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Microfoaming of flax and wood fibre reinforced polypropylene composites

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Abstract Natural and wood fibre reinforced plastics as a relatively new group of environmental friendly materials have been extensively applied in interior, building applications and in the automobile industry. Among others, natural and wood fibre reinforced foamed polymer materials are of high significance because of the possibility of their reducing the density of automotive components which have a cellular structure. However, the properties of these materials have not been fully investigated and described. Microcellular composites of polypropylene containing natural and wood fibre was prepared using an injection moulding process. In the present work, the manufacturing technology of natural and wood fibre reinforced polymer microfoams was developed and the influence of fibre and microvoid content on its property spectrum was systematically investigated. The forming of microvoids and the degree of foaming related to the variation of the processing parameters in connection with manufacture technology was characterised. Measurement of density, cell size, tensile and flexural properties of the prepared composites was carried out. The cell structure is dependent on flow direction of foaming. The density of microfoamed wood fibre-PP composites was reduced by about 24% and decreased by as much as 0.77 g/cm^3. Light microscopy showed that the cells are circular and it was also observed that the maximum cell sizes are between 10–50 μm. Water absorption and scanning electron microscopy of the composites were also investigated. Considering the experimental results, it can be deduced that the microcellular composites exhibit a possible combination of relatively good engineering properties and lower density for different technical applications.

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Mikroverschäumung von Flachs- und holzfaserverstärkten Polypropylen-Compositen

Zusammenfassung Natur- und holzfaserverstärkte Kunststoffe, als relativ neue Gruppe von umweltfreundlichen Werkstoffen, werden derzeit in beachtlichem Umfang in der Bauindustrie (Dach-, Boden- und Wändeabdeckungen) und Automobilindustrie verwendet. Ausserdem haben natur- und holzfaserverstärkte Polymerwerkstoffe –Mikroschäume, die wegen ihrer zellularen Struktur die Dichte der Bauteile noch reduzieren können, eine immer größere Bedeutung erlangt. Bisher sind die Eigenschaften dieser Werkstoffe nur beschränkt untersucht und beschrieben worden. Natur- und holzfaserverstärkte PP-Mikroschäume wurden im Spritzgießverfahren hergestellt. Im Rahmen der vorliegenden Arbeit wurde die Fertigungstechnik der natur- und holzfaserverstärkten Polymere-Mikroschäume entwickelt und systematisch der Einfluss des Faser-/ und Mikroporengehalts auf das Eigenschaftsspektrum dieser Werkstoffe untersucht. Mikroporen und Schäumungsgrad in Abhängigkeit von der Variation der Verarbeitungsparameter wurden in Verbindung mit dem Herstellungsprozess charakterisiert. Die Dichte, die Zellgröße, die Zug- und Biegeparameter der hergestellten Compositen wurden ermittelt. Die Schaumstruktur ist vom Fließweg abhängig. Die Dichte von geschäumten holzfaserverstärkten PP kann bis ca. 24% reduziert werden und erreicht Werte von 0,77 g/cm^3. Die mikroskopischen Aufnahmen zeigen, dass die Mikroporen eine runde Struktur aufweisen, und der Porendurchmesser zwichen 10–50 μm liegt. Die Wasseraufnahme wurde ebenfalls untersucht. Die erzielten Ergebnisse belegen, dass diese Werkstoffe für verschidene Anwendungen in der Technik geeignet sind.

1 Introduction

Recent research and developments (Sperber et al.2002) show that natural and wood fibre-plastic composites represent a relatively small but rapidly growing industry such as in the Caribbean, where a project was taken up to prepare 50,000 houses from wood-plastic composites which are specially durable against hurricanes. These composites were first introduced to the market quite some time ago, and they have become more widely accepted recently. Although the natural and wood fibre-plastic composites have been commercialised, their potential for use in many industrial applications is limited. The potential range of uses for these materials in innovative applications would expand if these shortcomings could be met. Recently, the concept of creating microcellular-foamed structures in the composites has been successfully demonstrated. Foamed plastics can often be stronger than their non-foamed analogues (Barth 2000) and, because of the reduced weight, can achieve outstanding cost-to-performance and favourable strength-to-weight ratios (Thorne 1996). Due to these unique properties, microcellular plastics can be used in many industrial applications, including light-weight, high-strength parts for the automotive and aerospace industries, containers, sporting goods and thermal and electrical insulators.

