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Characterisation and identification of plastics through microwave treatment and temperature measurement

M. Labbert[1], T. A. Baloun[1], J. I. Schoenherr[1] and H. Z. Kuyumcu[2]

[1] Hochschule Zittau/Görlitz, Institut für Verfahrensentwicklung, Torf- und Naturstoff-Forschung (iTN),
Friedrich-Schneider-Str. 26, D-02763 Zittau
[2] Technische Universität Berlin, Institut für Prozess- und Verfahrenstechnik,
Straße des 17. Juni 135, D-10623 Berlin

Abstract A research project on a thermo-sensitive sorting process was carried out at the Institute of Process Development, Peat and Natural Matter Research (iTN) of the Zittau/Goerlitz University of Applied Sciences to show a new way of recycling of plastics. The aim of the research is to evaluate the separability of plastics with a new sorting process and to clarify relevant influencing parameters. The laboratory tests involve microwave heating of plastic specimens in a cavity resonator at a frequency of 2.45 GHz and a non-contact temperature measurement by means of infrared detection. The results confirm the suitability of the thermo-sensitive sorting process to distinguish many different types of plastics and reveal the significant influence of parameters such as microwave heating time, microwave power, particle size, and water content on the differentiation of plastics on the basis of microwave heating.

1 Introduction

Plastics are organic polymers with excellent properties so that they are used in many applications today. Their production has increased steadily since the 1950s, leading to an equally steady rise of the amount of plastics waste [1]. To protect the environment and to conserve natural resources it is useful and desired by society and government in general that plastics waste is recycled as much as possible. For reuse, the quality of recycling products should be almost equivalent to virgin materials [2]. With today's sorting processes for plastics waste, for example gravity separation and near-infrared spectroscopy, many recycling tasks are being performed, but due to various problems such as overlapping property values and compounds, they are not yet capable to reach maximum recycling rates and product qualities. Therefore, it is necessary to optimise plastics recycling by utilising other separation characteristics of the materials.

2 Basic idea of the thermo-sensitive sorting process

In the thermo-sensitive sorting process, plastics are heated selectively according to their dielectric properties in a microwave oven followed by non-contact measurement of the thermal radiation emitted from the particles' surface by infrared detection [3, 4]. The tailor-made control software converts the measured irradiation values into temperature values. By means of temperature differences the materials can be identified and differentiated from each other. The separation of particles meeting the separating criterion can be realised by air nozzles.

3 Experimental

The employed materials, the test specimen, and the materials preparation as well as the performance of microwave heating and cooling experiments are described below.

3.1 Materials

Research was carried out with 9 different types of plastics (cf. Table 3.1). These are semi-finished products obtained from REIFF Technische Produkte GmbH, Reutlingen, Germany. Some of the plastics are available in various colours. In the following, the sample materials used are characterised by the abbreviation of the plastics type and the colour. The colour abbreviations are (according to [5]): BK black, GN green, GY grey, RD red, TR transparent, WH white, lBN light brown, dBN dark brown. Red polyvinyl chloride, for example, will be referred to as PVC_RD.

Table 3.1: Employed plastics, their colours and related cooling constants *a*.

Abbreviation	Name	Colour	a in s^{-1}
PTFE	Polytetrafluoroethylene	WH	0.0036
PE	Polyethylene	WH/GN/BK	0.0039/0.0043/0.0041
PP	Polypropylene	WH/GY	0.0044/0.0046
PMMA	Poly (methyl methacrylate)	TR	0.0043
PC	Polycarbonate	TR	0.0050
PVC	Polyvinyl chloride	RD/GY/BK	0.0047/0.0049/0.0047
PA 6	Polyamide 6	WH/BK	0.0039/0.0040
POM	Polyoxymethylene	WH/BK	0.0037/0.0036
PUR	Polyurethane	lBN/dBN	0.0037/0.0037

The cooling constant a of a sample depends on the thermal properties of the material, the sample volume, and the sample surface. Values listed in Table 3.1 are determined by cooling experiments. The specimens employed are cylindrically shaped with a height of 5 mm and a diameter of 5, 10, 15, or 20 mm. For sample preparation, the polymers were dried at 50 °C in a drying oven (according to [6]). To evaluate the influence of different materials' water content on microwave heating, specimens of selected plastics were placed in deionised water for different periods of dwelling.

