INVESTIGATION OF INFRARED HEATING OF NATURAL FIBER REINFORCED THERMOPLASTIC POLYMERS

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Abstract
For the compression molding of natural fiber reinforced thermoplastic organic sheets to finished parts, the semi-finished material first needs to be heated above the melting temperature of the matrix. An alternative to the conventional contact heating, which generates high investment costs and space requirement, is given by using infrared (IR) radiation as heating method. In order to facilitate an optimal heating without thermal degradation of natural fibers, the main aim of this study is to investigate an optimum IR heater temperature. This will result in a wavelength range which is well absorbed by the polymer matrix and well transmitted by the natural fibers at the same time. Thus, a selective heating of polymer without thermal impact in natural fibers is pursued. For the selection of a suitable wavelength range emitted by the IR heater at a certain temperature, the substance-specific absorption spectra of natural fibers and PP are recorded by infrared spectroscopy. Based on these results, heating trials are performed and a process window for the heater temperature of 490-600 °C is defined for a suitable heating of natural fiber reinforced polypropylene. The mechanical performance of the heated specimens after pressing shows that it is beneficial to select a heater temperature precisely between the two barrier temperatures in order to avoid insufficient formability or overheating of the surface.

1. Introduction

For decades, natural fiber reinforced polymer composites (NFRPC) have been applied in the European automotive industry for semi-structural interior parts like door panels, backrests, dashboards and others [1, 2]. Due to their low density (1.2-1.5 g/cm) and good mechanical performance, natural fibers like flax, hemp and kenaf show a very good lightweight potential compared to standard reinforcing materials like glass fibers with a density of about 2.5-2.6 g/cm³ [3]. More than half of the NFRPC used in the automotive industry are thermoplastic based, whereby the most common matrix system is polypropylene (PP) because of its low density (0.9 g/cm³), excellent processing properties, and good mechanical and impact performance [4, 5].
Due to the new European Regulation EC 443/2009 for the decrease of emissions of light-duty vehicles [6], it is expected that the production of NFRPC based on above-mentioned natural fibers will increase by at least 30 % until 2020 [1]. However, further market penetration of natural fiber reinforced polypropylene (NFPP) can only occur if the production costs can be economically competitive to the common injection-molded thermoplastic materials in the automotive industry [2]. Therefore, the process optimization for the manufacture of NFPP composites is a major issue.

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In case of NFPP, the semi-finished products are preferably nonwoven hybrid textiles, consisting of 50 wt.-% natural fibers like hemp and kenaf, and 50 wt.-% PP melt fibers. They are cost-effective and easy to manufacture in large-scale. In general, at least two presses are needed for the processing of these semi-finiesd products to components. The first one is used as contact heating press (> 200 °C) in order to heat the PP fibers beyond their melting temperature and to compress the nonwoven to the desired thickness and density, depending on the particular application. The molten PP impregnates the natural fibers in this stage and afterwards, the nonwoven is thermoformed at lower temperatures (~ 60 °C) in a second press to a finished part. Often, a third press is used in between for the adhesive-free application of a décor.

This multistage press process requires high capital expenditure and floor space [8]. Moreover, the heating step in the manufacture of NFPP is accompanied by several critical issues. The thermal impact during the hot pressing of the nonwoven can lead to a variety of physical and chemical changes and damages in the natural fibers, because the thermal decomposition temperature of the fiber constituents is regarded as being around 200 °C [7]. It is presumed that, among other, these are the main issues which limit the market penetration of NFPP. An infrared (IR) heater is offering a reliable alternative for the heating step of the compression molding process, since IR heating of semi-finished fiber reinforced composites is a well-known and established method for the common thermoforming process [9].

So far, the use of IR radiation for the heating of NFRPC has been considered unsuitable due to the uneven surface of NFPP semi-finished nonwovens and the stick out of individual fibers. Hence, a significantly high risk of overheating the material surface exists [10]. In order to prevent overheating and to minimize the temperature gradient, the nonwovens must be precompacted to already impregnated, but not fully consolidated organic sheets. This offers the advantage of separating the press stations of the conventional manufacturing process. Producer of finished parts only need one press for the processing of NFPP and moreover, the storage and shipment of natural fiber organic sheets is much easier, since they need less space and are less sensitive to humidity.

However, IR heating of NFPP organic sheets is a new process, that on the one hand must be competitive to the standard contact heating process in terms of processing time. On the other hand, there is still a risk to thermally affect the natural fibers by inappropriate selection of heater temperature. Therefore, the aim of this study was to investigate an optimum IR heater temperature for the selective heating of PP by selecting an IR wavelength range which is well absorbed by PP and well transmitted by natural fibers. This should result in a quick heating of NFPP organic sheets to the forming temperature without thermally degrade the natural fibers.

