Europe, and North America, respectively, share 10, 20, and 70% of the WPCs market in the world with a total global sharing of 900,000 tons [13, 14].

Rail and decking products are the main WPCs market in the USA while the automotive industries hold the main market in the EU [15]. As seen in Fig. 1a, the highest production and consumption of WPCs in the world is in the field of construction followed by automobiles, even though its growing consumption is evident by 2024. As Fig. 1b shows, Asia Pacific and specifically China will reach the USA in the global production of WPCs by 2024. Also, the forecast for 2027 indicates that Europe will overtake America in the production of WPCs [16, 17].

Figure 2 compares the application of WPCs including interior panels, headliners, car dashboards, car roofs, seat panels, parcel shelves, acoustic panels, fencing, decking, railing, cladding and siding, park benches, and so on [18, 19]. There are various methods to produce WPCs. The most important approach is the extrusion and injection molding processes [20].

Generally, there are several advantages for WPCs over mixed plastic-filler materials including low cost and processing difficulties with no expense on mechanical

[2] European Union (EU).

[3] Compound Annual Growth Rate (CAGR).

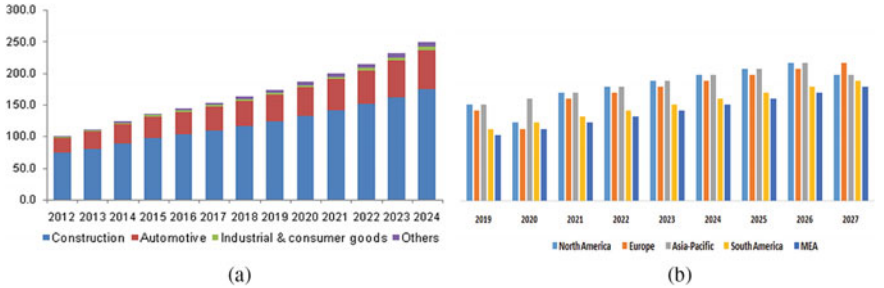

Fig. 1 Markets and countries dealing with wood/natural fiber-thermoplastic composites [16, 17]

Fig. 2 Some applications of wood-plastic composites

properties [21]. In comparison with natural woods, WPCs withstand harsh environments with better structural stability [22] as well as indicate good resistance to termites and fungi [23]. Exploiting wood and plastic wastes to produce high-performance products is the most important hypothesis behind the fabrication of WPCs [24].

Despite having huge advantages, there are several issues with the use of WPCs, as follows:

1. The WPC density is usually twofold the raw wood [25]. Fortunately, some research showed a great reduction in their weight by creating foam structures.
2. The long-term exposure of WPCs to ultraviolet (UV) radiation during outdoor use.
3. Thermoplastic composites usually lack function properly during the long duration of loading since polymers having a linear morphology can strongly respond to time and temperature or creep during loading. Even if, adding fibrous filler into

the polymer matrix may—to some extent—reduce the creep response during loading [26], the problem still remains.
4. Due to the high potential of the destruction of wood fibers at high temperatures, the processing of cellulose fibers in thermoplastics is challenging, thereby limiting their use.
5. Due to the presence of the wood fibers in the polymer field, they act as points of stress concentration and initiation of cracking and failure and thus reduce the impact resistance of WPCs.

In addition to the above-mentioned issues, there are more important problems in the use of WPCs which limit their applications. The high hydrophilicity of natural fibers is against the hydrophobicity characteristics of thermoplastic polymers which makes them incompatible since the mechanical properties of composites strongly depend on the adhesion between fibers and polymers. By improving the bonding and adhesion properties between the fibers and polymers this challenge can be tackled.

3 Improvement of the Interface Region of WPCs

Different methods are needed to modify the characteristics of WPCs in the interphase region with improved adhesion. By modifying the polymer matrix using various coupling functional groups, the interfacial strength and mechanical properties of the products were improved. Maleic anhydride grafted styrene-ethylene-butylene-styrene (SEBS-g-MA) [27] and maleic anhydride grafted polyolefins such as HDPE-g-MA [28], PP-g-MA [29], and LDPE-g-MA [30] are the most common examples of reported researches in the literature. Another approach for the enhancement of interfacial adhesion in the natural fiber-reinforced thermoplastic matrix is the fiber treatment before mixing with polymer. Some of these treatments have physical nature and some of them are chemical. Furthermore, plasma, microwave irradiation, and corona treatments of the fibers and wood waste products are some physical treatments reported in the literature with enhanced polymer-matrix adhesion [31–33].

Employing nanoparticles is another strategy to improve the physical and mechanical properties of the composites. Polymeric composites can be turned into nanocomposites by integrating with nano-sized particles which led to the improved mechanical strength of composites [34–37]. Nanocomposites made of carbon nanotubes (CNTs), nanoscale clays, and nanoscale SiO_2 received great excitement for industrial applications [38–40].

Nano-based composites present a new venue for the enhancement of wood properties on the horizon of wood modification processes for different applications. Wood-polymer nanocomposites could be a promising approach to obtaining effective products with better physical, thermal, and mechanical properties [41].

For example, utilizing silicate minerals in multilayer played as an in-situ reinforcement to substantially enhance the physical and mechanical properties of various thermosets and thermoplastics at low levels [40, 42–46], thanks to their excellent features

such as rich intercalation chemistry, high mechanical stability, high aspect ratio for individual platelets, abundant in nature as a low-cost material, high gas barrier ability, and strong absorbability [42, 43, 47, 48].

4 Microwave Treatment

In the usual heating methods, heat flows mainly through conduction, convection, and radiation. In this way, the inherent properties of the material and the heating rate affect the temperature change in the material. Fiber burning, non-uniform temperature distribution, chemical damage to fibers, longer curing time, higher thermal gradient, poor efficiency, high energy consumption, waste generation, and expensive equipment are some of the disadvantages related to the conventional thermal techniques used for manufacturing fiber-polymer composites [49].

Since World War II, major advances have been made in the use of microwaves for heating applications. Afterward, it realized that microwaves have the potential to create rapid and high-efficiency heating in materials. Today, the main applications of microwaves include food processing, wood drying, plastic and rubber processing, ceramic baking, preheating, and post-curing. Microwaves broadly include electromagnetic waves in the frequency range of 300 MHz–300 GHz. Household and industrial microwave ovens usually operate at a frequency of 2.45 GHz, a wavelength of 12.2 cm, and an energy of 5–10 $\times$ 2.1 eV [50]. However, all materials can not be heated quickly by microwaves. Materials can be classified into three groups: conductors, insulators, and heat absorbers [51].

Depending on the frequency, dielectric heating can be categorized into two technologies, i.e., radio frequency and microwave technology. Radio frequencies below 100 MHz are generated with open-wire circuits and applied between metallic electrodes. Microwaves are emitted from vacuum tubes direct into the materials through metallic tubes called waveguides and can be generated at 85–94% electrical efficiency [52–54]. Compared to conventional heating in which thermal energy is transferred to the bulk of the material by radiation and/or thermal convection. Microwave heating can transfer thermal energy to the bulk of the material by conduction.

