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# A REVIEW OF MICROWAVE CURING TECHNIQUE TO FABRICATE NATURAL FIBER REINFORCED POLYMER COMPOSITES

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### **Graphical abstract**

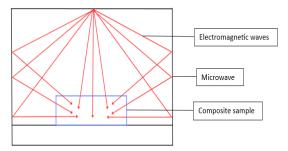


Figure 1 Electromagnetic waves of microwave curing

### **Abstract**

Advanced sustainable materials with high performance are garnering a lot of attention nowadays due to rising environmental concerns. Natural fiber reinforced polymer (NFRP) composites are currently one of the huge demands to replace conventional materials as it inhibits great properties, low cost, abundantly available, and environmentally friendly. There are various ways to fabricate NFRP composites such as compression molding, resin transfer molding, etc. However, these methods have several disadvantages (e.g., non-uniform temperature distribution, longer processing time, and high cost). This paper reviews the current trend of utilizing microwave curing in fabricating NFRP as it provides volumetric heating, less power consumption, and is more efficient.

Keywords: Natural fiber reinforced polymer composite; chemical treatment; microwave curing; thermoset; thermoplastic

#### **Abstrak**

Bahan mampan yang berkualiti tinggi mendapat banyak perhatian pada masa kini disebabkan oleh peningkatan kebimbangan alam sekitar. Polimer bertetulang gentian semula jadi komposit kini merupakan salah satu permintaan besar untuk menggantikan bahan konvensional kerana ia mempunyai sifat yang berkualiti, kos rendah, tersedia dengan banyak dan mesra alam. Terdapat pelbagai cara untuk membuat komposit polimer bertetulang gentian semulajadi seperti pengacuan mampatan, pengacuan pemindahan resin, dan lain-lain. Walau bagaimanapun, kaedah ini mempunyai beberapa kelemahan seperti taburan suhu yang tidak seragam, masa pemprosesan yang lama dan kos yang tinggi. Kertas kerja ini mengkaji trend semasa menggunakan pengawetan gelombang mikro dalam fabrikasi polimer bertetulang gentian semulajadi kerana ia menyediakan pemanasan isipadu, penggunaan kuasa yang kurang dan lebih cekap.

Kata kunci: Polimer bertetulang gentian semulajadi; rawatan kimia; pengawetan gelombang mikro; termoset, termoplastik

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### 1.0 INTRODUCTION

Over the past few decades, there has been a significant increase in the need for advanced sustainable materials to replace conventional materials for incorporation into human life in the form of variety of products. Synthetic fibers were generally used as reinforcement in conventional materials as they exhibit great mechanical properties. However, synthetic fibers are high in density, non-renewable, non-biodegradable, and have high energy consumption during production [1]. Hence. advanced sustainable materials have provided researchers and engineers with an abundance of high-performance, opportunities to create environmental-friendly composites that reduce fuel consumption, emissions, and combat climate change. Advanced sustainable materials such as natural fiber reinforced polymer (NFRP) composites are currently a famously investigated topic due to their less environmental impact, energy consumption during fabrication, and recyclability [2-5]. The use of natural fibers, like bamboo, kenaf, hemp, sisal, and banana for polymer composites reinforcement are significant in various industries such as automotive, construction, aerospace, electronics, etc [6-7]. The advantages of natural fibers are the availability of a variety of fibers, low cost, and density, lightweight, renewability, biodegradability, and environmentally friendly. However, these fibers have some drawbacks i.e., poor compatibility and wettability, sensitivity to high temperature, and moisture absorption which will affect the properties and performance of the composites. These drawbacks can be eliminated by treatments after extraction [1-8].

