



## Feasibility study on microwave joining of ‘green’ composites

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Threatened by a global ecological, environmental and energy crisis, worldwide governmental regulations are challenging manufacturers to achieve all-time high standards of sustainability and eco-efficiency. This has fueled research aiming to replace non-biodegradable petroleum-based resins, synthetic fibers (e.g. glass and composites), with fully bio-degradable environmentally friendly lignocellulosic natural fibers. Due to their high specific strength and modulus, fiber reinforced polymer (FRPs) composites have received widespread attention. In the present study, ‘green’ sisal fiber (SF)-reinforced polylactic acid (PLA) composites were prepared by a compression molding technique. Two types of PLA/SF composites were developed with a fiber content of 10 and 20 percent by weight. An innovative, microwave energy based process was used to join PLA/SF composites. Microwave wattage and exposure time were optimized for each type of composite. A charcoal additive was used to accelerate the rate of joining. These results suggest that microwave energy provides a feasible option for the joining of ‘green FRPs.’

**KEYWORDS:** Microwave joining, green composites, FRP, PLA

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Plant fiber-reinforced polymer (FRP) composites have outstanding potential as an alternative for artificial fiber composites [1]. Due to their structural properties and the fact that they are relatively cheap, recyclable, light-weight and abundant, researchers are interested in exploiting natural fibers as load bearing constituents in composite materials. Natural fibers are generally lignocellulosic in nature. The reinforcing ability of natural fibers is governed by the nature of cellulose and its crystallinity [2]. The use of such biodegradable materials in composites is advantageous because it (i) increases awareness of environmentally-friendly manufacturing (ii) reduces dependency on petroleum products (iii) increases

flexibility (iv) invites the possibility of thermal recycling, and (v) reduces health concerns in the manufacturing process [3]. Moreover, natural fibers provide a viable and abundantly available substitute to expensive and non-renewable synthetic fibers. ‘Green composites’ in which natural fibers (e.g. flax, abaca, bagasse, banana, kapok, kenaf, sun hemp, hemp, jute, henequen, pineapple leaf fiber and sisal) are reinforced by polymer matrices (both from non-renewable and renewable resources) may prove to be competitive or even better alternatives than synthetic fiber (e.g. glass, carbon, aramid) reinforced composites. For example, natural FRP composites have already found use in automotive part manufacturing due to their good mechanical properties and light weight. Polylactic acid (PLA), a corn-based polymer, is made from renewable agricultural raw materials, which are fermented into lactic acid [4]. PLA-based automotive parts emit

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less CO<sub>2</sub> than petroleum-based thermoplastics while providing good aesthetics and ease of processing.

Polymer composites, as fabricated, cannot always be used as the end product. To produce a composite based automotive part, for example, there are generally several parts or components joined together to make the complete assembly. These parts are interconnected with each other through joints to make the final product. Joints are potential sources of weakness and additional weight. The purpose of the joint is to transfer loads from one member to another. There are two main types of joints used in the fabrication of composite products: adhesive bonding and mechanical joints. Both bonding processes have several disadvantages (table 1).

Inevitable limitations of mechanical and adhesive bonding can be minimized using microwave joining of composite materials. The application of microwave energy in the processing of various materials such as ceramics, metals and composites offers several advantages over conventional heating methods. These advantages include unique microstructure and properties, controllable electric field distribution, rapid heating, improved product yield, energy savings, reduction in manufacturing cost and synthesis of new materials [6]. Since microwave joining is an emerging field for polymer matrix composites (PMCs), the literature is sparse in this area. Yarlagadda and Chai [8] investigated welding of engineering thermoplastics using focused