Theoretically, electromagnetic waves consist of two components: electric and magnetic fields. These two fields oscillate vertically relative to each other and are perpendicular to the direction of propagation. The effect of the magnetic field on wood is negligible therefore is not taken into consideration for practical purposes because in dielectric materials magnetic permeability is comparable to the free space. Besides, the effect of the electric field on wood is very high. The high-frequency electromagnetic waves can polarize charges in wood material [55]. A monochromatic electromagnetic wave (Fig. 3) is a sinusoidal wave defined by frequency (f) and wavelength (λw). The wavelength is related to frequency through the speed of propagation (c) as Eq. 1 [56]:

$$c = \lambda \mathrm{w} \cdot f \ (\mathrm{ms}^{-1}) \tag{1}$$

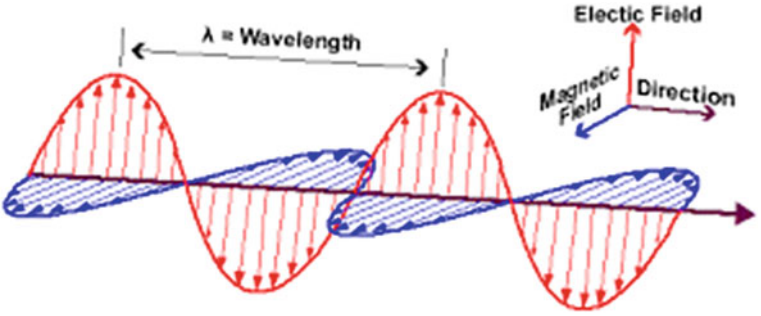

Fig. 3 Monochromatic electromagnetic wave

The interaction of an electromagnetic field with material may cause several responses, and microwaves can be reflected, absorbed, or transmitted. Reflective materials tend to be bulk metals with many free electrons [53]. Transparent materials tend to have low conductivities associated with members of the glass and ceramics family [57]. Absorbing materials consist of all those which exhibit dielectric character [55].

When an electromagnetic field is penetrating a dielectric material, i.e., wood or any natural fibers, the energy is gradually absorbed by the material due to the polarity. The electromagnetic field strength at the surface is therefore decreasing exponentially during the penetration. The attenuation inside the material can be explained by the exponential Eq. 2 [55]:

$$E(Z) = E_0 \cdot \mathrm{e}^{-\alpha z}\left(\mathrm{V\ m^{-1}}\right) \tag{2}$$

where α is the attenuation factor, and z is the distance of the dielectric from the surface. The effect of attenuation and phase shift on the wave is shown in Fig. 4.

The penetration depth (Dp) depends on the dielectric properties of wood, density, moisture content, temperature, grain orientation, etc. The Dp is defined as the thickness of the material when the transmitted power is reduced to $1/e$ of its original value. It means that the material would absorb approximately 63% of the incident electromagnetic power [58, 59]. The penetration depth is given by Eq. 3:

$$\mathrm{Dp} = 1/2 \cdot \alpha(m) \tag{3}$$

Material with large thickness and high loss factor may cause heating occurs only in surface layers. To prevent this phenomenon, proper electromagnetic heating must be chosen so that enough time is provided for a subsequent heat flow between the surface and core layers.

Processing with microwave radiation has a great potential to improve the current methods of drying wood and processing WPCs. The ability of microwave rays to penetrate and directly interact with materials is abundantly used in the food and

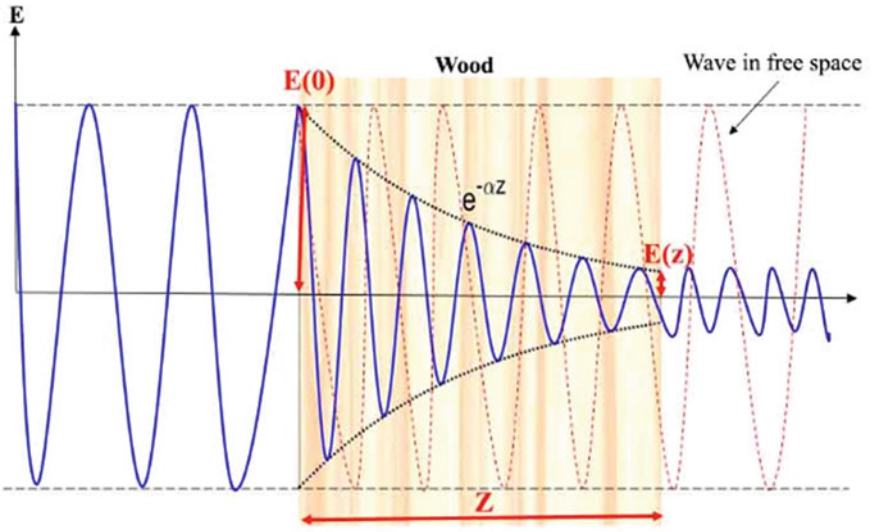

Fig. 4 Wave attenuation: electromagnetic wave transmitted into wood

pharmaceutical industries, and recently in the production of composites in laboratory dimensions. The heating generates from the microwave beam in the composites while reducing the thermal gradient and rapid heat transfer throughout the thickness of the material, thereby improving the production time and productivity [60].

Heating with microwave radiation is volumetric and is not limited to surfaces; thermoplastic polymer materials can be processed faster with microwave beams [61]. Processing with microwaves in composites, by the convective heat transfer method, has several advantages: faster heating, increased adhesion of fibers to the matrix, and more controllability. Heating with microwaves strongly depends on the dielectric properties of the material [62]. The energy of the microwave is directly transferred into the very thin boundary layer of the polymer matrix [63].

5 Wood Drying with Microwave Irradiation

One of the important applications of microwaves is wood drying. Although 6 values of microwave frequencies are used industrially, only two of them (0.922 and 2.54 GHz) are used in heat treatment and wood drying [64]. Microwaves affect wood resistance, moisture content (MC), weight, and thickness of wood species. The temperature of wood during microwave heating depends on several factors such as the power of microwave radiation, time of treatment, dimensions of material, MC, frequency, and wood permeability [65–69].

The main advantage of this method in wooden materials is the rapid heat of the material throughout the whole cross-section due to the dielectric properties of wood

and water [55, 70]. Another advantage is the ability of microwave heating to be used in a continual process, which will be beneficial to flow technology production. The principle behind microwave heating is based on the polar characteristic of molecules and their ability to absorb and transform microwave radiation into heat [55, 71]. Permanent dipoles of molecules begin to move with the same frequency as the electromagnetic field. Therefore, rapid changes in the field polarity cause vibration and rotation of molecules, which transforms the microwave energy into frictional heat [71, 72]. Some researchers investigated the effect of microwaves irradiation on raw wood, as given below.

