

# Microwave-Assisted Recycling Process to Recover Fiber from Fiberglass Polyester Composites

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Fiberglass/thermoset matrix composite present important properties, such as specific mass and mechanical strength, aiming to replace some engineering materials. However, its mixture of different materials transforms them into an environmental problem, due to the complex recycling process. The highlight is meeting the demands of environmental pressures and for establishing recycling mechanisms, the proposal is to study physical recycling processes, focusing on the microwave-assisted pyrolysis process and also recovering fiberglass. The results were: humidity around 3 wt. %; the average loss of mass around 68 wt. %; the tensile strength is reduced by about 76 % when a composite with recycled fiberglass is laminated, as compared to the use of virgin fiberglass. This work establishing the physical recycling mechanism and route for composite materials, identifying the thermal degradation processes, for thus applying them to other materials, still considered environmental issues, technically and economically making these materials manufacturing processes viable, as well as establishing a recycling route.

**Keywords:** *Fiberglass, recycling, composites, polyester resin, microwave.*

## Introduction

The demand for lightweight structures in automotive, civil and aeronautic industries, with high strength, low cost and high structural performance started to be developed and improved over time<sup>1</sup>. Therefore, composite materials, especially fiberglass reinforced polyester, gained visibility and their production was increased. In 2018, the glass fiber to composites market reach 2.5 billion pounds in terms of volume and is expected to reach 3 billion pounds in 2024<sup>2,3</sup>.

The estimated compound growth rate will be 10% between 2018 and 2022 according to the Global fiber reinforced plastic report<sup>2</sup>.

The waste generated is classified as not hazardous residue and directly taken to landfills, originating R\$ 90-million expense<sup>2</sup>. Some alternatives like mechanical recycling<sup>4,5,6</sup> and thermoset composites recycling have been investigated to related low cost and good properties<sup>7</sup>. The pyrolysis method is an alternative regarded to energy recovery coupled to the fiberglass recovery in composites<sup>8,9,10,11,12,13,14,15</sup>.

As from the aforementioned aspects, the present study aims to propose a solution technique for the waste-related issues and to find a way of reusing the composites by means of the microwave-assisted pyrolysis, allowing its sustainable consumption and preventing greater environmental liabilities.

One limiting aspect is the nature of the composite, largely constituted of a thermoset polymer and continuous

or discontinuous fibers. This type of polymer has covalent cross links which hinder its remodeling, as occurs in the recycling processes of the most common thermoplastics<sup>16</sup>.

The thermosetting polymers' recycling is usually difficult considering the cross-linked systems. Some studies<sup>16,17</sup> shows different ways of recycling treatments. Some of them are incineration<sup>18</sup>; thermolysis process<sup>19</sup>; mechanical recycling like granulation and comminution to incorporation in a thermoplastic matrix<sup>20,21,22</sup> or in concrete<sup>23</sup>; solvolytic process<sup>24</sup>, chemical recycling process<sup>25,26,27</sup>.

Yang, et al.<sup>28</sup> propose three recycling methods for thermoset matrix composites: mechanical; chemical and thermal.

The mechanical recycling process is the size reduction from composites by cutting or hammer mill, after size classification by a cyclone, which separates the fine particles to the recycling process<sup>29</sup>. But the investigation of this recycling process is to produce building materials with the same properties from virgin fiber<sup>30,31</sup>.

In the chemical recycling can involve solvolysis<sup>32,33</sup>, hydrolysis<sup>28</sup>, glycolysis<sup>34</sup> and acid digestion<sup>28</sup>, using solvent, water, glycols, and acid respectively. In this case, the process involves the chemical degradation or chemical dissolution to remove polymeric matrix. In this process, it can recover the matrix in the form of monomers or petrochemical feedstock by degradation and clear fibers<sup>35</sup>.

The thermal recycling process involves high temperatures which can include 3 types of operations<sup>28</sup>: to recover energy only with incineration or combustion process; to recover

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energy and fiber to combustion recycling and fiber and fuel recovery to pyrolysis process. The pyrolysis processes can involve microwave-assisted recycling.