2. Materials and Methods

In this work, a NFPP material, commonly used in the European automotive industry with an area weight of 1,700 g/m², was examined. The constituents are 30 wt.-% hemp fibers, 20 wt.-% kenaf fibers and 50 % PP melt fibers. The semi-finished nonwoven was precompacted to 3 mm thick organic sheets (density: 0.55 g/cm³) in a hot press process. The precompaction aims to reduce the heat gradient between surface and middle of the nonwoven and to pre-impregnate the natural fibers, which usually stick out of the conventional uncompacted nonwovens. Preliminary tests showed that a sheet thickness of 3 mm is cost-effective and easy to manufacture in a continuous process and it is beneficial for the IR heating of NFPP organic sheets.
In order to process NFPP organic sheets, they must be heated beyond the melting temperature of PP, which is approx. 160-165 °C [11]. Usually, a minimum temperature of 185 °C through the entire thickness of the sheets is aimed in order to facilitate optimal forming conditions. The maximum temperature during heating is limited by the degradation point of natural fibers (approx. 200 °C for > 60 s and up to 215 °C for < 60 s) [7].

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2.1. IR Absorption Behavior of Natural Fibers and PP

Infrared radiation is transfer of thermal energy by electromagnetic oscillations. When radiation strikes a material, one part will be absorbed, another part will be transmitted and the rest will be reflected. For IR heating, it can be taken advantage of the fact that different materials absorb IR radiation at different wavelengths. Especially for temperature sensitive materials like NFPP, it is beneficial to select a heater temperature in a wavelength range that is well absorbed by the polymer matrix and well transmitted by the natural fibers at the same time. The substance-specific absorption spectra of natural fibers and PP are of fundamental importance for the selection of a suitable range of wavelengths emitted by the IR heater. These spectra are determined by a fourier transform infrared (FTIR) spectroscopy.
FTIR analysis on hemp and kenaf fibers and polypropylene in fiber and in pressed state were carried out using a Nicolet 510 spectrometer. Transmission spectra were recorded at wave numbers from 650-4000 cm^{-1}, because they represent the middle wave IR range 2.5-15.38 µm and correlate with most middle wave IR heaters. As measuring mode, the attenuated total reflection (ATR) was used with an average of 64 recorded scans for each spectrum.

2.2. Heating Trials with NFPP

For the heating of NFPP, a medium wave IR heater from Krelus type G14-25-2.5 MINI 7.5 was used with a continuously variable temperature between 20-840 °C. The emission degree of the heater is approx. 90 %. Two IR emitters were mounted on a frame, movable in height, so that the NFPP samples can be placed between the heaters and the distance between heater and material can be varied. The heating trial is schematically shown in Figure 1.

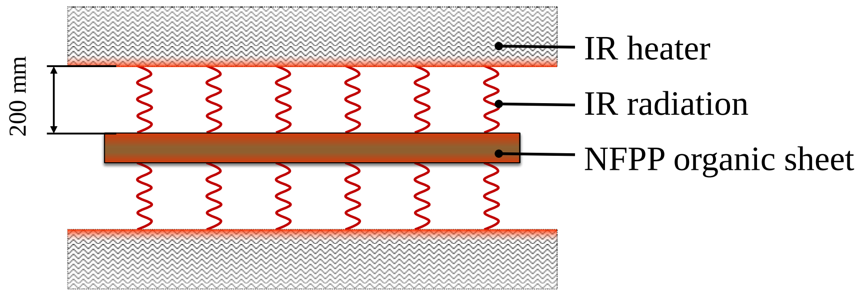

Figure 1. Schematic description of IR heating trials of NFPP organic sheets.

For a quick and gentle heating, the ideal distance between NFPP organic sheet samples and IR heaters was examined in preliminary tests and amounts to 200 mm. The temperature over time was recorded during the entire heating trials at the upper and lower surface and, furthermore, in the middle of the organic sheets by type K thermocouples.

2.3. Mechanical properties of IR heated NFPP

After heating NFPP via IR radiation up to the forming temperature, the organic sheets were thermoformed in an isothermal press tool at 60 °C to flat panels by a hydraulic press to a wall thickness of 2 mm, i.e. a defined final density of approx. 0.75 g/cm^3. Since in NFPP organic sheets the fibers are already impregnated by the matrix, but the sheets are still not fully consolidated, no dimensional changes but the thickness reduction occurred during pressing. Depending on the application, this is a common density for NFPP parts. The suitability of different heater temperatures was investigated by mechanical testing of the processed panels. The bending modulus and bending

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strength of the NFPP samples were examined by a 3-point bending test according to DIN EN ISO 187 on a Zwick universal testing machine. The results are an indication for thermal fiber damages, since the mechanical properties decrease with increasing thermal stress in natural fibers [7].

