INVESTIGATION OF A MICROWAVE SUPPORTED POLYMER PELLET DRYER

Oliver Kast, Tobias Schaible and Christian Bonten, Institut für Kunststofftechnik, University of Stuttgart, Germany

Abstract

This paper investigates the effect of microwave application for the drying of pellets for five different polymers. As microwaves stimulate water molecules directly, they can be used for a volumetric heating of the pellets and increase the speed of migration of moisture from within the pellet to its surface. Experimental results show how microwaves lead to a temperature invariant drying speed, at least above a polymer specific threshold temperature. Comparisons with a reference dryer showed an increased drying speed through microwaves at lower drying temperatures, but not necessarily at higher ones. However, taking into account constructive inefficiencies of the prototype microwave dryer, microwave application shows the potential to significantly reduce drying times also at higher temperatures, which is shown representatively for polyamide 6. An analysis of material properties after drying did not show significant differences between microwave drying and conventional drying.

Introduction

The majority of engineering plastics are so called hygroscopic plastics, i.e. they absorb moisture and bind it within. Therefore, they need to be dried before processing and converting, as the presence of moisture would otherwise lead to material damage. [1] This may range from simple surface defects like clouding and loss of transparency or gloss to severe changes in the performance characteristics of the final part due to molecular degradation via hydrolysis. [2] Another possible consequence of insufficient drying is unwanted foaming of the melt. Also, the processing itself can be disturbed due to viscosity changes. In all cases, insufficient drying leads to wastage, as the products do not meet the quality standards.

Most State-of-the-Art (SoA) dryers for plastic pellets are so called desiccant dryers. They use a stream of dehumidified hot air to convectively dry the pellets. The drying performance is therefore physically limited by the speed of heat conduction and diffusion of water particles within the polymer, as these are the determining mechanisms for the removal of molecular bound water [3, 4]. Currently, the most widespread technology is closed-loop drying with integrated air dehumidification. The air is first dehumidified to a certain dew point (usually -30 or -40 °C), by passing through a molecular sieve cartridge filled with desiccant agents, e.g. zeolites. Then it is heated up to a material specific drying temperature. This guarantees constant drying conditions not depending on the current weather or climate. The hot dehumidified air is then led through the pellets to remove the water and back to the molecular sieve cartridge again in a continuous loop. Before passing the molecular sieve it needs to be cooled down for a better dehumidification. Since the desiccant agents over time become saturated with water, the systems are equipped with at least two cartridges, with one being in use to dehumidify the hot air and the other one being recovered from adsorbed moisture itself. Thus, the system requires a separate air cycle and an additional heating device for the recovery of the desiccant cartridge, which consumes a lot of energy. Energy efficiency has been an issue for producers of desiccant dryers and in recent years systems have been optimized via intelligent control and regulation systems as well as waste heat recovery via integrated recuperators. Nonetheless, the efficiency limitations due to the air first being heated and then cooled down in a constant loop can only be reduced but not completely be overcome, as they are a direct consequence of the process design itself. [5]

The idea behind microwave-based drying (MW drying) is to use the microwaves for a) volumetric heating of the pellets, and b) increasing the partial pressure of the bound moisture due to the microwave stimulation of water molecules (Figure 1). There are very few industrial approaches of microwave supported drying for polymers, and so far it is not yet well established in plastics processing, even though it is considered promising [6]. To the authors’ knowledge, there is only one system on the market in Europe which is specifically designed for polymer pellet drying. The system is only designed for high throughput rates and mainly used for PET drying [7]. Other than that, no systems are known to the authors. Generally, microwave supported drying poses a challenge to converters, as the parameters have to be carefully adjusted to not cause thermal damage or even melt the pellets and there is only little knowledge on how to select the right settings for each process. Different polymers react differently to microwave application, depending on their dielectric properties. [8, 9, 10]

This paper investigates the drying kinetics of a prototype drying unit, which combines microwave application with the design and advantages of a
conventional air dryer (see Figure 1) in order to evaluate the influence of microwaves on drying speed, but also possible changes in material properties.

![Figure 1. Principle of microwave supported drying](image)

**Experimental**

**Materials**

Five different polymers were chosen for the tests, based on an online survey to identify typically critical materials in industry: polyamide 6 (two different grades), polyethylene terephthalate, polylactid acid and polyhydroxybutyrate. The selection includes two typical hygroscopic polymers with high market share, but also two types of bio-based polymers, as these are known to be specifically sensitive to any kind of thermal treatment. Also, two different grades of PA6 were chosen in order to take into account the varying characteristics of different grades from different raw material producers even within one polymer family. Table 1 gives an overview on the five materials, the respective suppliers and their glass transition temperatures (as measured via Differential Scanning Calorimetry at IKT, 1<sup>st</sup> and 2<sup>nd</sup> heating).