The microwave-assisted recycling is a physical process which consists in using thermal energy, heat, or a physical treatment so that the polymeric waste is degraded into basic components, such as monomers or other products, from which they can be recombined into new polymers or be used for other applications<sup>36</sup>. The recycling process using microwave is the physical process although the degradation which occurs in the microwave process is a chemical process, in this case, the thermolysis<sup>33</sup>.

Some methods used for thermal recycling are pyrolysis infused salt bath, pyrolysis at high temperatures and gasification. Thermal recycling requires high investments, which makes it viable only for large-scale operations, comparable to petrochemical industry operations<sup>28</sup>.

Microwave heating is totally different from the conventional one, by heat transfer. This type of heating is also called dielectric, with two different mechanisms. The first mechanism for transforming electromagnetic energy into heat is dipole rotation, whereby the molecules (with induced or permanent dipoles) are oriented in one direction according to the application of the magnetic field. After the magnetic field is removed, the energy absorbed by the molecule is liberated as heat. The other microwave-heating mechanism is called ionic conduction. The heat generated derives from losses by friction, which occurs by the migration of ions dissolved under the action of the electromagnetic field. For the most part, substances such as water, acetonitrile, and ethanol, polar substances in general, are good at absorbing microwaves. Conversely, less polar substances (aliphatic or aromatic hydrocarbons) absorb fewer microwaves. Other materials that do not absorb much are the highly ordered crystalline materials. Teflon and type S glass are transparent to microwave<sup>37</sup>.

Using microwaves to provide the necessary heating for conducting pyrolysis proved to be very efficient in the pyrolysis of polyethylene, polyolefins, polystyrene and also of tires. Polymers are usually good microwave absorbers; therefore, so that they receive enough heat for pyrolysis, absorbent materials are added to the polymers, transmitting heat by conduction and reaching the necessary temperature<sup>38,39</sup>.

The micro-wave assisted pyrolysis advantage is the very fast thermal transfer enabling energy savings<sup>40</sup>. The most suitable solution to dispose fiberglass composites was considered the cement kiln route, though in this process there is no way to recover products from the resin to reuse in new resins. The micro-wave assisted pyrolysis was classified like a not economically viable, but with this process it can be possible to recover fiberglass and valuable product after resin degradation to reuse in new resins in a very short time of pyrolysis<sup>40</sup>.

For fitting recycling, references present composites with thermoset matrixes as non-recyclable. Their use has thus faced

great problems. Some technological solutions have already emerged for the technical viability of composite recycling.

The composite recycling does part of the European Union Directive 2000/53/EC which defines some management to the end-of-life vehicles (ELV) and hazardous substances<sup>41</sup>.

The pyrolysis process is considered environmentally friendly because i.e. the related to carbon fibers can produce fibers back with lower energy consumption. The energetic cost from produce virgin fibers is 183 MJ/kg to 286 MJ/kg while the waste fiber will consume around 7.5 % +/- 2.5% of the energy required and approximated 30 €/kg from virgin fibers consumption<sup>42, 43, 44, 45, 46</sup>.

The purpose is to propose a recycling method for thermoset polyester fiberglass composite, aiming to reuse the reinforcement (fiberglass) into reusable residue, which implies a significant reduction of this material waste in landfills and also in the costs related to this issue.

## Materials and Methods

A fiberglass/thermoset composite panel of 1.0 m × 1.5 m was produced in the Polymer and Composite Laboratories of the Institute Maua of Technology.

This composite was prepared with 35 wt.% chopped fiberglass and 65 wt.% to orthophthalic resin, using MEKP (methyl ethyl ketone peroxide) catalyst. The virgin fiberglass used for manufacturing specimens was fabric, with 150 g/m<sup>2</sup> grammage, without the addition of mineral load and UV stabilizer. The thickness plate was 3,00 mm +/- 0,50 mm.

This process occurs with a manual press. From this plate was taken the samples according to ASTM D3039. The manufacturing process was presented in Figure 1.

With the same process was manufacture the recycled fiberglass, with 35 wt.% to fiberglass and 65 wt.% to orthophthalic resin, using MEKP (methyl ethyl ketone) catalyst, thickness plate was 3.00 mm +/- 0.50 mm, the recycled fiberglass was laminated without aligned in a manual press. From this plate was taken the samples according to ASTM D3039.