microwave energy. The thermoplastic- joining process was carried out in two separate stages. In the first stage microwave energy was directly induced at the specimen joint interface, and in the second stage epoxy-based resin based primers were applied to promote the joining of the materials by microwave energy. The dielectric properties of thermoplastic polymers (ultra-high molecular weight polyethylene, polycarbonate and acrylonitrile-butadiene-styrene) under room temperature were explored. Bond strength and quality at the joint interface was determined by conducting tensile tests and microscopic examination of the fractured joints. Heating times were also compared with respect to their effects on bond strength and quality of joint achieved at the interface. Ku *et al.* [9] presented a state of the art review of microwave technologies, processing methods and industrial applications using a variable frequency microwave (VFM). VFM offers selective volumetric heating at high energy coupling efficiency. This is accomplished using a preselected bandwidth sweeping around a central frequency employing frequency-agile sources such as travelling wave tubes as the microwave power amplifier. While using VFM processing, a particular frequency is applied for less than 1 millisecond. Experiments were conducted using carbon reinforced polystyrene (PS/CF (33 wt. %)). The manipulated variables of the experiment were

Table 1. Disadvantages of mechanical and adhesive joints\*

Disadvantages	
Mechanical Joints	Adhesive Bonding
Stress concentrations are created by holes and cut-outs. This is worsened by the lack of plasticity that limits stress redistribution	Adhesive bonding usually requires extensive surface preparation before bonding
De-lamination originating from the localized wear occurring during drilling	Pressure may be required during the bonding operation
Differential thermal expansion of fasteners relative to composite	With some adhesives, a long cure time may be needed
Mechanical joints add weight to the structure and thus minimize the weight-saving potential of composite structures	Health and safety could be an issue
Hole drilling requires extensive time and labour	Inspection of a bonded joint is difficult
Mechanical joints made of dissimilar materials create potential galvanic corrosion problems	Adhesive bonding requires more training and rigid process control than mechanical joints
They create fiber discontinuity at the location where a hole is drilled and expose fibers to chemicals and other, potentially destructive, environments	Surface treatments used for adhesive bonding are generally hard to control in an industrial environment and affect directly the strength and durability of bonded joints

\*Information adapted from: [5, 6, 7]

frequency, time and power. The authors found that processing time and tensile strength of the bond were inversely related and that the strength of the bond obtained was less than the strength of parent metal. The authors concluded that up to certain limit, better strength of the joined material could be achieved by reducing the time of its exposure to microwave energy.

In the present study, SF reinforced PLA composites were prepared by a compression molding process. Feasibility of microwave joining was investigated for different combinations of power input and microwave exposure time. The effect of different fiber loading and use of a charcoal additive as a coupling agent was also studied.

#### *Mechanism of microwave heating*

Microwave heating is fundamentally different from conventional radiation and/or convection heating in which thermal energy is delivered to the surface of the material and transferred to the bulk of the material via conduction. Microwave energy is delivered directly to the material through molecular interaction with the electromagnetic field. Microwave heating is the *conversion* of electromagnetic energy to thermal energy rather than heat *transfer*. Since microwaves can penetrate and supply energy throughout the material volumetric heating, making it possible to achieve rapid and uniform heating of thick materials. The thermal gradient in microwave processed materials is the reverse of that in materials processed by conventional heating. In conventional heating, slow heating rates are selected to reduce steep thermal gradients that lead to process-induced stresses. Likewise, in microwave heating, there is a relationship between processing time and product quality.

When microwave energy is in contact with materials having different dielectric properties, it will selectively couple with the higher loss tangent material. Therefore, with microwaves, the joint interface can be heated by incorporating a higher loss material at the interface [10]. Microwaves are electromagnetic waves with wavelengths from 1 mm to 1 m, corresponding to frequencies between 300 GHz and 300 MHz. Within this portion of the electromagnetic spectrum are frequencies used for cellular phones, radar, and television satellite communications. Frequencies reserved by the Federal Communications Commission (FCC) for industrial, scientific, and medical (ISM) microwave heating purposes are 0.915, 2.45, 5.8 and 24.124 GHz. 2.45 GHz (corresponding to a wavelength of 12.2 cm and energy of  $1.02 \times 10^{-5}$  eV) is a commonly used

frequency for microwave heating because low frequencies achieve greater penetration depth [9].