For example, BinHua et al. [73] summarized the possible applications of microwave technology for the wood industry, like wood drying, curing of adhesives, defect detection of wood, MC measuring, and volatile organic compound (VOC) emission reduction. This technology can be used also for the plasticization of wood [69] and the acceleration of chemical reactions, e.g., microwave acetylation [70, 75, 76]. Research by Mori et al. [77] showed that the surface temperature of wood spices and internal layers of wood by fiber optical temperature sensor reached 90–110 °C and 100–130 °C, respectively, when a microwave power in the range of 0.6–2.4 kW at a frequency of 2.45 GHz and time of treatment of 1–3 min is applied. Based on wood drying, Brodie [78] showed that heat treatment of wood with microwaves reduces the density and MC of wood, therefore resulting in decreasing the cost and energy consumption during the wood drying process.

By drying *Picea Abies* wood through traditional and microwave irradiation methods, Hansson and Antti [71] investigated the wood resistance according to the moisture content, density, and fiber direction. Their results showed that the resistance performance can be controlled by the moisture content, the number of annual rings, weight, thickness, and width of wood species. A reduction in drying time was observed when the microwave method is applied. Employing two types of high and low-intensity microwaves, Balboni et al. [79] studied the effect of microwave treatment on drying defects, density, MOE, MOR, compression strength parallel to the grain, and shear strength of *Eucalyptus Macororhyncha* wood samples. They found that low-density microwaves reflected better impacts on moisture content distribution, and wood density, while MOE and MOR values remained statistically unchanged. He et al. [80] investigated the permeability and drying properties of *Eucalyptus Europhyla* wood samples by controlling the radiation power, radiation time, and initial moisture content. Their results showed that by increasing the power and time of radiation, the wood permeability increased in both longitudinal and transverse sections, followed by decreasing the wood moisture content. Researchers believe that microwaves can accelerate the quality of wood drying having reduced drying time by 65%. By applying microwave irradiation, the change in the internal structure of wood was demonstrated through SEM micrographs. As shown in Fig. 5a–c vessels and ray cells of the eucalyptus were filled with many tyloses. After the microwave treatment was implemented, the tyloses in the vessels were ruptured (Fig. 5d). The shape of the ray cells along the tangential direction of wood remained the same, but the tyloses were considerably reduced followed by minor cracks in the middle of the lamella (see red arrows shown in Fig. 5e). Pit membranes in the radial direction of wood

were seriously damaged after applying microwave irradiation with 15 kW for the 90 s (see red arrows shown in Fig. 5f); this led to increased pathways for the liquids and gases to get in or out of wood during the impregnation or drying process. It was concluded that the microwave modifications led to increasing the wood permeability by up to a factor of 2.3 and a shortened drying time by a maximum of 65%.

Vongpradubchai and Rattanadecho [81] reported that moisture content has a significant effect on temperature development since microwave energy is mainly absorbed by water in the wood. Work by Studhalter et al. [69] showed that green timber reflects the lowest and highest temperature distribution on the surface and internal parts, respectively, when there is a large temperature gradient. Zielonka and Dolowy [68], Antti and Perré [82], Zielonka and Gierlik [83], and Brodie [84] demonstrated that when thick material is heated by microwaves, the highest temperature is located at a distance away from the surface. From a heat transfer point of view, the temperature increases from the surface to the critical points and vice versa, even though it linearly decreases away from the points [68]. From the above-mentioned research works, it is concluded that microwave modification may be an energy-saving process for drying raw wood with a short drying time.

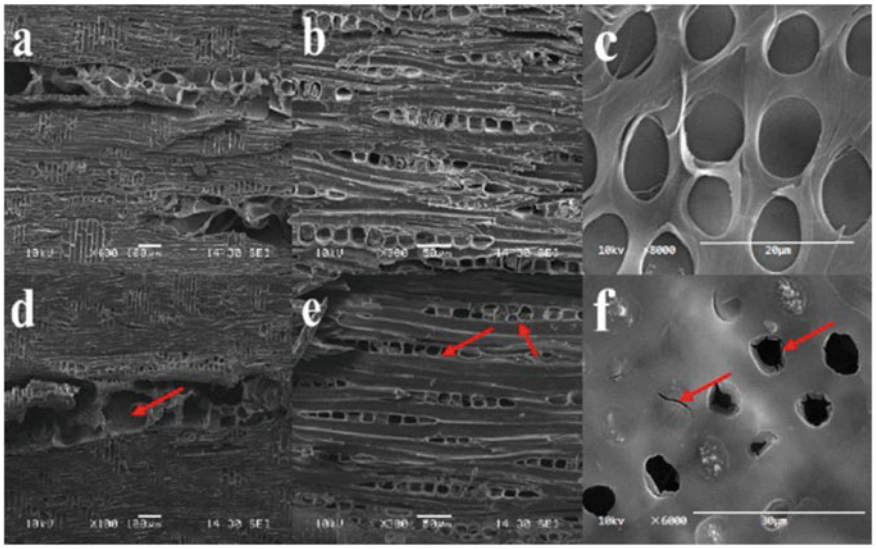

Fig. 5 SEM micrographs of reference and microwave-treated wood; (**a**)–(**c**) are the radial-section, tangential-section, and pits of control wood, respectively; (**d**)–(**f**) are the radial-section, tangential-section, and pits of microwave-treated wood, respectively

6 Microwave Treatment of Wood-Plastic Composites

The motivation behind the use of microwaves in the process of heating biobased composites is the hydrophilicity characteristics of plant-based reinforcements. The interaction between the microwaves and polar water molecules with asymmetric charges present in the plant-based reinforcements results in the partial dissipation of heat energies in the bio-composite materials [85]. The amount of energy dissipated varies depending on the concentration, structure, reinforcement/matrix humidity degrees, temperature, and microwave radiation frequency [86].

Microwaves have been utilized in the development of polymeric compounds for the last three decades [87–90]. Microwave treatment is a potential approach that outperforms conventional techniques in the production and processing of composites [63]. Moreover, microwaves in the form of convection having electromagnetic energy sound more useful compared to thermal energy with heat transfer. Microwaves are not limited to the surface as they are volumetric thus polymeric materials can be processed faster with a microwave [61]. From a heat transfer point of view, energy is transferred because of thermal gradients, while microwave heating is the process of transferring electromagnetic energy to thermal energy and is considered energy conversion rather than heat transfer. Therefore the way that energy is delivered in the microwave heating process could be beneficial to many potential applications related to material processing. Upon the energy concept that explains the generation of heat throughout the volume of the material, microwaves can penetrate materials. As the energy transfer is independent of the heat diffusion from the surfaces, uniform and fast heating of thick material can be achieved via microwaves [91]. Microwaves propagate through space at light speed and, usually, the frequency ranges swap from 300 MHz to 300 GHz [92].