The 2.5 GHz microwave system assembled in the Microwave Laboratory of the Institute Maua of Technology, as shown in Figure 2 was used for the composite thermal degradation. This system comprises a high voltage source Figure 2j which feeds the microwave generator (Figure 2i), connected to a waveguide (Figure 2g) and unidirectional coupler (Figure 2f) to measure the power irradiated. This power is measured with a power meter (Figure 2h). For protecting the magnetron against the power reflected, a circulator is used (Figure 2e). This reflected power is measured by another directional coupler and absorbed by the water through the dissipative load (Figure 2c). The impedance matcher (Figure 2d) major function is to reduce the effective power sent to the cylindrical cavity (Figure 2b), which has a mobile and short end (Figure 2a).

Firstly, heating tests were required as it was an important parameter for the microwave set up electric power. In this

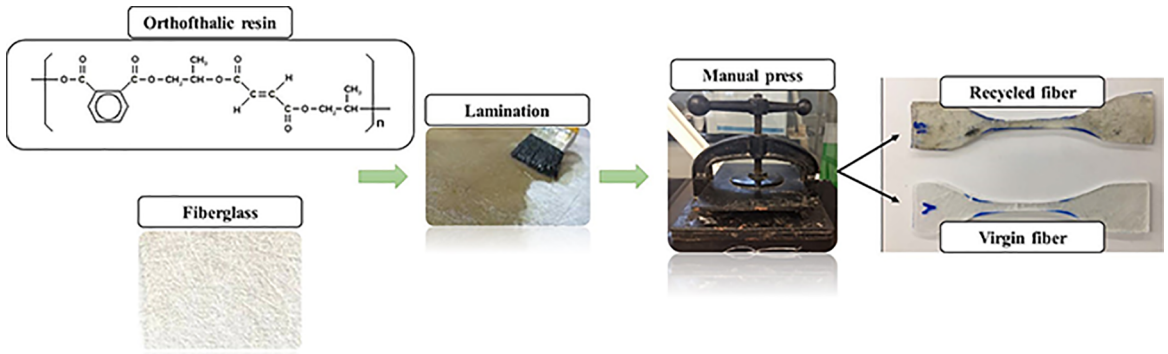


Figure 1. Fabrication of orthohtalic resin with recycled and virgin fiberglass composites



Figure 2. Microwave system at Instituto Mauá de Tecnologia

stage was performed for determining the humidity and burning in a muffle furnace, also for determining how much material was lost, also to determine how much material was lost, and the temperatures at which the occur the loss mass.

For determining the power, burning tests of samples of about 1.8 g were initially performed, with different values from 101W to 558 W of the micro-wave, for thus determining a suitable power. With this selected power, 20 tests of 10 to 20 minute times were conducted to visually determine the best product. This process was adopted only to define suitable power from micro-wave. Due to execute the experiments was used 10 g samples of chopped fibers.

To better understanding what is happening, it was delimiting and optimizing the polyester composite with fiberglass micro-wave recycling process employing a central composite factorial planning ( $\alpha=\sqrt{2}$ ). To reduce the variability it was decided to study only two variables, the effective power (W) and the air flow going through the microwave cavity ( $m^3/h$ ). A preliminary test was performed based on the results from determining the power and the burning time, so as to set the limits at which this study would be applied. In the test, the entire recycling process described in the previous item was conducted, but varying the effective power of the micro-wave and determining the time necessary to reach

the average a loss of mass also obtained in the previous test (about 70 %). The results are illustrated in Figure 3.

Figure 3 shows that from 11 to 17 minutes, the ratio effective voltage and burning time is represented by a curve, differently from the rest of the process, which is simply represented by a straight line. We thus decided to study, model and better understand this curve segment. The qualification of the fumes was not performed herein.