Orientation polarization and interfacial space charge polarization are the two mechanisms which, together with DC conductivity, form the basis of high frequency heating. The electric field component of microwaves exerts a force on the charged particles found in the compound. If the charged particles are have restricted movement by being bound in the compound, they reorient themselves with the applied electric field. This is termed as dielectric polarization. Dielectric polarization can be made up of four components based on the different types of the charged particles in matter: electrons, nuclei, permanent dipoles and charges at interfaces [11]. The total dielectric polarization is given by the equation:

$$\alpha_t = \alpha_e + \alpha_a + \alpha_d + \alpha_i \quad (\text{eq.1})$$

Where,  $\alpha_t$  is total dielectric polarization,  $\alpha_e$  is electronic polarization due to polarization of electrons surrounding the nuclei,  $\alpha_a$  is atomic polarization due to polarization of permanent dipoles in the material,  $\alpha_d$  is dipolar polarization due to polarization of permanent dipoles in the material; and  $\alpha_i$  is interfacial polarization due to polarization of charges at interfaces.

Not all materials can be successfully processed by microwave. The material properties requiring greatest consideration in microwave processing of a dielectric are the complex relative permittivity,  $\epsilon = \epsilon' - j\epsilon''$ , and loss tangent,  $\tan\delta = \epsilon''/\epsilon'$ . The real part of the permittivity,  $\epsilon'$ , sometimes called the dielectric constant, mostly determines how much of the incident energy is rejected at the air-sample interface, and how much enters the sample, and imaginary part,  $\epsilon''$ , is the loss factor and is governed by lag in polarization upon application of field and energy dissipation associated with charge polarization. This represents energy loss in material. The most important property in microwave processing is the loss tangent,  $\tan\delta$  or dielectric loss, which predicts the ability of the material to convert incoming energy into heat [9].

## **Materials and Methods**

### *Fibers and matrix*

Sisal fibers were collected from local Himalayas region of Hamirpur, Himachal Pradesh, India. PLA was supplied by Dow Cargill in pellet form. Charcoal was purchased from RanBaxy, Fine Chemicals Ltd. The biopolymer has a density of 1.25 g/cm<sup>3</sup>. The glass transition temperature ( $T_g$ ) and

melting temperature ( $T_m$ ) are 58 and 165 °C respectively.

#### Microwave

A fixed-frequency microwave (FFM, 2.45 GHz, LG; **fig. 1**) with a maximum output power of 900 W was employed during the feasibility study. The size of the cavity was 527 x 392 x 480 mm.

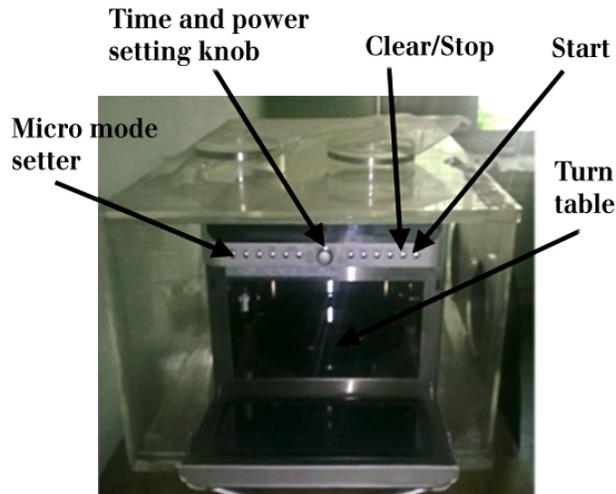


Figure 1. Fixed-frequency microwave

#### Fixture used for joining

A Teflon fixture (**fig.2**) was fabricated for holding the two adherends of PLA/SF reinforced composites. The dimensional details are: Length: 50 mm, width: 25 mm, total height: 14 mm, depth of cavity: 6 mm and thickness: 0.8 mm. Teflon was used to construct the specimen holder because it permits microwaves to pass without any loss of energy. As the melting point of Teflon (243°C) is higher than that of the specimen, the fixture was safe from any heat damage from conduction or radiation.