3.2 Procedures

Microwave tests were carried out in a resonant cavity developed inhouse at a frequency of 2.45 GHz. The plastic specimens were placed on a carrier made of PTFE in the range of the largest field strength in the resonator. During microwave heating, the temperatures were directly measured at the centre of the samples' surfaces by an infrared camera (detection wavelength λ: 8-14 μm). The microwave power, the specimen diameter, and the water content were altered. In addition to microwave heating tests, cooling experiments were carried out as well using a temperature chamber. The plastic specimens with diameters of 20 mm were heated to a temperature of 50 °C and the surface temperature was measured during the cooling process by an infrared camera. On the basis of these experiments, the cooling constants a of the individual plastics types are determined. Cooling constants are used to calculate the cooling rates.

4 Results and discussion

Experimental results show that there is an influence of individual materials' characteristics on microwave heating which can be used in thermo-sensitive sorting processes. Thereafter the procedure in differentiating plastics using their remaining temperatures is explained.

4.1 Microwave heating behaviour and influence of the microwave heating time

The plastics exhibit different temperature increases in microwave heating, as seen in Figure 3.1. This behaviour is a result of different dielectric properties of the plastics, which are defined by their chemical structure [7]. Non-polar materials such as PTFE, PE and PP show no significant warming. In contrast, polar materials such as PA 6 and POM heat up fast and to a considerable degree.

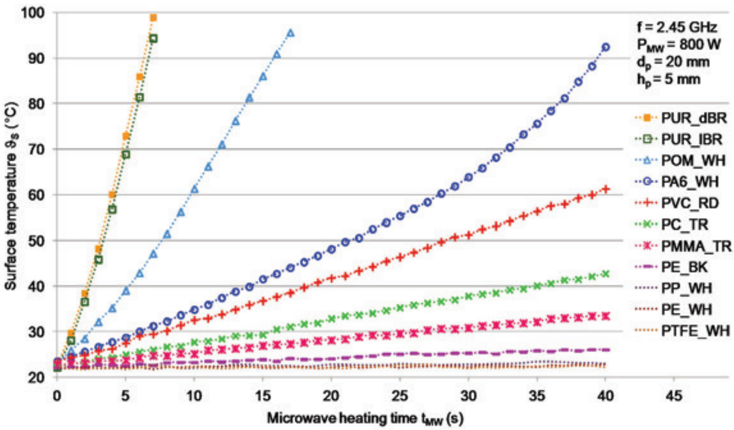

Figure 3.1: Surface temperatures during microwave heating.

However, all plastics show a significant dependence on the residence time t_{MW} within the microwave field. A longer microwave heating time was found to cause higher measured surface temperatures and larger temperature differences between the plastics types. Most of the plastics variants show nearly the same heating profiles, with the exception of polyethylene. PE_WH heats up only slightly, which can be explained by its basis of non-polar chemical structure. In contrast, PE_BK shows a much larger change in temperature, an effect which might be caused by additives contained in PE_BK which lead to a stronger microwave heating. A well-known example of a substance which improves the heatability of non-polar plastics is carbon black [8, 9]. The slopes of the heating or temperature curves are not constant. This is due to the change in dielectric materials properties [10]. The cooling rate may show changes as a function of temperature.

With regard to the thermo-sensitive sorting process many plastics can be distinguished from each other due to the different dielectric properties and the associated microwave heatability. A longer microwave heating time promotes the differentiation between the plastics types. Due to strong heating of some plastics such as PUR and POM, a step-by-step identification and sorting process is required.

4.2 Influence of the specimen temperature on the microwave heating

The microwave heating rate, in this paper referred to as v_{MW}, is the change in the measured surface temperature $d\vartheta_S$ in the time period of dt. Figure 3.2 shows the microwave heating rate of selected plastics as a function of surface temperature.