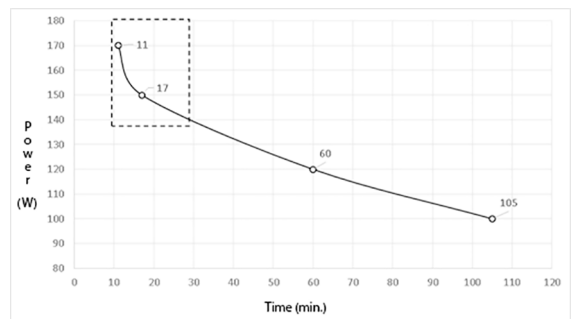


Figure 3. Power versus burning time

For the experimental planning, in this case, the limits of the variables must be defined, as displayed in Table 1. To minimize the errors, ten repetitions were made for the

central points. The air flow limits were defined based on the limit of operation of the rotameter and the compressor. In this process was done with air to determine the thermal degradation of condition to available fiberglass recover.

**Table 1.** Limits of the factorial test

	Effective Power (W)	Airflow (m <sup>3</sup> /h)
+α	188.28	1.28
+1	180.00	1.13
0	160.00	0.78
-1	140.00	0.42
-α	131.72	0.28

The response variables studied were loss of mass ( $m_{\text{initial}}/m_{\text{final}}$ ) and time of visible fumes, which represents the polyester resin thermal degradation. Some equipment adaptations were made to control the air flow through the microwave cavity.

For performing the experiment, experimental planning was proposed, the limits of the effective microwaves power variables and the air flow were defined. The procedure basically constituted in adjusting the power and the air flow according to the factorial planning, introducing the 1.8 g sample with the aid of the ceramic adapted support of high alumina. According to the observation, the time of visible fumes was measure. The sample remained in the equipment for 30 minutes; it was then removed and weighed again, calculating the mass loss calculating.

So, a tensile test was done according to standard ASTM D3039 with recycled fiberglass and virgin fiberglass composites and the results performed was compared.

## Results and Discussion

In burning tests for determining the power of micro-wave was calculated of percentage mass from humidity and loss of mass (determined in burning test), the media value 3 wt. % and 68 wt. %, respectively.

The results from the power and the burning time were solely based on the visual aspect for obtaining a clean and light-colored fiber. For the voltage, at about 170 W, all the polyester resin of the samples was observed to be eliminated at a level similar to the results from the preliminary test (68 wt. % of burning). After the voltage was identified, the times that corresponded to the total elimination of the resin were 11, 12, 13 and 14 min. Thus, a mass balance was conducted for the burning times (20 tests for each condition) and the results are presented in Table 2.

Figure 4 allows identifying the stages of the burning process, by images of the sample captured along the 11-minute test.

According to the numbering of the images, Figure 4a to 4c shows the moment at which the thermal degradation of the resin occurs, depositing residual materials in the fiber, causing the sample to darken.

**Table 2.** Results of the average loss of mass at different times

Time (min)	The average loss of mass (%)
11	70
12	71
13	70
15	71

In Figure 4d to 4f, can be observed the decolored of samples. The test thermal degradation emitted the fumes of carbonization of the polymeric matrix and CO<sub>2</sub> liberation, only the fiberglass remaining.

The results from the tests performed are represented in the graphs relating load (kgf) and displacement (mm) in Figure 5, and the tensile strength of each sample in Table 3. Sample 1 and 2 (Figure 5a) were made of virgin fiberglass and sample A and B (Figure 5b) were made of recycled fiberglass.

The samples 1 and 2 (Figure 5a) from virgin fiberglass have media 1,139 kgf/cm<sup>2</sup> while recycled fiberglass have media 266.5 kgf/cm<sup>2</sup>. The results from Figure 5 were used to calculate the tensile strength represented in Table 3.