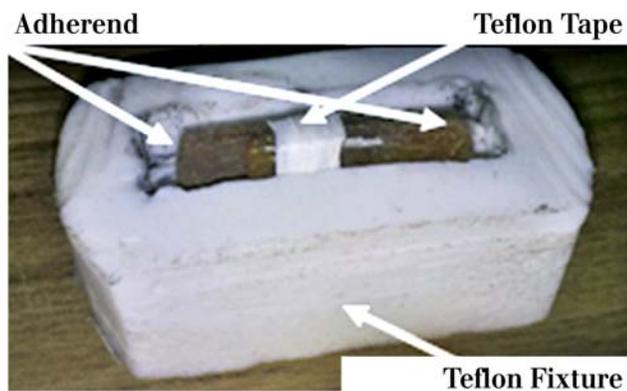


Figure 2. Teflon fixtures holding composite sample

#### Composite preparation

To ensure all absorbed moisture was removed, both SF and PLA pellets were dried in an oven at 80°C for 4 hours before composite processing. PLA pellets were converted into PLA films of 1 mm thickness by compression molding machine at a temperature of 180°C. First, 0.4 MPa of pressure was applied for a contact time of 8 minutes. Second, pressure was increased to 4 MPa at the same temperature. It was then allowed to cool under pressure until finally the PLA film was removed at 80°C.

Composites with two different fiber weight fractions (A, B; **table 2**) were produced by compression molding. Sisal fibers in short fiber form (2 mm) were uniformly distributed in random orientation alternately between the PLA films in a 'picture frame' type mold. Teflon sheets were used at the top and bottom to keep the PLA films from sticking to the steel plates. The whole assembly was placed in the compression molder at a temperature of 180°C with a pressure of 4 MPa for 10 minutes. The pressure was subsequently increased to 8 MPa for 5 minutes, then the composite was cooled under pressure. Composite plates were removed from the mold when the temperature reached 80°C. The thickness of the prepared composite was 3 mm.

Table 2. Weight fraction of SF and PLA in green composites

Composite	SF (Wt. %)	PLA (Wt. %)
A	10	90
B	20	80

#### Sample preparation for joining

Samples of dimension (20 x 6 x 3 mm) were cut from the composite plates. The smooth surfaces of all the samples were roughened by emery paper (grade 80) to increase the surface area and to remove the contaminants and loose particles from the surface. After abrading, the solid particles were removed by blasting with clean air. Samples were initially bound with Teflon tape (arrangement shown in **fig. 2**) before joining and for every run of experiment, fresh samples were used.

## Results and Discussion

#### Microwave processing of PLA/SF composites without additives

Results for both types of composite samples (A, B) are summarized in **table 3**. In the first set of experiments, a feasibility test was carried out by microwaving at a constant power of 360 W. The specimen was unaffected when duration was varied from 30 sec. to 480 sec. in increments of 30 sec. In the second set of experiments, power was increased

to 600 W, again without an effect on the samples. Finally, power was increased to 900 W. From 60 sec. to 90 sec., there was no effect of microwave energy on the samples. When time was set at 120 sec. at the same wattage, there was rise in temperature of the specimen. With increasing durations (up to 480 sec), there was continuous rise in temperature of the samples without bonding. When microwave duration was set to more than 480 sec., the whole sample heated up and began to deform.

Table 3. Effect of microwave process parameters on joining for composites A and B without charcoal additive

Sample No.	Power (W)	Duration (sec.)	Effect
1	360	30-480	No effect
2	600	30-480	No effect
3	900	60-90	No effect
		120-480	Rise in temperature

*Microwave processing of PLA/SF composites with a charcoal additive*

Charcoal powder, a coupling agent usually used as an additive, was applied at the joining area. Microwave energy was concentrated to the area where charcoal was applied, which accelerated the joining.