3. Results and Discussion

The investigation of the IR heating behavior of a standard NFPP material was performed in three stages. First, the IR absorption behavior of the individual constituents hemp, kenaf and PP was examined via FTIR analysis. Then, suitable IR heater temperatures according to this absorption behavior were selected and heating trials were performed on NFPP organic sheets. Lastly, after heating, the organic sheets were thermoformed to panels with a density of approx. 0.75 g/cm³. The assessment of the suitability of heater temperatures was investigated by mechanical testing of the panels.

3.1. IR Absorption Behavior of Natural Fibers and PP

Transmission spectra of hemp, kenaf, and PP in fiber and pressed state are shown in Figure 2 for the wave number range 650-4000 cm^{-1}. The transmission coefficient τ indicates qualitatively the amount of radiation passing the sample without any effect.

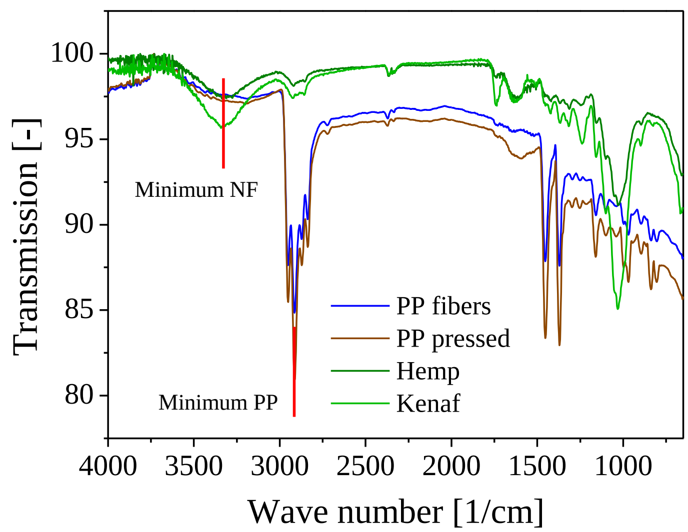

Figure 2. Difference in absorption behavior of different relavant natural fibers.

It can be observed that the natural fibers hemp and kenaf qualitatively show a similar transmission behavior since their minima are located at almost identical wave numbers. The same behavior can be observed for PP fibers and pressed PP. By neglecting the reflection of the samples, the absorption coefficient α can be calculated according to equation (1).

$$\alpha = 100 - \tau \quad (1)$$

The absorption coefficient indicates the qualitative amount of energy absorbed by the specimen and transformed into thermal energy by excitation of atoms and molecules. Unfortunately, according to Plack's law, IR heaters radiate energy over a broad spectrum of wavelengths. The radiant power S_λ of an ideal IR heater at a given temperature T as function of wavelength λ is given in equation (2), whereas h is the Plack constant, c_0 is the speed of light in vacuum, and k is the Boltzmann constant.

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$$S_\lambda = 2\frac{hc_0^2}{\lambda^5}\cdot\frac{1}{e^{\frac{hc_0}{\lambda kT}}-1} \tag{2}$$

As the heater temperature T increases, the peak energy wavelength λ_{max} for a given IR heater temperature decreases (maximum of radiant power shifts to higher wave numbers, see Figure 3) and the energy is distributed in a narrower wave band (Wien's displacement law). This correlation is given in equation (3) with b as Wien's constant.

$$\lambda_{max} = \frac{b}{T} \tag{3}$$

For temperature sensitive materials like NFPP, it is of crucial importance to select an IR heater temperature in a wavelength range that results in peak energy wavelengths that are well absorbed by the PP and well transmitted by the natural fibers at the same time. In this work, five possible IR heater temperatures between 459-626 °C were investigated. The average absorption of natural fibers and PP as also the the radiant power of an IR heater at five exemplary heater temperatures are displayed in Figure 3.