The production of microcellular-foamed structures in wood fibre-PVC (poly vinyl chloride) composites through a batch-foaming process (Matuana et al. 1998, 1997, 1996, 2001abc) was investigated. The batch-foaming process used to generate cellular-foamed structures in the composites is not likely to be implemented in the industrial production of foams because it is not cost-effective. The microcellular-batch-foaming process is time consuming due to the multiple steps involved in the production of foamed samples.

Considering these shortcomings, the manufacture of polymer-wood fibre composite foams in an extrusion process needs to be investigated. Relatively, only limited research has been conducted and there is very little information available on investigation of the foamability of polymer-wood fibre composites through a continuous extrusion process (Park et al. 1999; Matuana et al. 1999, 2001a, b, c).

Park et al. (2001) experimented on two-system configurations (tandem extrusion system vs. single extruder system) for wood fibre-HDPE (high-density polyethylene) composites to demonstrate the system effect on the cell morphology and foam properties. Natural fibre (jute and flax fibre) reinforced epoxy foams (Bledzki et al. 2001), polyurethane microfoams (Bledzki et al. 2001) were also introduced.

Injection moulding is one of the most commercially important fabrication processes for moulding a broad spectrum of thermoplastics. A great deal of attention has been paid to defining the engineering aspects of the operation for maximising production rates and for controlling part strength, brittleness, shrinkage and appearance characteristics (Baer 1964, Bernhardt 1959). Unfortunately, no literature was available on injection moulding process of microcellular foaming in PP (polypropylene) containing natural fibre (flax fibre), except for this work (Zhang 2001), which is the first from this institute.

There are several variables to consider when operating an injection moulding machine. Some of these variables can affect the physical properties of the microfoam. It is well established, for example, that the mould temperature and curing time are important variables in this regard. However, there are many other factors that can be adjusted, including such variables as screw speed, compression pressure, cycle time to name a few, which might also have an effect on one or more microfoam properties.

Foamed polymers can be produced by utilizing either a chemical or a physical foaming agent. Chemical foaming agents were used due to a small variation in comparison with compact injection moulding procedures are requested. Chemical foaming agents are substances which decompose at processing temperatures thus liberating gases like CO_2 and/or nitrogen. There are several different types of chemical foaming agents, which differ mostly in the type of gas that is generated and the type of reaction that generates that gas. The reaction that produces the gas can either absorb energy (endothermic) or release energy (exothermic). This presentation will also explore the effects of chemical foaming agents on properties also.

The concept of creating microcellular structures in the flax fibre-PP and wood fibre-PP composites through an injection moulding process will be presented. To achieve the desired properties, it is necessary to optimise the processing parameters and the feasibility of using flax and wood fibre as a filler for microfoaming in polymer.

2 Experimental

2.1 Materials

The material used in our experiments is polypropylene containing short flax and wood fibre. The short flax fibre (15–25 mm) and standard hard-wood fibre (150–500 μm) were supplied by Mühlmeier GmbH and J. Rettenmaierhne GmbH + Co. respectively.

The polypropylenes were manufactured by DSM, Gelsenkirchen, Germany and described in detail in Table 1.

To get foamed natural and wood fibre reinforced composites, four types of chemical foaming agents have been used in this research work and they were obtained from Lehmann Co. The characteristics of these chemical foaming agents are listed in Table 2.

Table 1 Different types of polypropylenes
Tabelle 1 Verwendete PP-Typen

PP Types	Density [g/cm^3]	Melting Index [230°C;2.16 N] g/10 min	Izod Imapct Strength kJ/m^2
Stamylan 16M10	0.905	5.7	2.7
Stamylan 17M10	0.905	10.5	2.0
Stamylan 112MN40	0.905	47	2.0
Stamylan 213MNK40	0.905	90	2.5

Table 2 Different types of chemical foaming agents
Tabelle 2 Verwendete chemische Treibmittel

Used Name	Commercial Name	Reaction Type	Decomposition Temp. [°C]	Gas Yield ml/g
T1	LUVOPORAZ/40 G-UT	Exothermic	165	80
T2	LUVOPOR 9241	Exothermic	200	140
T3	LUVOPOR 9341	Exo-/Endothermic	140	110
T4	LUVOPOR ETMF 20/G-PE	Endothermic	155	25

With the intention of improving the mechanical properties of wood fibre-PP foamed product, a compatibilizer maleic anhydride polypropylene copolymer (LOCIMONT FG 504) was used and it was obtained from Clariant Corp., Frankfurt, Germany.