There were broad studies on NFRP composites that have already been investigated based on their manufacturing techniques. For instance, hand layup, compression molding, injection molding, etc. are common techniques used to produce these materials [9-10]. Different NFRP composites requires different manufacturing techniques, processing conditions, and tools. The processing conditions including rate and temperature affect the quality of adhesively bonded composites [10]. In some studies, after the composites are manufactured, they undergo a post-curing process to improve their mechanical and thermal properties and diminish any residual stresses that may be present [11-13]. In conventional traditional heatina processina techniques, the heating rate affects the temperature change and properties of the material. Hence, there is a high chance that the material will be overheated and degrade the natural fiber [14]. When natural fiber is exposed to high temperatures, it affects its microstructure and mechanical properties. Besides that, it will take longer for the material to cool down and will eventually cause the surface of the material to be harsh. Thus, one approach to avoid low-quality materials is by utilizing microwave-assisted processing of materials [14-15].

Researchers have recently interested in studying the ideal processing parameters to produce high quality of composites by utilizing microwave-assisted curing. According to Naik, Singh and Sharma (2022), microwave curing has been employed in the creation of thermoset polymer composites since 1980 [13]. Microwave curing of composites has more benefits than just saving energy and time during production. It also has lower running costs, better for the environment, and heats a material from the inside out [16]. The focus of study these days is on microwave curing thermoplastic composites. This review focuses on analyzing the effects of microwave curing to manufacture NFRP composites.

### 2.0 NATURAL FIBER REINFORCED POLYMER COMPOSITE

NFRP composites have recently drawn a lot of interest from material researchers because of their excellent mechanical properties, low cost, and environmental friendliness. Due to the growing environmental concern relating to global warming and pollution, biological and recyclable materials undoubtedly meet the future sustainable design criteria [17]. Extensive studies were done on natural fibers to see if they could replace synthetic fibers, and the results showed that they could be a promising reinforcing agent for polymer matrix composites. In comparison to synthetic fibers, natural fibers have various advantages such as being lightweight, low in density, cheaper, renewable, and biodegradible [1-8,18-19]. Natural fibers are also abundantly available in certain countries like India [20]. Azwa et al. reported that the demand for natural fibers in industrial in the US alone has increased by 60% each year with an increment of 10% to 20% every year due to the need of less energy during the production of NFRP composites [21].

Despite their significant values, natural fibers are not strong enough to be used on their own, and they can easily absorb moisture in either humid environment or immersed in water. This causes incompatibility between the hydrophilicity of fibers and the hydrophobic behavior of polymer matrix [2-8,19]. The hydrophilic property of the natural fiber is due to the presence of hydroxyl groups in the cellulose which will lead to poor adhesion with polymer matrix, and poor mechanical, physical, and thermal properties of a composite. Natural fibers also tend to degrade easily at high temperatures (>200°C). These drawbacks can be improved by interfacial treatments such as chemical treatments and coatings [18-24].

Merlini, Soldi, and Barra (2011) investigate the effect of 10% sodium hydroxide (NaOH) treatment on banana fibers. It was observed that the treated banana fiber composite had better tensile strength than the untreated composite due to their

enhanced interfacial bonding between fiber and matrix. Also, the scanning electron micrographs (SEM) showed there was no presence of fiber pull-out from the treated composites compared to the untreated ones [25]. Cai et al. (2016) studied the mechanical properties of chemically treated abaca fibers. The abaca fibers were treated with 5, 10, and 15 wt% of NaOH solutions for 2 hours. In comparison to untreated abaca fiber reinforced composite, the results showed an increase in 5wt% of treated abaca fibers' tensile strength of 8% and Young's modulus of 35% [26].

Wong, Youslf, and Low (2016) carried out a study on the effects of NaOH solution chemical treatment of bamboo fibers on the interfacial bonding between polymer matrix and concluded that 5 wt% of treated bamboo fibers had rougher surfaces due to the removal of unwanted substances on the fiber surface. This results in the composite having lower density, improved interfacial bonding, and better mechanical properties than the untreated fiber composite [27]. Guo, Sun, and Satyavolu (2019) reported that alkaline treatment removed hemicellulose, lignin, and impurities while increasing the cellulose content of the natural fibers. As a result, the mechanical properties of the treated fibers were improved [28].