As the materials were received in a pre-dried state and in most cases ready for processing, they had to be conditioned first, in order to allow for reasonable drying experiments. For this, a climate chamber from Espec Europe GmbH. Munich, Germany, was used. The conditions were set to 86 % relative humidity and 23 °C. The materials were put into the climate for two hours in batches of 50 kg, which is the same amount as was later used for the drying experiments. As can be seen in Figure 2, the moisture take-up during conditioning differed widely among materials, with both PA6 types absorbing the most humidity. Also, the variation between conditioned batches was highest for the PA6-2. For this reason, the moisture of each individual batch was measured again right before it was introduced into the dryer.

![Figure 2. Average moisture contents after conditioning](image)

**Equipment and Experimental Set-up**

All tests were done with a pilot plant dryer designed and built by consortium partners within the EU-funded project “HiPerDry”. The drying unit consists of a conventional drying silo, which is equipped with specially designed microwave antennas and four magnetrons. The dryer can be used with circulating (un-dried) hot air only or with dehumidified air. For the main series of tests, dehumidified air was used. The air flow was 130 m³/h. Reference tests were done with a SoA desiccant dryer from Bierther GmbH, Bad Kreuznach, Germany, with the same air flow of 130 m³/h.

First, screening tests were made with PA6 to evaluate the microwave power settings. For this, microwave power was slowly increased in steps of 10 % of total installed power, until visible damage (discoloration, melting) to the pellets occurred. Based on the results, the microwave settings for the main tests were chosen. Main tests were carried out at different temperatures and for different drying times. For each test, the dryer was filled with 50 kg of the respective material. As a convective medium dehumidified hot air was used for the majority of the tests, using the same desiccant unit as for the SoA reference drying tests. The pilot plant was also used in additional tests with a) only microwaves and b) only dehumidified air to separate effects and to rule out a possible bias when comparing a pilot plant, which may suffer from

<table>
<thead>
<tr>
<th>Material</th>
<th>Supplier and Trade Name</th>
<th>Glass Transition (1&lt;sup&gt;st&lt;/sup&gt; / 2&lt;sup&gt;nd&lt;/sup&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PA6-1</td>
<td>BASF Ultramid B40</td>
<td>56 °C / 52 °C</td>
</tr>
<tr>
<td>PA6-2</td>
<td>Repol Dinalon B2S40</td>
<td>65 °C / 52 °C</td>
</tr>
<tr>
<td>PET</td>
<td>Novapet CR</td>
<td>76 °C / 79 °C</td>
</tr>
<tr>
<td>PLA</td>
<td>NatureWorks Ingeo 4032D</td>
<td>68 °C / 63 °C</td>
</tr>
<tr>
<td>PHB</td>
<td>TianAn Enmat Y1000</td>
<td>− / 5 °C</td>
</tr>
</tbody>
</table>
constructive inefficiencies, to a fully developed SoA system.

Samples of dried material were taken in intervals of one hour and the current moisture content was determined via calcium hydride method with moisture detection system Aquatrac® from Brabender, Duisburg, Germany. To have a fixed frame of reference for all experiments, drying curves were fitted to the experimental results. For this, the moisture content values were logarithmized and thus linearized. Then a linear equation of the following type could be applied:

\[
\ln(y) = \ln(\alpha) + \beta \cdot t
\]  

(1)

where \(y\) is the moisture content, \(t\) is the drying time and \(\ln(\alpha)\) and \(\beta\) are the linear equation parameters. The fitted curve can then be transformed into an exponential curve as follows:

\[
y = \alpha \cdot e^{\beta t}
\]  

(2)

To evaluate the drying performance, the drying time \(\tau\) from a moisture content of 0.2 wt.-% down to 0.1 wt.-% was calculated from the acquired curves for all tests as follows:

\[
\tau = \frac{\ln(0.1) - \ln(0.2)}{\beta}
\]  

(3)

It has to be noted that in some cases this was achieved by means of extrapolation.

**Results and Discussion**

**Microwave Power**

The screening for the effect of different power levels of the magnetrons was made for PA6-1, as it has the highest dielectric constant of all materials, thus is able to absorb the most microwave energy (dielectric properties were determined by Fraunhofer IGB within the common HiPerDry project). Therefore, it has to be ensured that the energy absorption does not result in damage or melting of the material. Also, the reflected power of the microwaves was measured to evaluate the effectiveness. The power was increased step by step up to 3 kW. At this point, discoloration and agglomeration of the pellets occurred.

Figure 3 shows the shares of effective power and loss power for the different absolute power levels. It can clearly be seen that with higher total power levels, the share of effective power is more and more decreasing. Therefore it is not reasonable to use power levels beyond 1.8 kW as the energy, which is actually absorbed, only increases slightly when going up to 2.4 kW, whereas the loss power almost doubles. Consequentially, all main tests were made with 1.8 kW microwave power.

![Figure 3. Effective and loss power of MW application for PA6-1](image)

**Drying Performance**

As can be seen in Figure 4, microwave application has a significant influence on the drying kinetics. The above graph shows the reference tests on a desiccant dryer for PA6-1 at three different drying temperatures. As it is to be expected, drying is faster for higher temperatures. However, this effect cannot be seen in the graph below for drying with microwave application. In this case, the microwaves seem to be the dominant driver and temperature variation becomes irrelevant for the drying speed.