Fast heating, fiber-matrix adhesion, and higher controllability are some of the advantages of microwave processing using convective microwave methods. Microwave heating strongly depends on the dielectric characteristics of the polymers. The transfer of the energy of microwaves into narrow materials takes place at the boundary layers [90, 93, 94]. This offers multiple advantages in the material processing of ceramics, metals, and composites in regard to conventional heating methods, including unique microstructure and properties, improved product yield, energy savings, and reduction in manufacturing cost and synthesis of new materials [95]. The microwaves do not interact with the majority of polymeric materials due to the lack of dipolar moment. Thus, additives acting as heating susceptors can be used to prepare materials capable of absorbing microwaves [92]. Some of the research in this regard is given below.

Erchiqui et al. [86] studied the thermoforming of PP/WF composite by employing microwave and infrared radiation heating sources. The WPC samples treated by microwave radiation reflected a uniform temperature distribution in comparison with infrared radiation treatment. The time duration of microwave heating has a direct relation with the reinforcement concentration and the sample size. Another new method of using microwaves on WPCs was done by Tewari et al. [14]. They used

the microwave-assisted compression molding (MACA) method to make WPCs, and at the same time, they used alkaline chemical treatment of pine cones to improve the bonding. The properties of composites were controlled through physical, mechanical, and thermal tests. Their results indicated a significant effect of the MACA method in the strengthening of WPCs. They also showed that the dielectric constant of pine cone filler is 1.6 while the dielectric constant of rHDPE at 2.45 GHz is 2.3. Having the complex permittivity of the material during microwave interaction (ε^*), dielectric constant ($\varepsilon^{'}$) as the energy absorbed by the material, and dielectric loss factor ($\varepsilon^{\varepsilon}$) as the ability of the material in dissipating microwave energy, one can calculate the dielectric response of materials (representing the interaction between microwave and the nonmagnetic material), as shown in Eq. 4:

$$\varepsilon^* = \varepsilon' - j\varepsilon^{\varepsilon} \tag{4}$$

Dielectric loss tangent or energy dissipation factor (δ) is a function of $\varepsilon^{'}$ and $\varepsilon^{\varepsilon}$ via Eq. 5:

$$\tan\delta = \varepsilon^{\varepsilon}.(\varepsilon^{-1}) \tag{5}$$

Dipole polarization is represented as a parameter contributing to the dielectric response which is the dominating energy transmission mechanism in nonmagnetic materials at the molecular scale such as polymeric composites, ceramics, etc. [96]. A pine cone filler having a higher dielectric constant can be heated by interacting with the microwave. This led to heat transmission to the rHDPE and adjacent pine cone via conduction, resulting in melting rHDPE pellets and consequently bond formation between the pine cone and matrix. Next, the mixed melted rHDPE and pine cone filler were exposed to room temperature to get cool down. Erchiqui et al. [86] conducted a numerical study on the potential of microwave radiation with 140 °C heating temperature for thermoforming WPCs. They also compared infrared and microwave heating modes on pine cone/polypropylene composite sheets and control polypropylene. Their results showed that microwave irradiation creates somehow uniform temperature distribution in the sheets compared to the infrared method.

7 Post-treatment of WPCs by Microwave Irradiation: Case Study Outline

Another application of microwave irradiation is to modify the characteristics of composites after their production process. Some researchers have addressed this issue in recent years. For example, the work by Yuan et al. [97] showed the potential of microwave irradiation to modify carbon fibers immersed in water. Their results indicated that a large number of oxygen-containing groups which are exposed to exfoliation of the surface layers were placed on the carbon fibers when microwave

irradiation is applied. Although the tensile strength of carbon fibers was slightly lower, the shear strength of carbon fibers in epoxy resin composite significantly increased. Work by Zhang et al. [94] reported the fabrication of PES/pine wood powder composites using selective laser sintering (SLS) with a layer thickness of 0.1 mm, preheat temperature of 83 °C, and internal power of 13 W. A CNT was utilized as an additional reinforcement aimed to improve the mechanical and physical properties of the produced WPCs. Next, a microwave oven at 385 W power and 2.45 GHz frequency was used for WPCs post-treatment. The 10 s was an optimal treatment time to produce samples with the highest bending strength as high as 15.7 Mpa, while microwave treatment for less than 60 s improved the bending strength by 64.2%.

Nightingale [98] compared autoclaved composites with microwave post-cured composites and fully microwave-cured composites by performing physical and mechanical tests. The bending test results showed that the post-cured composites by the microwave possessed the lowest bending strength compared to the composites cured by the normal autoclave. Boey and Lee [99] and Yue and Boey [100] reported that the elastic modulus of microwave-cured composites is higher compared to the conventional thermal method, however they have lower tensile strength. Comparing the mechanical behavior of microwave-cured epoxy resin composites over thermal curing, Bai et al. [101] concluded that microwave-cured resin showed slightly higher strength and tensile modulus due to greater homogeneity and proper temperature distribution. The effects of microwave post-curing and traditional autoclave heating methods on the mechanical and microstructure properties of composites were investigated by Adeodu et al. [102]. They found that there is a direct relationship between tensile strength and the weight percentage of fillers according to post-curing methods. Also, the composites produced by the microwave post-curing method showed fewer defects compared to the composites produced by the autoclave method. Moreover, the microwave post-curing heating methods improved the strength of the microstructure of composites compared to autoclave heating. Zlobina et al. [33] used microwave modification to improve the resistance properties of the composite incorporated with carbon and glass fibers for aviation applications. The effect of the microwave beam on the composite at a specific power level increased the bending strength. At the same time, the microwave beam caused significant changes in the microstructure of the composite. They realized that the homogeneity and density of composites increased since microwave treatment was implemented. Chavooshi et al. [103] investigated the effect of microwave radiation heat treatment on the mechanical and morphological properties of polypropylene/MDF[4] powder nanocomposites. They used nano clay and MAPP as coupling agents to improve the bonding and properties of the composite. Their results showed that the mechanical properties of samples treated with microwave radiation were better than other samples. From FE-SEM for the nanocomposites treated with microwave, they observed that MDF powder is more optimally mixed with the polymer matrix, thus the encapsulation of MDF powder by the polymer is better.

[4] Medium Density Fiberboard.

Owing to the importance of microwave treatment in the improvement of the mechanical properties of the composite, the following section aims to fully explain our research recently conducted regarding microwave heating on WPC by injection molding method. The purpose of this research is to investigate the improvement of the mechanical, physical, and morphological properties of composites treated with microwave radiation in the presence of nano-silica as an accelerator of microwave absorbance. Recent advances in nanotechnology provide a source of excellent microwave-absorbent materials due to their exceptionally high dielectric constant which allows for absorbed electromagnetic energy to be dissipated into heat [104]. Wood flour, high-density polyethylene (HDPE), and Nanao-SiO_2 (i.e., 0, 1, 2, and 3 wt%) were used as nanomaterials to fabricate WPCs. When the samples were fabricated, they were exposed to 900 W microwave radiation for 7 min before performing the physical and mechanical tests.