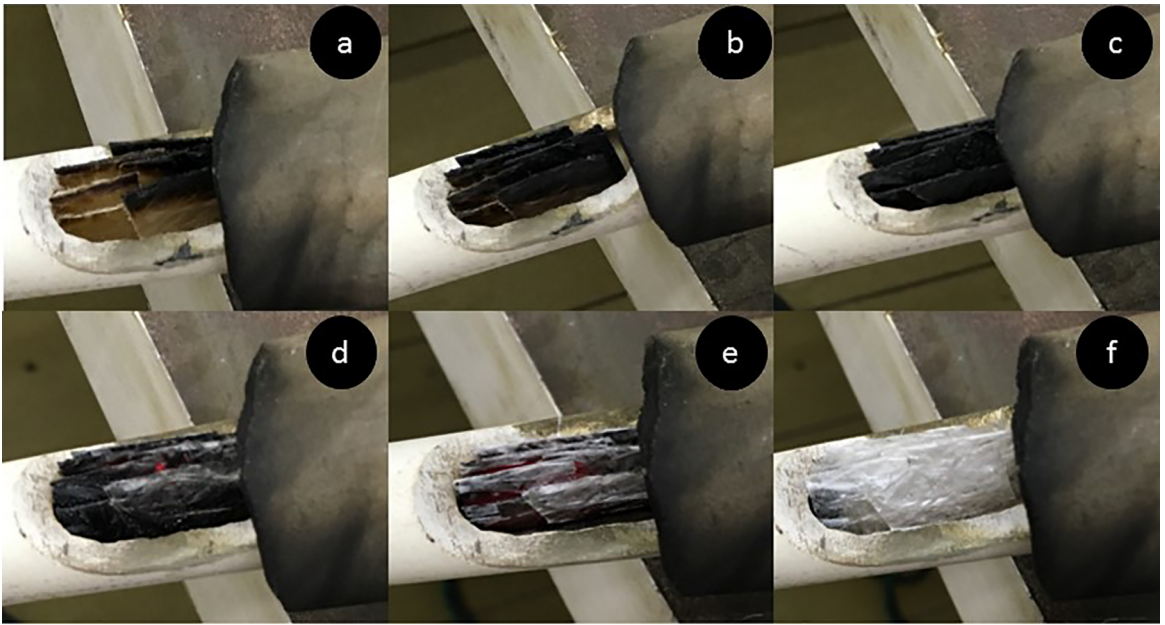
The analysis of the results shows that the tensile strength is reduced by about 76 % when a composite with recycled fiberglass is laminated, as compared to the use of virgin fiberglass.

According to Torres *et al*<sup>47</sup> the mechanical properties of recycled fiberglass after the pyrolysis process of fiberglass polyester sheet molding compound was smaller the virgin fiberglass, the results showed the tensile strength around 18 MN/m<sup>2</sup> to 24 MN/m<sup>2</sup>. Comparing to sample A and B that represents the recycled fiberglass with results from Torres *et al*<sup>47</sup> that is possible to see the correlation, because the recycled fiberglass developed by micro-wave pyrolysis is around 26.10 MN/m<sup>2</sup>.

Another study publish by Giorgini *et al.*<sup>48</sup> which evaluated the properties of recycled carbon fiber in epoxy resin describe the mechanical properties appear to be 65 % to 95 % of the initial values, the same was founded by Lee, Wei and Takahashi in various atmospheres<sup>49</sup>.

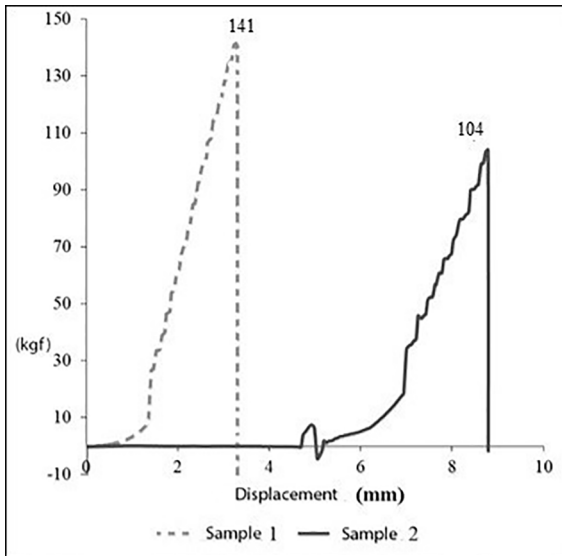
This is probably due to the degradation of the silane encapsulating the glass fibers, responsible for the adhesion of the resin to the fibers, and to the consequent transference of the stresses. For reusing the fibers, they have to be re-silanized. They would hence present better mechanical properties in the composites produced with them<sup>50, 51, 52</sup>.