The microwave heating rates of polymers, which are very strongly heated in the microwave field, such as PUR, POM, PA 6, and PVC show a significant increase in their surface temperature. This can be explained by the reduced physical bindings within the matter, e.g. between the dipoles and their improved mobility [10]. In case of plastics which are less heated by microwave irradiation, such as PC and PMMA, the heat transfer to the ambient air is larger than the increased heating due to improved dipole mobility. Therefore, the microwave heating rate rather decreases with increasing temperature. The microwave heating rate can be seen as the difference of the dielectric heating rate and the materials cooling rate. This relation is shown in Equation 3.1, Equation 3.2, and, simplified in Equation 3.3.

$$\left(\frac{d\vartheta_S}{dt}\right)_{MW} = \left(\frac{d\vartheta_S}{dt}\right)_{diel} - \left(\frac{d\vartheta_S}{dt}\right)_{cool} \tag{3.1}$$

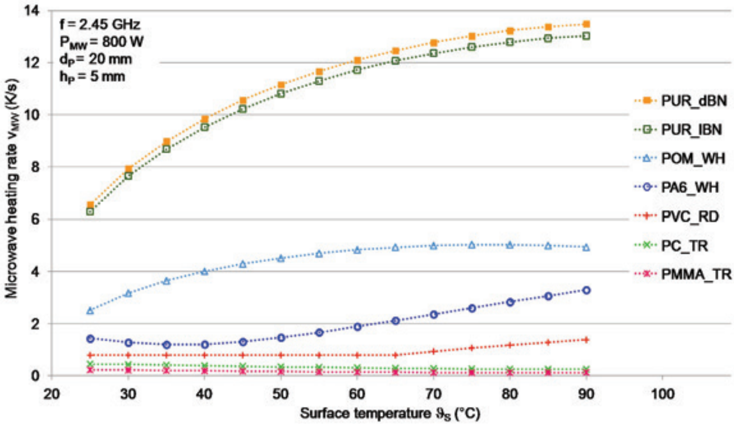

Figure 3.2: Microwave heating rate as a function of surface temperature.

$$\left(\frac{d\vartheta_S}{dt}\right)_{MW} = \left(\frac{d\vartheta_S}{dt}\right)_{diel} - a \cdot (\vartheta_S - \vartheta_U) \tag{3.2}$$

$$v_{MW} = v_{diel} - v_{cool} \tag{3.3}$$

The dielectric heating rate v_{diel} describes the temperature increase due to the interaction of the dipoles with the electric field and the cooling rate v_{cool} describes the temperature decrease due to the heat released to the ambient air. The latter results out of the fact that the ambient air is not heated by microwave radiation (ϑ_U = const.). The cooling rate can be calculated by using the cooling constants of the materials, which depend on the thermal properties of the material as well as the size and shape of the specimens. As seen in Figure 3.2, the microwave heating rate of PVC_RD rises suddenly at the temperature of 65 °C. The reason is the glass transition of the material in this temperature range. From this temperature range on the dipoles of an amorphous material have an increased mobility and can align better in the electric field. This leads to a stronger heating by the microwave irradiation.

With regards to the sorting process, it should be noted that temperature has a significant influence on the microwave heating of the plastics. However, the risk of material destruction results from an increase in microwave heating rates (thermal runaway).

4.3 Influence of the applied microwave power on the microwave heating

Tests show that the applied microwave power has a noticeable influence on the microwave heating of plastics. An increase of the microwave power leads to a more or less considerable increase in the microwave heating rate (see Figure 3.3).

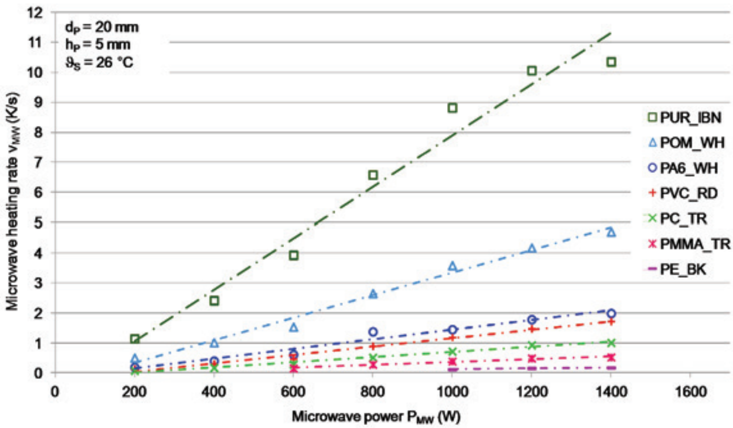

Figure 3.3: Microwave heating rate as a function of the fed microwave power.

The average power absorbed by an object in an electric field is given by Equation 3.4 [11].

$$P_{MW} = \omega \cdot \varepsilon_0 \cdot \varepsilon_r'' \cdot E^2 \cdot V \tag{3.4}$$

Absorbed power depends on the angular frequency ω, the permittivity of the vacuum ε_0, the dielectric loss ε_r'' of the material, the electric field strength E, and the volume V of the object. The increased heating rate due to increased applied power results from higher power consumption by the material. If the other parameters of Equation 3.4 are constant, an increase in the electric field strength is indicated.