Table 4 shows results for composite ‘A’. At a power setting of 360 W, samples were unaffected for microwave durations from 30 sec. to 90 sec. The temperature started rising in the specimen when heating time was increased to 120 sec. This trend was observed up to a duration of 200 sec. A bond formed (fig. 5) when the time of exposure was 250, 180 and 150 sec. at power inputs of 360, 600 and 900 W, respectively.

Table 4 Effect of microwave process parameters on joining for composite A with charcoal additive

Sample No.	Power (W)	Duration (sec)	Effect
1	360	30-90	No effect
		120-200	Rise in temperature
		210	Joining started
2	600	250	<b>Bond formed</b>
		30-90	No effect
		100-140	Rise in temperature
3	900	150	Joining started
		180	<b>Bond formed</b>
		30	No effect
		60	Rise in temperature
		130	Joining started
		150	<b>Bond formed</b>
		170	Sample Arced

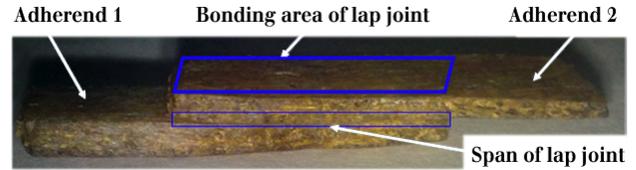


Figure 3. Successful bonding between composite samples

Table 5 summarizes the results of the microwave joining process for composite B. For short microwave durations there was no effect for all three power inputs. As the microwave duration was increased at a power of 360 W, the temperature rose. Joining started at 180 seconds, with the bond forming fully after 200 sec of microwave heating. At 600 W and 900 W, the bond formed when time of heating was 130 seconds and 100 seconds respectively. Samples were distorted when time of microwave exposure was increased to 150 sec. (at 600 W) and 120 sec. (at 900W) due to excessive heating (fig. 4).

Table 5. Effect of microwave process parameters on joining for composite B with charcoal additive

Sample No.	Power (W)	Duration (sec)	Effect
1	360	30-90	No effect
		120-150	Rise in temperature
		180	Joining started
2	600	200	<b>Bond formed</b>
		30-60	No effect
		90-110	Rise in temperature
		120	Joining started
3	900	130	<b>Bond formed</b>
		150	Sample Arced
		30	No effect
		60	Rise in temperature
		90	Joining started
		100	<b>Bond formed</b>
		120	Sample Arced



Figure 4. Distorted joint due to excessive exposure time

The required time of microwave exposure was slightly higher for joining composite 'A' samples than for composite 'B' samples. This may be due the presence of more polymer (90 wt. %) in type 'A' samples than type 'B' samples (80 wt %).

## Conclusion

Here, sisal fiber reinforced polylactic acid (PLA) composites were fabricated using randomly oriented short sisal fibers and thermoplastic type biodegradable polymer by compression molding process. The following results and observations were obtained:

- i) Without an accelerator/additive no joining occurred in either type of composite under investigation.
- ii) By using charcoal as a microwave coupling agent bonds formed at different times of exposure with different power inputs. For composite samples of type 'A', joining occurred at exposure time of 250, 180 and 150 seconds with power input of 360, 600, and 900 W respectively. For composite samples of type 'B', joining occurred at exposure times of 200, 150 and 100 seconds with power input of 360, 600 and 900 W respectively.
- iii) As time of heating was increased beyond the bond formation time, samples were distorted.
- iv) Time taken to form the bond decreased as power level increased.
- v) The duration of microwave exposure required for bond formation was more for samples with lower fiber content.

Future work will focus on investigating the bond strength of joints formed and parametric optimization of the microwaving variables for improving joint characteristics.

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