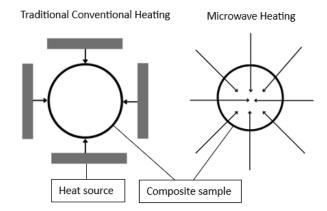
Pradeepa and Kiruthika (2022) examined the influence of 5 wt% of NaOH chemically treated jute fibers reinforced sodium alginate composites and found that the bonding between the fiber and matrix was strengthened due to the treatment. They also reported that the treated jute fiber reinforced composites showed higher mechanical properties than untreated composites [29]. Archana Babu and Narayanankutty (2022) studied the effect of 5% NaOH treated coir fiber reinforced high density polyethylene (HDPE). The results show that the NaOH treatment increased the mechanical properties of the composites and had the highest thermal stability than the untreated one [30].

## 3.0 MICROWAVE CURING OF NATURAL FIBER REINFORCED POLYMER COMPOSITE

Nowadays, NFRP composites are used in various industries like automotive, construction, and aerospace due to their exceptional qualities. The most common processing techniques to fabricate NFRP are compression molding, hand layup, resin transfer molding, and injection molding. In conventional heating, heat is transferred through convection, radiation, and conduction to the material. Heat is transmitted from the outer to inner parts of the materials from external heat sources: thus, the material's temperature changes which will affect the properties [14-15]. Therefore, there is a high chance that the surface of composite will be overheated or damaged. Once the composite is damaged, it is no longer usable, which leads to waste [15]. As the material was maintained at a higher temperature for a greater amount of time during heating, the material may develop a coarse grain structure while the cooling down process was taking place. As a result, this will influence the microstructure and properties of the composite. Researchers have found some drawbacks relating to the conventional heating technique used to fabricate NFRP composites which include [31-32]:

- Non-uniform distribution of temperature
- Degradation of fiber
- Longer production cycle
- Higher energy consumption during production

These drawbacks require a new advance approach to fabricate better qualities of NFRP composites. One of the methods is by microwave curing technique. Microwave processing has already been applied in food processing and drying of textiles, but it is yet to be utilized in manufacturing industries. Microwave processing has been studied by researchers as an efficient alternative technique as it produces a high production rate. Microwave curing of composites has several advantages over the conventional heating method. For instance, volumetric heating throughout the thickness of the material, rapid processing, and less power consumption [14-15, 31-33]. Figure 2 shows the heating of composite samples in traditional conventional heating and microwave heating. In traditional conventional heating, the composite is heated up from the outer surface to the core of the composite, leaving the outer surface at a greater temperature than the core of the composite. Microwave heating, on the other hand, occurs from the core of the composite due to electromagnetic waves, generating heat volumetrically throughout the composite.



**Figure 2** Heating of composite sample in traditional conventional heating and microwave heating

Microwave processing is an eco-friendly method of rapidly heating material from the inside by converting electromagnetic energy into thermal energy. In addition, the microwave heating process takes place at the molecular level and uses 100 times less energy compared to conventional heating, hence, less power consumption [34]. This is a huge advantage especially for thick, large, and uneven composites because it can be challenging to ensure consistent and thorough curing due to unwanted heat variations from conventional heating. Therefore, microwave processing can offer more effective and efficient curing, reducing production time and cost. Microwave processing technique also has the potential to enhance the interfacial bonding and improve the microstructure between polymer reinforced composites which will also improve their mechanical and physical properties [31-35].