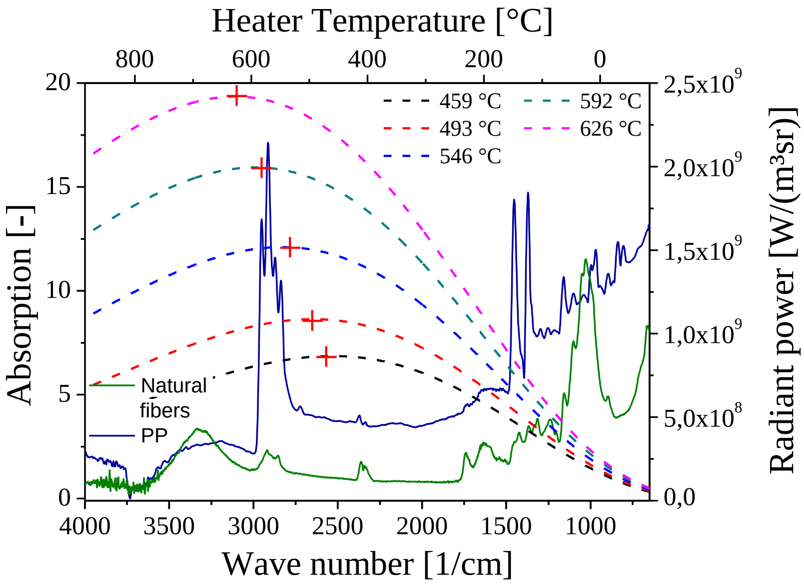

Figure 3. Absorption behavior of natural fibers and PP and radiant power over wave number for different IR heater temperatures.

Theoretically, the thermal energy input in the natural fibers is highest at a heater temperature of 626 °C which could lead to thermal damage of the fibers. At a heater temperature of 459 °C, their energy input is the lowest in the natural fibers, but also in PP, which could lead to a very slow heating of the organic sheet. These theoretical considerations were investigated in heating trials at all five temperatures in Figure 3.

3.2. Heating Trials with NFPP

For the IR heating of NFPP in the industrial scale, the time until forming temperature (~ 185 °C) in the middle of the organic sheet should be achieved within 60 s, whereas the surface of the sheets must not exceed the degradation temperature of natural fibers. Surface temperatures of NFPP up to 215 °C are acceptable for a few seconds. For the five temperatures, 459 °C, 493 °C, 546 °C, 592 °C, and 626 °C,

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the time until forming temperature is reached in the middle of PP and the corresponding surface temperature are given in Figure 4.

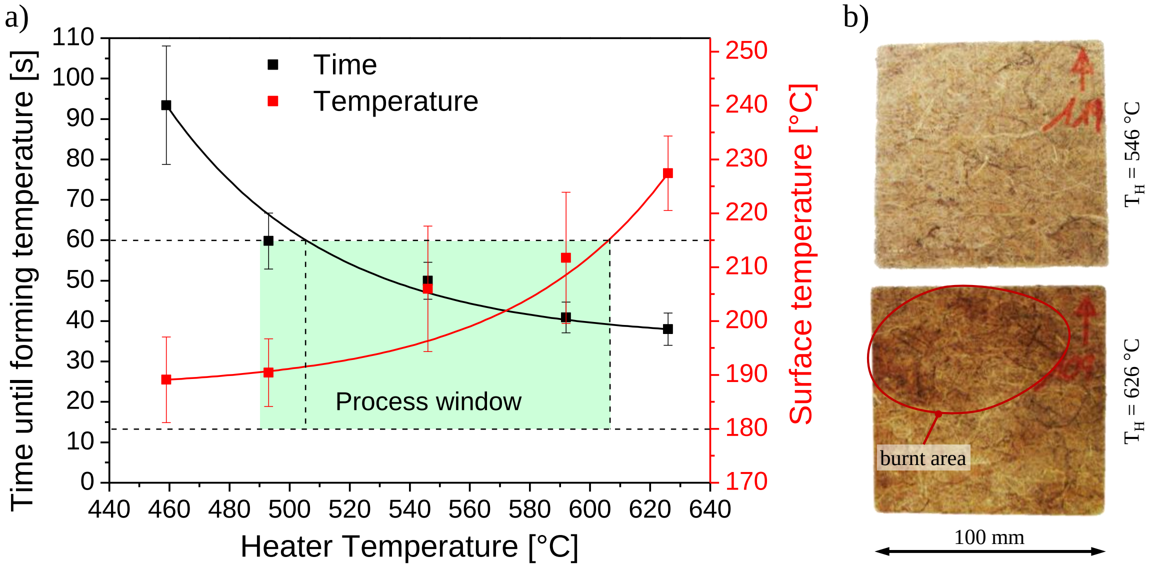

Figure 4. a) Process window for IR heating of NFPP at 200 mm radiator distance and b) specimens heated at different temperatures