With regard to the sorting process, an increase in the applied microwave power has a positive effect due to larger differences in microwave heating rates, resulting in larger temperature differences and improved differentiation between the plastics.

4.4 Influence of the sample size on the microwave heating

A closer look at Equation 3.4 shows the dependence of the absorbed power of an object in the microwave field on its volume or even size. The experiments show that a larger sample diameter, and therefore also a larger sample volume, leads to increased microwave heating rates. Figure 3.4 shows the microwave heating rates of POM_WH, PVC_RD, and PC_TR as a function of the sample volume.

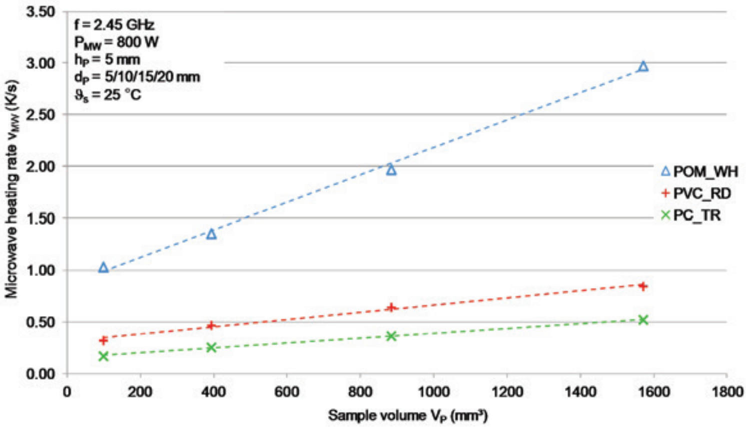

Figure 3.4: Microwave heating rates as a function of the sample volume.

In addition to higher energy absorption, the change in volume results in different cooling characteristics. Smaller particles are said to have a higher cooling rate based on their larger surface-to-volume ratio than the larger particles, since the heat loss to the environment is large if the particle surface is big.

With regard to the sorting process, the particle size, and hence the particle volume and mass, have to be known in order to distinguish the types of plastics using their individual heating behaviour. This can be implemented by the determination of the particle size during the running process or by ensuring almost the same particle sizes by a narrow screening.

4.5 Influence of the water content on the microwave heating

Plastics are able to absorb different amounts of water. Since water molecules are dipoles, their presence influences the microwave heating of the materials dramatically. The experimental results show that the more water is contained in the plastic, the larger is the microwave heating rate. Figure 3.5 shows the microwave heating rates as a function of the gravimetric water content (dry state) for selected plastics.

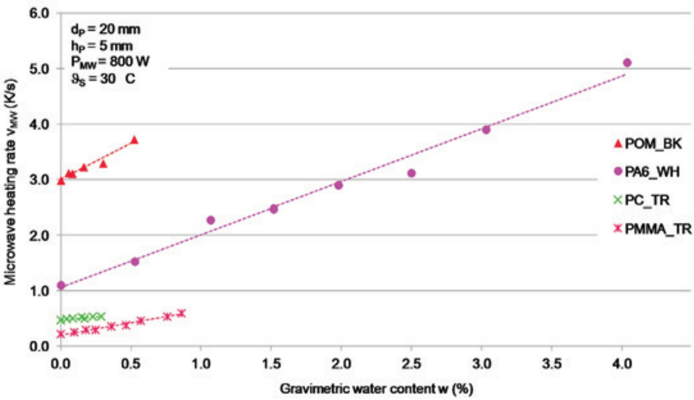

Figure 3.5: Microwave heating rates as a function of the gravimetric water content (dry state).

Under certain circumstances, the water content affects the distinguishability of the plastics. This means that several types of plastics, which undergo different temperature increases in the dried state, show in the moist state an almost identical heating behaviour. PC_TR and PMMA_TR, for example, show the same microwave heating rate if PC_TR has a water content between 0 and 0.3 % and PMMA_TR between 0.63 and 0.77 %. This means the temperature increase by microwave heating is almost the same for both plastics and differentiation between them is rather impossible.