According to Naik, Singh, and Sharma (2022), thermoset polymer composites have been processed using microwave curing since 1980. Microwave curing of elastomers was employed between 1970 and 1980 [14]. Unfortunately, there has yet to have enough information on microwave curing on thermoplastic composites, especially on their optimum curing power and curing time. Hence, the current research trend is focusing on microwave curing of natural fiber reinforced thermoplastic composites. Microwave energy can also be utilized in microwave-assisted techniques for compression

molding or resin transfer molding [34-36]. Other than that, microwave curing can be used as a post curing technique for fabricating composites. Post curing is done to strengthen the interfacial bonding between the natural fiber and polymer matrix as well as to reduce the residual stress. Traditionally, post curing techniques were done by room temperature curing and thermal curing which takes longer curing time compared to microwave curing [11-12, 37]. Hence, this shows that microwave heating has the potential to produce a better quality of NFRP composites. However, there are limited works of literature available on microwave post curing of NFRP composites. Table 1 and Table 2 showed a brief overview of the research that has been done on the curing of thermoset and thermoplastic polymer composite, respectively. From Table 1 and Table 2, it is shown that the two main parameters for microwave curing which influence the properties of the composites are power and time exposure. However, it is important to note that different polymers have different process parameters optimization. Hence, studying the kinetics of curing is essential in determining and optimizing the curing cycles of each polymer.

**Table 1** A brief overview of the curing of thermoset based polymer composites

Composite	Fabrication technique	Conventional curing conditions	Microwave curing conditions	Results	Reference
Glass fiber reinforced bifunctional epoxy resin	Vacuum bag molding	Pre curing: Room temperature for 24 hours  Post curing: 85°C for 4 hours in an oven	Post curing: 2.4 kW for 52 minutes	<ul> <li>Mechanical properties of microwave cured composites are in close comparison but better than thermally cured composites by 5%.</li> <li>The microwave curing process was 30 times faster and 20 times more than thermal curing.</li> </ul>	[38]
Carbon fiber reinforced epoxy	Vacuum assisted microwave curing	-	No vacuum pressure:  • 180 W for 422 s • 360 W for 298 s  With vacuum pressure (0.1 MPa): • 180 W for 367 s • 360 W for 209 s	<ul> <li>The microwave curing time decreases with vacuum pressure.</li> <li>Interfacial bonding between fiber and epoxy was enhanced with lower microwave power.</li> <li>The tensile strength and hardness of composites were higher by 116% and 45%, respectively, with vacuum assisted microwave curing.</li> </ul>	[39]
Carbon fiber reinforced epoxy	Vacuum assisted resin transfer molding	Pre curing: Room temperature for 1440 minutes  Post curing: 120°C for 300 minutes in an oven	Post curing: 200 W for 20 minutes	<ul> <li>The use of microwave curing was 15 times faster than thermal oven curing.</li> <li>Microwave curing showed better mechanical properties</li> </ul>	[40]

Composite	Fabrication technique	Conventional curing conditions	Microwave curing conditions	Results	Reference
	- 42-2			<ul> <li>in a short time than thermal curing.</li> <li>Microwave curing has better potential to improve the resin curing rate for carbon fiber reinforced epoxy composites.</li> </ul>	
Flax fiber reinforced epoxy	Vacuum infusion	Pre curing: 80°C for 24 hours	Post curing:  • 100°C for 2 hours  • 120°C for 2 hours  • 150°C for 2 hours	The higher the microwave curing temperature, the higher the glass transition temperature. Composite with 2 hours post curing at 120°C performs better mechanical properties. Post curing leads to a strong modification of the properties of the fibers.	[8]
Carbon fiber reinforced epoxy	Hand layup	Pre curing: 50°C for 2 to 3 hours	Pre curing: 100 W for 65 s	Microwave cured composites have better interface conditions in comparison to thermal curing.      Microwave cured composites showed an increase of 7.78% in tensile strength, 3.46% in tensile modulus, and 0.1% in glass transition with low curing heat time.	[41]
Jute fiber reinforced epoxy	Vacuum assisted resin transfer molding	Pre curing: 60°C for 12 hours  Post curing: • 140°C for 1 hour • 160°C for 45 minutes	-	Natural fiber reinforced epoxy composites showed better water-resistant behavior and mechanical properties compared to commercial resin.	[42]
Carbon nanotubes (CNTs) reinforced epoxy		Pre curing:  • 25°C for 12 hours  • 80°C for 4 hours  Post curing: Room temperature for 24 hours	300 W for 25 minutes	<ul> <li>The composites showed good compatibility with the epoxy matrix.</li> <li>Mechanical properties of CNTs reinforced epoxy after post curing showed better properties compared to polymer matrix alone.</li> </ul>	[43]
Sunn Hemp fiber reinforced epoxy	Hand layup	-	<ul> <li>160 W for 2, 6, and 10 minutes</li> <li>320 W for 2, 6, and 10 minutes</li> <li>480 W for 2, 6, and 10 minutes</li> <li>640 W for 2, 6, and 10 minutes</li> <li>640 W for 2, 6, and 10 minutes</li> </ul>	<ul> <li>Microwave treatment of the fiber showed an improvement in crystalline index and size.</li> <li>SEM study on the microwave cured fiber showed roughness, fibrillation, and interfibrous layers which enhanced the interfacial bonding</li> </ul>	[44]