7.1 Materials and Method

HDPE matrix with the specification of 18 g melting flow index in 10 min and 190 °C melting point supplied from Tabriz Petrochemical Co., Iran. The 0.8% Maleic anhydride grafted with polyethylene (MAPE) was employed as a coupling agent with 0.4 g melting flow index in 10 min which was supplied from Merck Co., Germany.

In this study, the wood flour was supplied by a local factory based in the North of Iran and was further pulverized by a lab-scale grinder. The produced powder was used as the filling fiber by passing through 60 and 80-mesh sieves. SiO_2 nano-sized particles (with an average diameter of 12 nm, a density of 0.37 g/cm^3, an apparent ratio of 208, and a specific surface area of 200 m^2/gr) supplied by Evonik Degussa Co., Germany, were utilized as reinforcement. The XRD pattern of Nanao-SiO_2 used in this study with the 2θ of 23.2° is shown in Fig. 6. The polymer and SiO_2 nanoparticles were used right after receiving them. An air-circulating oven was used to dry the lignocellulose materials at 100 °C for 24 h to reach a moisture content of <1 wt% before their use.

7.2 Preparing the Composites

The prepared composites are given in Table 2. The concentration of SiO_2 nanoparticles varied from 0, 1, 2, and 3 wt% at a constant 50% WF for all the samples.

The mixing phase was implemented at 150, 155, 155, 155, 150, and 145 °C and 60 rpm with 1500 MPa die pressure in a counter-rotating twin screw extruder (Brabender® Plasti-Corder®). Afterward, the sample was grounded using WIESER WGLS200/200 Model semi-industrial crusher and then transferred to the injection molding machine. Five test samples were prepared for each treatment at 160 °C and 45 rpm loading speed, 80 bar injection pressure, 75 s cooling time, and 200 bar

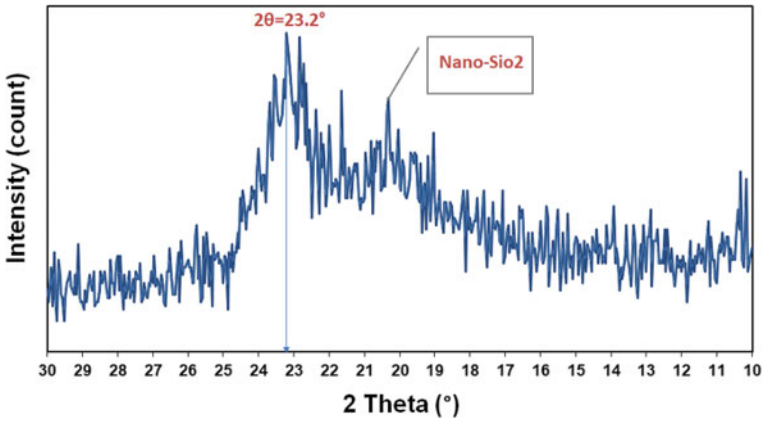

Fig. 6 X-ray diffraction pattern of the nanao-SiO_2

Table 2 Composition of evaluated formulations

Number	Composite formula	HDPE (wt%)	Wood flour (wt%)	SiO_2 nanoparticles (wt%)	MAPE (wt%)
1	47HDPE/50WF/0NS/3 M	47	50	0	3
2	46HDPE/50WF/1NS/3 M	46	50	1	3
3	45HDPE/50WF/2NS/3 M	45	50	2	3
4	44HDPE/50WF/3NS/3 M	44	50	3	3
5	47HDPE/50WF/0NS/3 M*	47	50	0	3
6	46HDPE/50WF/1NS/3 M*	46	50	1	3
7	45HDPE/50WF/2NS/3 M*	45	50	2	3
8	44HDPE/50WF/3NS/3 M*	44	50	3	3

* Treated with microwave irradiation

loading pressure. The samples were restrained minimum of 40 h in an environment with 23 °C temperature and 50% relative humidity followed by the 99-618ASTM standard. A 900 W microwave oven (NN-CD997S model, Panasonic Co.) was utilized for the samples heat treatment (Fig. 7). Five, seven, and ten minutes of irradiation were considered to find an optimal microwave irradiation period. Controlling the color and morphology of the samples after irradiation and following Chavooshi et al. [103], 7 min of microwave irradiation was chosen as an optimal period of irradiation.

Five samples from each combination were selected and dried in an oven for 24 h at 100 ± 3 °C to control the water absorption and thickness swelling of the samples following the ASTM D7031-04 standard. Measuring the weight and thickness of the dried samples with an accuracy of 0.001 g and 0.001 mm, respectively, the samples then were immersed in distilled water at a temperature of 20 ± 2 °C for 24 h. The thickness and weight properties of the samples were measured after rinsing the

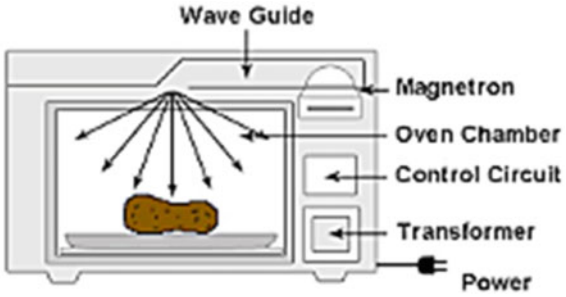

Fig. 7 Image of microwave device and its components

samples with an excessive amount of water on their surfaces. The following equation (Eq. 6) calculates the percentage of the water absorption of the samples:

$$\mathrm{WA}(t) = \frac{W(t) - W_0}{W_0} \times 100 \tag{6}$$

where WA (t), W_0, and $W(t)$ denote the water absorption at time t, oven-dried weight, and weight of the sample at immersion time t, respectively.

Also, the percentage of the thickness swelling is calculated via Eq. 7.

$$\mathrm{TS}(t) = \frac{T(t) - T0}{T0} \times 100 \tag{7}$$

where TS(t), T_0, and $T(t)$ are the thickness swelling at time t, and the initial thickness and thickness of the samples at time t, respectively.

Flexural strength and modulus were taken following standard ASTM D747 using the UK Instron 6025 model. At a crosshead speed of 5 mm/min, the tests were implemented. An Izod impact strength test was performed following ASTM D-256 using Zwick 5102 model machine.

The structure of the composites is controlled by a scanning electron microscope (SEM) (LEO Oxford Co.). The fracture surfaces of the specimens were sputter-coated with gold after conducting the impact test right before the analysis is initiated. All images were taken at an accelerating voltage of 26 kV.