With regards to the sorting process, water content has a significant influence on the microwave heating behaviour and the distinguishability of plastics. Therefore, it is necessary to create defined conditions in terms of water content, e.g. by pre-drying of the material.

4.6 Procedure for the distinction of plastics in the thermo-sensitive sorting process

The temperature profiles of two different plastics during the entire thermo-sensitive sorting process are shown schematically in Figure 3.6. In the microwave heating phase, between t_{in} and t_{out}, the plastics are heated selectively according to their dielectric materials properties. After leaving the microwave oven at t_{out}, there will be a gradual levelling of particle temperature to the temperature of the ambient air. The rate of cooling depends on the thermal properties of the material, the ambient temperature, the particle shape, and the particle size, and can be calculated using the cooling constant a.

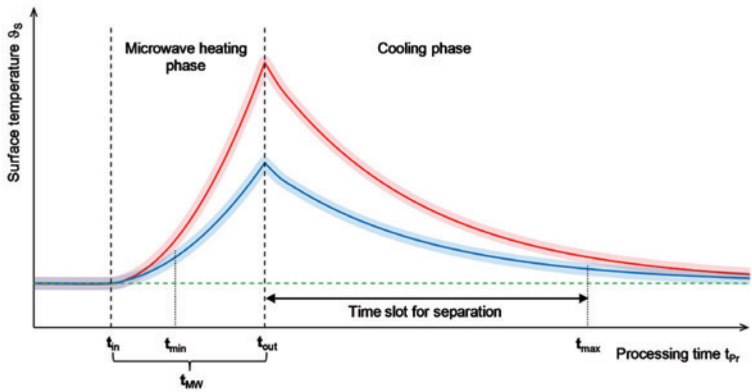

Figure 3.6: Temperature profiles of two different plastics during thermo-sensitive sorting process, schematically.

Within the thermo-sensitive sorting process, the distinction between the plastics should be made on the basis of their temperature differences. For this purpose, the temperature curves of the materials during the microwave heating and subsequent cooling stage have to be determined, applying the function of the applied microwave power, the water content, the particle size, and the particle shape. In order to take the signal noise into account, the confidence intervals around the curves are designed to include almost 95 % of the measured temperature values. Therefore, the distinction between two plastics is possible, as soon as their confidence intervals do no longer overlap (t_{min}). The larger the difference between t_{min} and t_{out}, the larger the temperature differences between the plastics and the longer the period to have their confidence intervals overlap again (t_{max}) after leaving the microwave oven. For the thermo-sensitive sorting process, infrared detection is intended to take place after leaving the microwave oven, resulting in a time slot between t_{out} and t_{max} in which the plastics can be identified and distinguished.

The described evaluation procedure was used for the differentiation of plastic specimens having the same shape (cylindrical), size ($d_P = 20\,mm$, $h_P = 5\,mm$), and water content ($w = 0\,\%$). The separation criterion is that the difference between t_{out} and t_{min} is larger than zero after 10 s microwave heating. Immediately after the microwave heating infrared detection takes place ($t_D = t_{out}$). The studies suggest that the sorting process should be done step by step. Based on the findings, the following approach is recommended. First the plastic types heated very strongly, such as POM and PUR, are distinguished and separated from the other plastics at low microwave power levels ($P_{MW} = 200\,W$). In each subsequent step, the microwave power is increased. Step by step, PA 6, PVC, PC, and PMMA can be separated from the mixture. At the end of the process, a mixture of the plastics only heated insignificantly (PMMA, PE, PP) remains. Benefits of this gradual separation are the reduction of the remaining amount of materials to be sorted and the prevention of material destruction by overheating.

5 Conclusions

Various plastics differ in their microwave heating behaviour due to different dielectric properties. That allows the identification and the differentiation between the plastics samples based on the temperatures reached. In addition to the dielectric properties, other parameters have significant impact on the microwave heating of the plastics and hence the distinction. These influencing parameters are microwave power, microwave heating time, the particle size and shape, additives, and the water content. The requirements for a successful plastic separation by a thermo-sensitive sorting process are a narrow screening to ensure almost identical particle sizes and shape, the drying of the materials, the adjustment of microwave power and microwave heating or duration time on the sorting materials, and a step-by-step separation. When meeting all requirements and employing sophisticated microwave system technology, the thermo-sensitive sorting process has great potential to run proper plastics recycling and to increase recycling rates and improved product qualities of recycled plastics.

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