Composite	Fabrication technique	Conventional curing conditions	Microwave curing conditions	Results	Reference
			• 800 W for 2, 6, and 10 minutes	between fiber and matrix.  The mechanical properties showed an improvement as power and time exposure increased.	
Carbon fiber reinforced epoxy	Hand layup	<ul> <li>90°C for 30 minutes then 120°C for 1 hour</li> <li>100°C for 30 minutes then 160°C for 1 hour</li> </ul>	90°C / 100°C for 10 minutes then 120°C / 160 °C for 30 minutes	<ul> <li>Microwave assisted processing produces uniform temperature distribution.</li> </ul>	[45]
Bamboo fiber reinforced cement	Microwave assisted alkaline treatment	Pre curing: Room temperature for 24 hours  Post curing: Submerged in water for 7, 14, and 28 days	300 W for 1 hour	<ul> <li>Microwave assisted alkaline treated composite showed an improvement in interfacial bonding between fiber and matrix.</li> <li>Microwave assisted alkaline treated composite produces better properties than only alkaline treated composite.</li> <li>Microwave assisted alkaline treated composite.</li> <li>Microwave assisted alkaline treated composite resulted in better tensile strength and flexural strength.</li> </ul>	[46]

**Table 2** A brief overview of the curing of thermoplastic based polymer composites

Composite	Fabrication technique	Conventional curing conditions	Microwave conditions	uring Results		Reference
Grewia optiva fiber reinforced polylactic acid / polypropylen e  Nettle fiber reinforced polylactic acid / polypropylen e	Hot compress molding	170°C for 10 minutes	Post curing: Polylace acid: W for 2 Polypro ene: 90 for 250	900 00 s ppyl 00 W	Microwave cured polylactic acid reinforced composite showed better interfacial bonding compared to polypropylene reinforced composite.  The polylactic acid composite showed a higher failure load than the polypropylene composite.  Power input and exposure time with suitable susceptor of microwave heating are important in the composite joining process.	[47]
Sisal fiber reinforced polypropylen e / ethylene vinyl acetate (EVA)	Microwave energy process	-	<ul><li>360 W</li><li>600 W</li><li>900W</li></ul>	•	Microwave cured of polypropylene reinforced composite was obtained at 570 s at 900 W.  Microwave cured of	[48]