As expected from Figure 3, the heater temperature 626 °C leads to thermal damage in natural fibers. This can be seen in the surface temperature of almost 230 °C in Figure 4 a). In Figure 4 b), two specimens are showed which reach the forming temperature in the middle. However, the upper specimen shows the color of NFPP heated at a suitable temperature of 546 °C, whereas the lower specimen confirms that a selected temperature of 626 °C results in a burnt area, which is caused by thermal degradation of natural fibers.
At a heater temperature of 459 °C, the heating trial until forming temperature takes too long for an industrial scale. Moreover, the specimens could not be thermoformed to a lower thickness (higher density). Only the three chosen temperatures between the temperature borders are suitable for an appropriate heating of NFPP. According to these results, a process window is given in Figure 4. Suitable IR heater temperatures for the efficient and careful heating of NFPP range from approx. 490-600 °C.

3.3. Mechanical properties of IR heated NFPP

In order to examine the most suitable heater temperature and to determine if there are fiber damages during IR heating at specific temperatures in the process window, the mechanical performance of the IR heated NFPP organic sheets was tested. Since the heater temperature 459 °C was too low for a suitable compaction of the organic sheets and the temperature 626 °C resulted in burnt specimens, only the temperatures within the process window (493 °C, 546 °C, 592 °C) were examined. The bending modulus and bending strength are shown in Figure 5.
The bending modulus of all samples is 2,390-2,740 MPa and the strength is 39-44 MPa. The values correlate very well with empirical values for NFPP at similar density. It can be observed that the modulus and the strength are slightly higher for the heater temperature 546 °C than for the other two heater temperatures. However, the results show that the process window for IR heating is relatively high and all temperatures within this process window do not thermally damage the fibers.

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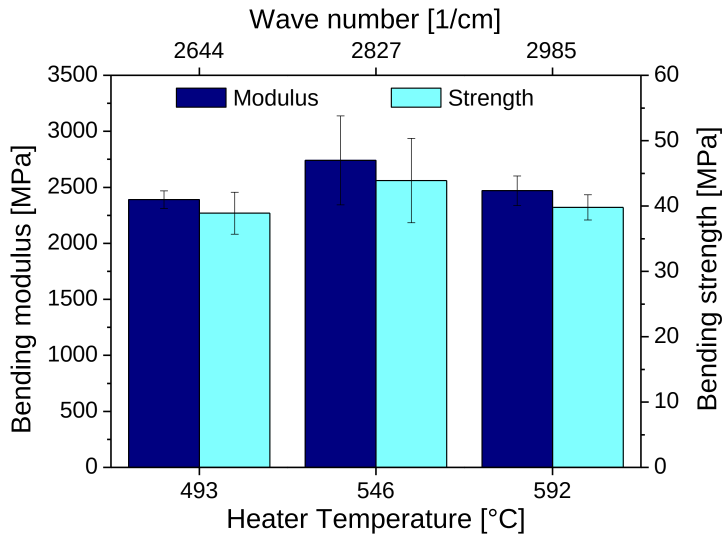

Figure 5. Mechanical properties of NFPP, heated by IR radiaton, in dependency of heater temperature.

4. Conclusions

The aim of this work was to investigate an optimum IR heater temperature for the selective heating of the polymer matrix in natural fiber reinforced thermoplastic polymers. This was performed exemplarily on PP, since it is the most common thermoplastic matrix for NFRPC. The heat gradient between surface and middle of the NFPP nonwovens was minimized by melting first the PP fibers and precompacting the mats in a press process from 8 mm to 3 mm wall thickness. The goal of the heating trials was to heat the organic sheets beyond the forming temperature of PP in the middle, whereas the surface of the sheets must not exceed the degradation temperature of the natural fibers at the same time. In order to determine suitable heater temperatures, the substance-specific absorption spectra of hemp, kenaf, and PP were determined by a FTIR spectroscopy. According to the recorded spectra, heater temperatures between 459-626 °C were selected for the heating trials. Considering the restriction of a short heating time of max. 60 s and the fact that the surface must not overheat, the result of the heating trials indicated a process window for suitable heater temperatures of 490-600 °C. The emitted wavelength range of those heater temperatures are well absorbed by PP and, for the most part, transmitted by natural fibers. The testing of the mechanical performance of the IR heated NFPP organic sheets showed that the temperatures within the process window are, in general, suitable to heat NFPP. They also showed that it is beneficial to select a heater temperature precisely between the two barrier temperatures in order to avoid overheating of the surface or insufficient formability due to insufficient heating in the middle of organic sheets.

This method can be applied on most thermoplastic polymers suitable for NFRPC, since most of them have a similar IR absorption behavior.

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