7.3 Results and Discussion

Figure 8a, b show the 48 h water absorption and thickness swelling of composites comprising wood flour, SiO_2 nanoparticles, and HDPE in two states of untreated and microwave-treated, respectively. Based on the 50% constant amount of wood fiber used in all compositions, the water absorption and thickness swelling properties of the tests show different behavior due to the presence of SiO_2 nanoparticles and the microwave treatment. The composites in the absence of SiO_2 nanoparticles reflect

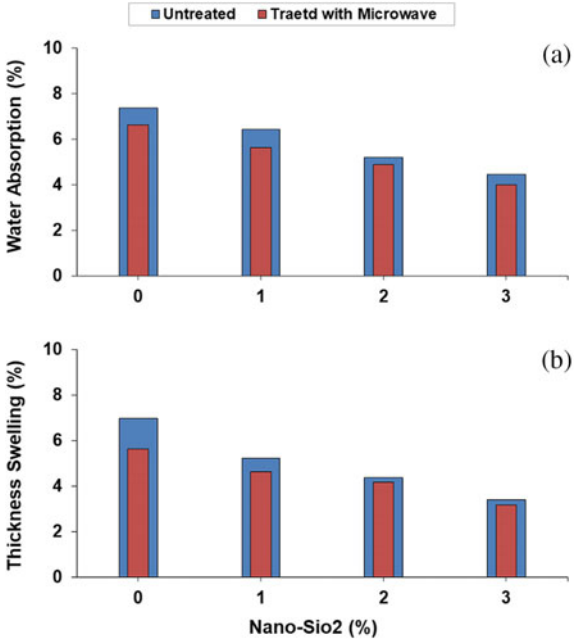

Fig. 8 Effect of SiO_2 nanoparticles and microwave irradiation on water absorption and thickness swelling of WPCs

the highest adsorption and swelling properties compared to the samples filled with SiO_2 nanoparticles.

The formation of hydrogen bonding between SiO_2 nanoparticles and the hydroxyl groups of wood flour (Fig. 9) reduces active sites for water molecules to form hydrogen bonds with hydroxyl groups. In other words, there is a kind of competition between the hydroxyl groups of wood flour and the hydroxyl groups of SiO_2 nanoparticles to form hydrogen bonds. Therefore, the bond between wood flour and SiO_2 nanoparticles reduces the chances for water molecules to form hydrogen bonds in this competition.

Chemically, according to Eq. 8, SiO_2 nanoparticles have hydroxyl groups, hydrogens attached to hydroxy groups, and siloxane groups. The first two compounds are hydrophilic and the latter is hydrophobic. Therefore, it suggests that the SiO_2 nanoparticle is polar and hydrophilic. Although SiO_2 nanoparticle has the moisture adsorption property because of its hydroxyl groups, when they are in contact with wood flour to form hydrogen bonds, the hydroxyl groups of both materials are practically unavailable, leading to the reduced water absorption of the composites [105].

$$\begin{aligned} &Si(OC_2H_5)_4 + H_2O \rightarrow Si(OC_2H_5)_3OH \\ &+ C_2H_5OH \rightarrow \equiv Si - O - Si \equiv + H_2O \equiv Si - O - H \\ &+ H - O - Si \equiv \rightarrow Si - O - Si \equiv + C_2H_5OH \equiv Si - OC_2H_5 \\ &+ H - O - Si \equiv \end{aligned} \tag{8}$$

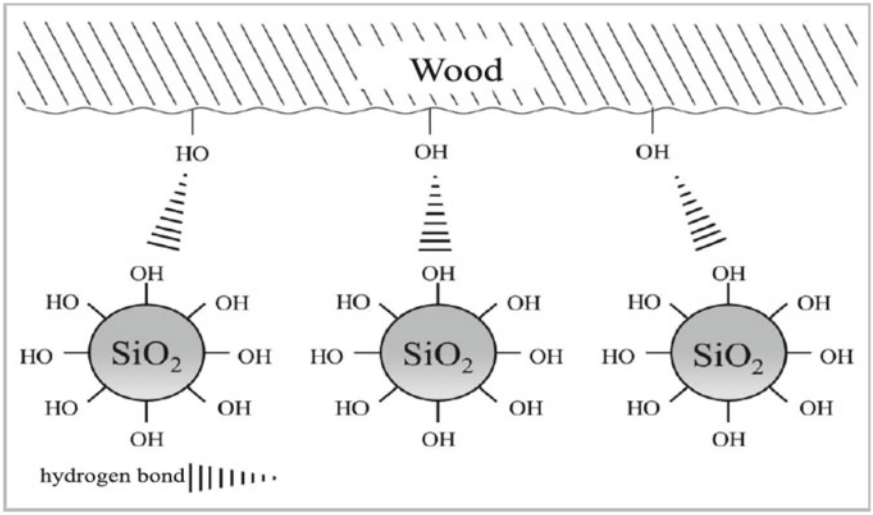

Fig. 9 The scheme of linking the hydrophilic groups of nano-silica with the hydroxyl groups of wood flour

According to Espert et al. [106], water absorption in natural fiber-plastic composites could be obtained through moisture absorption by either the cell wall of the lignocellulosic material or the capillary process through the existing voids gaps between the plastic and natural fibers. The former is because of the inherent characteristics of cellulose fibers and the latter is due to the weak interaction between wood fibers and plastic and the formation of possible defects during its production process. Among the samples treated with microwave radiation, the lowest amount of thickness swelling is related to the samples containing 3 wt% SiO_2 nanoparticle, which is due to the small dimensions of nano-silica particles which can fill the empty spaces in the matrix, thereby blocking a large number of capillary tubes [107]. Therefore, the presence of nano compounds inside the empty spaces and their interactions with water to form hydrogen bonds reduces the water absorption of the composites. This is in agreement with the research by Ismaeilimoghadm et al. [107] and Hosseini et al. [108]. Figure 8a, b also show that the water absorption and thickness swelling decrease further by performing the microwave treatment. The microwave radiation contributes to softening again the thermoplastic polymer material and thus the cellulose fibers are better enclosed and encapsulated by the matrix [103]. This leads to blocking the porosity of the matrix and the pores of the capillary tubes of cellulose fibers which are the potential spots for water penetration. In Fig. 8a, it is found that the microwave-treated composites with 3 wt% SiO_2 nanoparticles and 4% water absorptivity show a 66% absorption reduction in comparison to treated samples with no SiO_2 involved.