Composite	Fabrication technique	Conventional curing conditions	Microwave conditions	curing	Results		Reference
Grewia optiva fiber reinforced polypropylen e / EVA					•	EVA reinforced composite was obtained at 570 s at 600 W and 390 s at 900 W.  The flexural and tensile strength of the composite showed a significant improvement under microwave curing due to better interfacial bonding between fiber and matrix.	
Nano- hydroxyapatit e (nHA) reinforced polycaprolac tone (PCL)	Microwave energy process	-	850 • 650 650	W for S W for	•	Microwave processing time was reduced to 50% and, 5 times more than thermal processing. The optimum time and power for better tensile strength of composites were 650 s and 180 W, respectively.	[49]
Jute fiber reinforced polypropylen e / polyethylene  Kenaf fiber reinforced polypropylen e / polyethylene	Microwave energy process		150 Polyethylene	W for 0 s : W for	•	The microwave curing process can be used to fabricate NFRP composite as it is efficient and clean.  The mechanical properties of microwave cured NFRP composite are better than thermal	[50]
Sugar palm fiber reinforced polyurethane	Hot compress molding	190°C for 10 minutes	340 W for 90 at: • 70°4 • 80°4		•	conventional curing. Tensile strength was improved with the microwave cured treatment composite at 70°C as it reduces moisture and modified the microstructure of the fiber.  Tensile modulus was also improved at 70°C of microwave cured treatment composite.  The tensile strain for microwave cured treated was lower than untreated and chemically treated	[51]
Kenaf fiber reinforced high density polyethylene (HDPE)	Microwave assisted compression molding				•	composite.  Microwave assisted compression molding was reduced in time and temperature at 5.71 and 4.72,	[52]

Composite	Fabrication technique	Conventional curing conditions	Microwave conditions	curing	Results		Reference
					•	respectively than the conventional method.  Mechanical properties of microwave assisted compression molding composites were found to be better than conventional method composites.	

### 4.0 KINETICS OF CURING

It is known that the properties of a composite depend on the power and time exposure of curing. The curing stage of a composite involves several complicated processes; hence, this process affects the overall quality and properties of the composite. It is necessary to understand the kinetic parameters and their curing states to produce the final ideal product [53-55]. One of the most common ways to investigate curing reactions is by Differential Scanning Calorimetry (DSC). DSC enables the measurement of the heat emitted during crosslinking [55-56].

Johnston et al. (2015) investigate the kinetics of curing reaction between microwave curing and oven conventional curing of epoxy resin by using DSC. It is observed that the rate of microwave curing is higher than conventional curing and microwave. However, there is little difference in the aging behavior of the composites between these two curings. They also mentioned that the curing process is affected in a more complicated way by microwave radiation than by a rise in temperature [56].

Lopez de Vergara et al. (2014) studied the curing kinetics of furan resins between microwave curing and oven conventional curing. They observed that as the heating rate increases, the exothermic peak which controls the curing reaction also increases. Besides that, the activation energy of microwave curing was found to be 13% lower than the activation energy of oven conventional curing. Hence, the effect of microwave energy on furan resins is better compared to conventional curing [57].

Yarlagadda and Hsu (2004) carried out a study on the glass transition temperature of microwave and conventional curing of epoxy resins and found that microwave curing of epoxy resins composites has a higher glass transition temperature compared to the conventional curing of epoxy resins [58]. It is known that the glass transition temperature should be as high as feasible since once the temperature reaches the transition temperature, the composite will degrade, hence, it is better to have a higher glass transition temperature to produce a better quality product.

### 5.0 SUMMARY

In this article, a review of the NFRP composites fabricating technique which includes microwave curing has been provided. Microwave curing has been used to process food and textiles for years. however, due to limited works of literature, there has yet enough information on their feasibility to process NFRP composites. Microwave energy has attracted a lot of attention among researchers as it is well known to heat material volumetrically with low production cost and low power consumption. Besides, it has the potential to produce better qualities of composites. Hence, it is important to further understand the fundamentals of microwave curing. A summary of the information that was analyzed showed that microwave curing of NFRP composites is preferable to traditional thermal curing methods. It was also shown that microwave curing of thermoset and thermoplastic reinforced composites produces better mechanical properties and better interfacial bonding between the fiber and matrix. In the near future, although research has been reported on the microwave curing techniques of thermoset and thermoplastic based polymer composite, research can be improved by better understanding the optimization of process parameters of microwave curing of each polymer.

### **Conflicts of Interest**

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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