![Figure 4. Drying curves over time for desiccant drying (above) and drying with MW application (below)](image)
However, this effect differs for different materials. Figure 5 shows the drying curves of PET. Whereas the two curves for 130 and 150 °C coincide in a similar way to the PA6-1 curves, the one for 80 °C deviates. This implicates the existence of a threshold temperature above which the microwaves become the dominant mechanism for drying, but below which the drying speed is still dependent on the temperature. The same effect occurs for the drying of PA6-2 and PLA. It can be assumed that such a threshold temperature might be around the glass transition, but whereas this applies for PET and PA6-2, it does not for PLA. Lastly, for the drying of PHB the effect does not occur at all and temperature dependence is always given.

Drying time is similar for all temperatures when using microwaves, whereas it decreases with rising temperature when using a desiccant dryer, which makes the latter more effective at higher temperatures.

It has to be considered, however, that the SoA desiccant dryer is a fully developed system, whereas the MW dryer is a prototype, which still suffers from constructive inefficiencies, such as heat loss or unnecessarily excessive tubing, which can be optimized for future plants. To evaluate the actual potential of savings from microwave application, the pilot plant was used in two further modes:

- microwave drying without convective medium and
- dehumidified hot air drying without microwave application.

The latter of these modes allows to evaluate how a desiccant dryer with comparable constructive inefficiencies would perform and thus shows the actual potential of microwave application with all other factors being equal. As can be seen in Figure 7, drying time is 64 % shorter for the combination of microwaves and dehumidified hot air than it is, when only dehumidified hot air is used (as is the case in desiccant dryers). This demonstrates how the microwave application principally accelerates the drying in comparison to mere convection drying. Thus, in order to fully exploit this potential, the drying system has to be further optimized to reach a constructive effectiveness comparable to SoA desiccant dryers in order to compete with them.

**Effects on Material Properties**

In order to evaluate possible influences or damage to the material due to microwave application, the samples were rheologically and mechanically analyzed. For this, they were first dried to a moisture content of ≤ 0.1 % for PA6, PLA and PHB and ≤ 0.02 % for PET respectively. Then the samples were used for rotary rheometry to detect zero shear viscosity, as changes thereof indicate a change...
in molecular weight. Also, tensile bars were produced via injection molding.

Figure 8 shows the results of the rotary rheometry for PA6-1, which are representative also for PA6-2 and PLA. The graph shows a decrease in viscosity for both, desiccant drying and MW drying, which indicates a reduction of molecular weight. For 50 °C, this is more pronounced, which can be explained by the fact that drying at 50 °C takes a lot longer than at 120 °C. It can be concluded that long exposure times are more harmful to the material than higher temperatures. The values for MW dried samples are slightly smaller than for the conventionally dried ones, but to such a small degree that it seems negligible. PHB and PET both showed an increase of zero shear viscosity after drying. This can be explained by the fact that after drying, moisture levels were lower than those as received and therefore chain build-up via condensation polymerization occurred during the measurements.

![Zero shear viscosity before and after drying](image)

Figure 8: Zero shear viscosity before and after drying

The mechanical testing did not show significant changes due to drying. The Young’s Modulus of both PA6 types and PET was slightly reduced for all dried materials, with hardly any differences between desiccant and MW drying. For PHB and PLA, however, MW drying led to significantly smaller values than conventional drying.

**Conclusions**

The findings of this work confirm that microwaves can successfully be applied for polymer pellet drying. Five different types of polymers, including highly sensitive bio-polymers, were tested and dried without visible damage or melting. It was shown that the effective power of the microwaves does not increase linearly with total power but from a certain point on the additional power is lost. This, however, was only investigated for PA6 and needs to be confirmed for other polymers.

Whereas drying speed is determined by temperature in conventional drying, this work could show that microwave application may lead to a temperature invariant drying speed. For some polymers this effect did only occur beyond a specific threshold temperature. It remains yet to be further investigated if this threshold can be explained with the thermal characteristics of the respective polymer, as for some of the tested materials it seemed to coincide with the glass transition, but not for all.

With the current pilot plant, drying speeds were higher at low temperatures in comparison to a conventional desiccant dryer, but not at higher ones. This does not necessarily mean that desiccant drying is superior at higher temperatures, but can be attributed to an inefficient design of the MW dryer prototype. When using dehumidified air, thus practically transforming the pilot plant into a desiccant dryer suffering from the same inefficiencies, the drying took longer than in combination with microwaves, even at higher temperatures. This demonstrates the potential of microwave application in drying and shows that the prototype needs further constructive improvement to fully exploit this potential.

Future works will investigate the combination of MW drying with ambient air as a convective medium instead of dehumidified air. As the diffusion of water particles is primarily driven by the microwaves, not the concentration gradient, it might not be necessary to use a desiccant unit, which will hold the potential for vast energy savings.

**Acknowledgments**

The findings presented in this paper were funded by European Union’s Seventh Framework Program under Grant Agreement No 606425. The Authors would like to thank the whole HiPerDry project consortium for the good und trustful cooperation.

**References**