The average values of flexural strength and modulus of WPCs containing different amounts of SiO_2 nanoparticles in two states of untreated and treated with microwave

radiation are presented in Fig. 10a, b, respectively. Figure 10b shows the increase in the flexural modulus by adding SiO_2 particles. Figure 10b shows the highest flexural modulus of 116.86 MPa for the composites containing 3 wt% nano-silica treated by microwave radiation. While the lowest flexural modulus belongs to the microwave-treated samples without SiO_2 nanoparticles (control sample) with a value of 769.84 MPa. The modulus of elasticity of composites depends on several parameters such as the amount of fiber used, the orientation of fibers, the connection of fibers and the polymer matrix in the interphase region, the modulus of elasticity of the components, and its density [45]. According to the nano-silica information, the elasticity of SiO_2 nanoparticles has been reported between 66.3 and 74.8 GPa. Therefore, the replacement of wood fibers with an elasticity modulus of about 5 to 8 GPa with SiO_2 nanoparticles can have a significant effect on the flexural modulus of the composites. Moreover, the higher ratio of length to diameter (L/d) of the SiO_2 nanoparticles used in this research plays an important role in the increase of the flexural modulus of WPCs. These results are consistent with the research of Nourbakhsh et al. [45]. Also, by using a microwave treatment, the flexural modulus increased from 685.72 to 1116.86 MPa compared to the control sample. These results are in agreement with Zlobina et al. [33]. Another reason for the connection improvement between the fibers and the polymeric matrix, especially in composites reinforced with natural fibers, is the development of curing the matrix during post-treatment with a microwave beam which is heat transferred from the fiber surface to the internal volume of the composite [109]. In the curing process by the microwave beam, wood fibers and SiO_2 nanoparticles, because of their high dielectric constant [104, 109], absorb a large amount of microwave power in the form of heat which is transferred from the outer surface of the fibers to the volume of the polymer matrix. This leads to the formation of a stress-free interface area around the fibers [110, 111]. With microwave beam treatment, the flexural modulus of the composites containing 1, 2, and 3 wt% SiO_2 nanoparticles are 29.3, 44, and 62.87%, respectively, which are greater than that of the control sample (Fig. 10b). The elasticity modulus of 17.65, 6.14, and 10.93% are also observed for the samples containing 1, 2, and 3 wt% SiO_2 nanoparticles, respectively, which are higher than that of the untreated samples. In connection with the effect of nano compounds on the performance of microwave waves, research by Chavooshi et al. [103] showed that these waves together with nano clay particles improved the mechanical properties of composites. On the other hand, nanomaterials are excellent absorbents for microwaves beam and due to their high dielectric constant, they have the possibility of absorbing electromagnetic energy and of dispersing net energy in the material as heat [104]. The results of this research are confirmed by Zhang et al. [94]. In this regard, the positive effect of SiO_2 nanoparticles and microwave treatment on flexural strength is shown in Fig. 10a. As seen in Figure, by adding 3 wt% SiO_2 nanoparticles, the flexural strength increased from 19.13 MPa in the control samples to 26.86 MPa in the composites indicating a 40.4% improvement in flexural strength. By using microwave treatment, composites containing 1, 2, and 3% of SiO_2 nanoparticles show, respectively, 9.38, 19, and 29.1% improved flexural strength. The flexural strength of composites depends on the bond between

their components. The increase in the flexural strength by applying microwave treatment is due to the reduction of the polymer's viscosity with better wetting for the fibers. As a result, the adhesion and resistance in the interphase region between the fibers and the polymer improve [103]. These results are in line with the research by Chavooshi et al. [103]. Due to very small dimensions, mechanical strength, and high length-to-diameter ratio, nano-silica can create strong adhesion in the interphase region with the composite components. The results are consistent with Nourbakhsh et al. [45] and Farsi [48]. Qualitatively, the composites incorporated with 3 wt% SiO_2 nanoparticles have the highest elasticity modulus and strength with the respective values of 1116.86 and 25.38 MPa in the presence of a microwave beam.

Figure 11 shows the un-notch impact strength in the composites. From Fig. 11, it is obvious that, in general, there is a negligible difference between the impact resistance in the samples with and without microwave treatment, suggesting a lower impact resistance in the composites treated with a microwave beam. The effect of the amount of SiO_2 nanoparticles on the impact resistance of microwave-treated composites shows that the samples without SiO_2 nanoparticles have more resistance compared to the samples containing SiO_2 nanoparticles, which is probably due to the presence of nano-silica particles and the formation of energy absorption points [45, 48]. Works by Farsi [48] and Hosseini et al. [108] showed different impact resistance effects in the composites since nano-silica is added. The samples treated with microwave irradiation containing 1, 2, and 3 wt% SiO_2 nanoparticles reflected 3.9%, 6.03%, and 9.2% impact resistances, respectively, which are less than that of the control samples. By applying microwave treatment and increasing the amount of

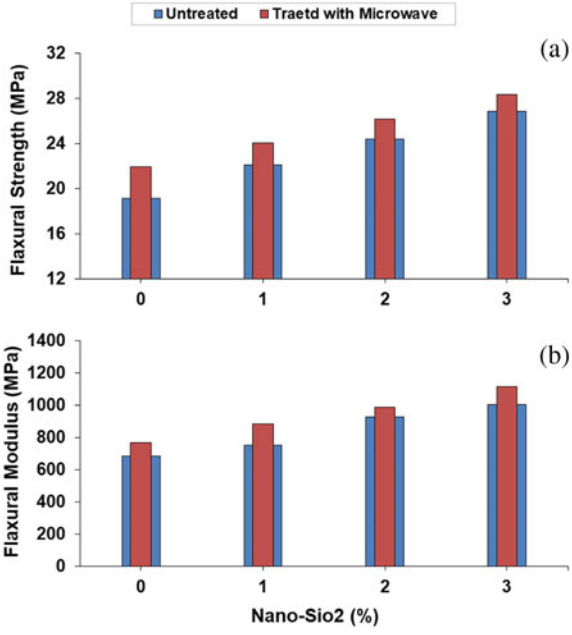

Fig. 10 Flexural strength and modulus of the WPCs as a function of SiO_2 nanoparticles and microwave irradiation

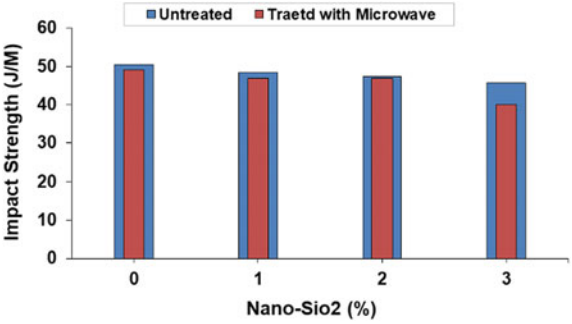

Fig. 11 Impact strength of the WPCs as a function of SiO_2 nanoparticles and microwave irradiation

SiO_2 nanoparticles from 1 to 3%, the impact resistance of the composites was reduced by 4.3%, 4.3%, and 18.5%, respectively, in comparison to the control samples and those treated with microwave radiation, which is not consistent with Chavooshi et al. [103]. The un-notch impact resistance shows the resistance of the material against breaking and cracking, suggesting regular cracks occurring at the weakest parts of the composite in the interface between the lignocellulosic material and the matrix. The excessive increase of the SiO_2 nanoparticles in the composites can also be considered as the parts with energy absorption and crack development [48]. Therefore, with the increase of SiO_2 nanoparticles, the composite impact resistance decreases. These results have been confirmed by Hosseini et al. [108]. Also, the results of the impact test show that the composite without SiO_2 nanoparticles and treated with a microwave beam has the highest impact resistance.