The samples show an average Young Modulus of 47.0 GN/m<sup>2</sup> +/- 1.5 GN/m<sup>2</sup> for virgin material and a 23.0 GN/m<sup>2</sup> +/- 7,2 GN/m<sup>2</sup> for recycled ones. The recycled fiber has a half value of Young Modulus that the value from virgin fibers, which may characterize the loss of material stiffness, however that fiber still remain usable in many other no structural application<sup>28</sup>. In structural application cases some additional treatment was required<sup>40</sup>

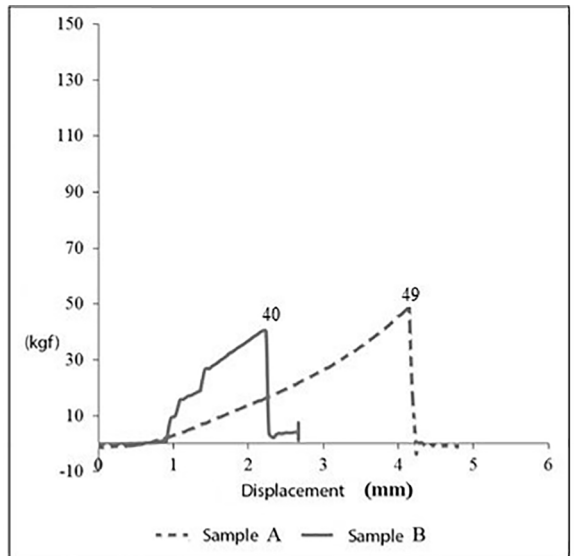


(a) 1 minute; (b) 3 minutes; (c) 5 minutes; (d) 7 minutes; (e) 9 minutes; (f) 11 minutes

Figure 4. Stages of the thermal degradation process



(a) Sample 1 and 2 were made of virgin fiberglass



(b) Sample A and B were made of recycled fiberglass

Figure 5. Graphs of load in relation to the displacement of samples 1, 2, A and B

Table 3. Results of the tensile tests for the different samples

	Area (mm <sup>2</sup> )	Tensile Strength (MN/m <sup>2</sup> )
Sample 1	12.300	112.73
Sample 2	9.210	110.67
Sample A	18.287	26.14
Sample B	15.120	26.13

Studies from Mazzocchetti show that carbon fiber recycled cannot be used in the same original application, but it can be used in wide number of application as internal car panels, thermal insulation or parts with mechanical properties are not essential but possibly a cheaper source of raw material, it also can be applied to the fiberglass recycled<sup>46</sup>.

## Conclusions

This work allowed concluding that it is possible to mitigate the impact of composite waste by means of the recycling process, based on burning irradiated by microwave, once the polymer matrix was fully eliminated by the heating at high temperatures, and the fiberglass was eventually isolated and free from the coke formed during the organic matter burning. The proposal herein generates a reduction of up to 73% of the waste volume, that is, the cost of dumping solid waste in an industrial landfill can be prevented, thus liberating the space the composite would occupy in the biosphere for about 50 years.

Under this study conditions, the fiberglass did not present adequate mechanical strength to be used for making new high quality composites, meantime offer a low cost fiber for a wide number of applications where other glass properties are very desirable. Yet it opens the possibility for optimizing the processing conditions so as to obtain a quality fiber comparable to the virgin fiber. Also indicated is the need to conduct a study on the calorific value of the fumes for its possible use as a secondary fuel. Even though the recycled fiber did not reach the desirable quality, after the process developed, it can be recycled in the glass industry, since it is completely separated from its organic matrix.

From the point of view of knowing the process of thermal degradation irradiated by microwaved composites, it was clear that there is a relationship between the air flow and the microwave power applied. The latter allows identifying that this work used an air flow heavier than that necessary. This reinforces the need for optimizing the processing conditions of this recycling.

The process proposed herein is more advantageous from the energy-saving viewpoint and for the reduction in greenhouse gas generation when compared to conventional thermal degradation. This is due to the microwave application having produced heating in the pyrolysis chamber region alone, not directly heating the reactor structure, which provides considerable energy saving.

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