Scanning Electron Microscope (SEM) images are suitable tools for justifying the physical and mechanical properties of composites. There is always a relationship between the properties of composite materials and their internal structure. Figure 12a is related to the samples containing 3% SiO_2 nanoparticles without microwave treatment, by 10 KX magnification. As it is noticeable, the matrix is dispersed in failure points showing frequent SiO_2 nanoparticle particles. In some parts, the wood fibers are separated from the matrix, and instead, empty holes (shown as a red arrow) are seen depicting the poor adhesion between the fiber and the matrix at these points. As seen in Fig. 10a, b, which are related to flexural strength and modulus, compared to untreated samples, the microwave-treated sample has lower resistance with higher water absorption. Figure 12b shows the composite containing 2% SiO_2 nanoparticles and treated with a microwave beam with 10 KX magnification. As it can be seen, by performing microwave treatment, the surface of the sample became more uniform and the matrix completely encapsulated the wood fibers, indicating the positive effect of microwave treatment on the reduction of the viscosity of the polymer matrix and the better embrace of the wood fibers and consequently the improvement of the resistance [94]. As shown in Fig. 12b (SEM photomicrograph b) the number of pores on the surface of composites was reduced. At the same time, the water absorption of the sample decreased. Such an issue is more evident in Fig. 12c with a magnification of 10 KX, as can be seen, the surface of the fibers gets covered by the polymeric matrix which is treated and melted by microwave treatment. This led to covering

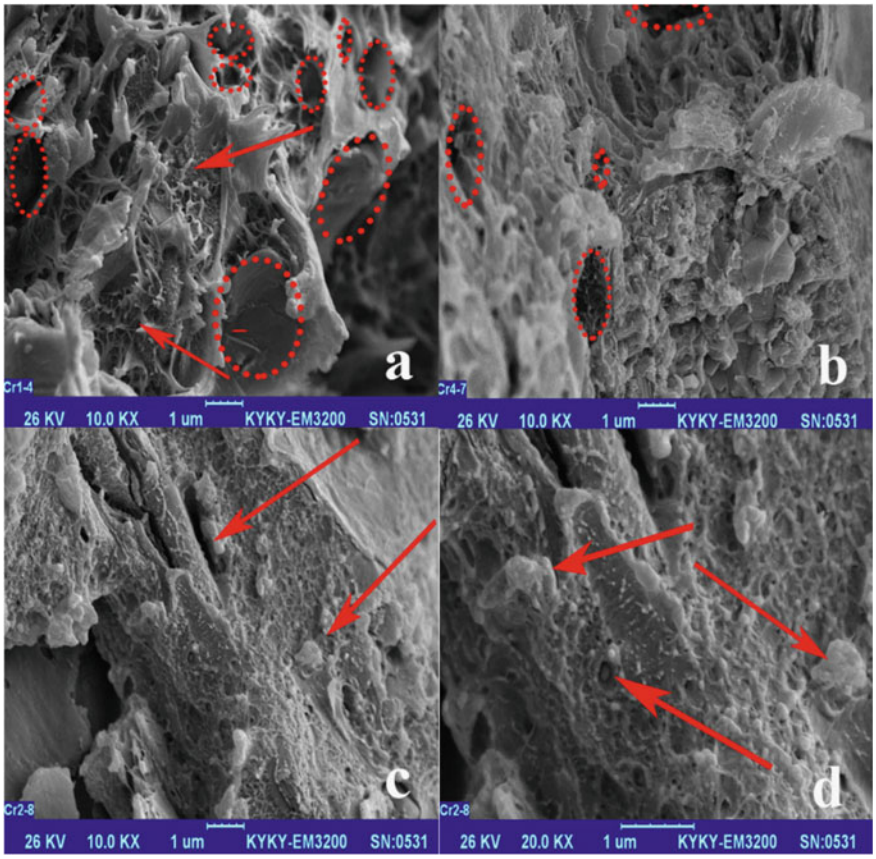

Fig. 12 SEM photomicrographs of fractured samples of nanocomposites: **a** 3% SiO_2 nanoparticles and no microwave, 10000X, **b** 2% SiO_2 nanoparticles and with microwave, 10000X, **c** 3% SiO_2 nanoparticles and with microwave, 10000X, **d** 3% SiO_2 nanoparticles and with microwave, 20000X

and disappearing the holes and pores in the matrix surface. Also, the increase in the amount of SiO_2 nanoparticles from 2 to 3 wt% in Fig. 12d with 20 KX magnification indicates the excessive accumulation of nano-silica on the surface, which forms energy absorption and crack development points (see red arrows shown in Fig. 12d), thus reduced impact resistance.

7.4 Conclusion

In this research, the effect of simultaneous use of post-treatment with microwave irradiation and different amounts of nano-silica on the physical and mechanical

properties of WPCs was investigated. Based on the obtained results, the following conclusions were reached:

1. By increasing SiO_2 nanoparticles up to 3 wt%, the flexural strength, and modulus of WPCs improved significantly.
2. The un-notched impact resistance of WPCs slightly decreased with the increase of SiO_2 nanoparticles up to 3 wt%. Adding different levels of SiO_2 nanoparticles to WPCS has led to different positive and negative results in the composites.
3. The use of SiO_2 nanoparticles reduces water absorption and the thickness swelling of WPCs, and with the increase of SiO_2 nanoparticles, this reduction increases.
4. The post-treatment of WPCs with microwave irradiation increases the flexural strength and modulus and decreases the impact resistance, water absorption, and thickness swelling of composites. SEM images indicate the microstructure surface improvement of the composites with microwave treatment. By applying microwave treatment, the surface of the composite is more uniform, and the fillers are better encapsulated by the polymer.
5. The general results indicate the proper effect of SiO_2 nanoparticles and microwave treatment on WPCs. Based on this, the highest amount of flexural strength and modulus, and the lowest amount of water absorption and thickness swelling are related to WPCs containing 3% of SiO_2 nanoparticles and treated with microwave radiation. The highest impact resistance belongs to composites without SiO_2 nanoparticles and treated with a microwave beam. The SEM images of the samples also confirm these results.

8 Summary

This chapter describes the basic, important, and practical concepts of applying microwave irradiation to wood and wood waste products. By reviewing extensive works of literature relevant to the field, a detailed discussion of wood drying and WPCs properties is provided. It was found that microwave treatment greatly influences the properties of wood waste products. It is suggested that new or innovative microwave treatments and methods are demanded to meet the practical application of wood and wood waste products in industries. There is still a lack of experimental investigations evaluating the impact of microwave treatment on wood waste products' resistance to biological agents, fire, environmental conditions, and so on. Finally, the authors discussed their findings for microwave irradiation post-treatment of WPCs by assessing their mechanical, physical, and morphological characteristics. They found that the microwave plays a significant role in the fabrication and wood drying of wood-based composites.

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