2.2 Processing and foaming

The processing of natural and wood fibre-PP microfoaming can be divided into three steps. The processing steps are as follows.

1. *Agglomeration of polypropylene with flax and wood fibre* Short flax fibre (30% by weight) and different types of PP were mixed using a mixer (Henschel Mixer) and the hard-wood fibres with PP were compounded by a twin-screw extruder (Haake Extruder, Rheomex PTW 25/32) with and without the coupling agent. Both the flax and wood fibres were dried at 100°C and 80°C in an air-circulating oven for 2 and 24 h respectively, before mixing.
2. *Homogenisation of agglomerate granules with chemical foaming agents* Then cold agglomerate granules mixed with different types of chemical foaming agents and before foaming in injection moulding, the mixed granules were dried again in the same manner as indicated earlier.
3. *Foaming and preparation of specimen in an injection moulding process* The specimens [Geometry. 200×90×4(2) mm] of both flax and wood fibre foamed composites were prepared by injection moulding process. The processing temperature was fixed at a temperature where the dispersed gas bubbles create the necessary expansion after injection. The expansion pressure sprayed the melt against the cold wall of the mould, creating the typical structure that includes solid skin and cellular core. The decomposition reaction of chemical foaming agents (azodicarbonamide) is stated below.

$$H_2N{-}CO{-}N{=}N{-}CO{-}NH_2 \rightarrow N_2\uparrow + CO \uparrow + H_2NCONH_2$$

When heated N_2 and CO are liberated from the decomposition of azodicarbonamide. The liberated N_2 and CO from the decomposition can act as a blowing agent.

2.3 Measurements

All physicomechanical tests were performed according to standard testing methods. The tensile and flexural tests (Zwick Machine, UPM 1446) were tested at a test speed of 2 mm/min according to DIN EN 61 and DIN EN 63 for flax fibre-PP composites, respectively, also according to EN ISO 527 and EN ISO 178 for wood fibre-PP composites, respectively. All the tests were done at room temperature (23°C) at a relative humidity of 50%.

The densities of non-foamed and microfoamed specimens were measured according to DIN 53479 and 15 replicates were used for each variable studied and the mean value was taken. The void content was calculated according to the ASTM D 2734–70 for foamed composites.

2.4 Water uptake

The water uptake of microfoamed and non-foamed samples was measured as a function of time according to DIN 53495. Specimens (50×50×4 mm) were kept at the bottom of a water-filled container at 23°C for varying lengths for flax fibre-PP composites (1–300 h) and for wood fibre-PP composites (1–672 h) of time. The percent of water absorbed was calculated.

2.5 SEM and light microscopy

The morphology of the flax and wood fibre reinforced microcellular PP composites and the cell size, shape and distribution of voids in microfoamed composites were investigated using the scanning electron microscope

(SEM) (CAM SCAN 4DV) and a light microscope. Cross sections of rough and polished surfaces were studied with light microscope, whereas fractured surfaces of flexural test samples were studied with SEM after being sputter coated with gold.

3 Results and discussion

3.1 Structure

The microfoam structure of foamed composites in various flax and wood fibre content was influenced by the mould and the melting temperature in injection moulding process. The foaming area and the distribution of cells are shown in light micrographs for flax fibre reinforced PP microfoamed composites (in Fig. 1a) which is also observed for wood-PP microfoamed composites (in Fig. 1b).The micrographs illustrated that the microfoamed structure, near the feeding point like a three-layer sandwich structure, contained a middle layer with distributed cells and identified a compact outer hull. In between the foaming area and the surface layer, there developed a transition zone where microcells ride from feeding point to the side area.

The microcells were distorted in this transition zone along the direction of flow at the boundary layer to the cooled down edge of the skin end. Such deformation of the microcells arises, preferentially, at spraying poured thin-walled shaped parts with long flow way, which is observed in Fig. 2a and 2b. Also the chain molecules of the thermoplastics are aligned by shears of the melt in the direction of flow. This orientation has large influence on the structure and mechanical characteristics of the construction unit (Tadmor 1974, Uejo et al. 1970, Wiegand et al.1966, 1967, Ischebeck 1984).

The form and the diameter of the microcells on the surface of fractured specimen were documented by means of the scanning electron microscope (SEM). The SEM micrographs in Fig. 3a and 3b showed that the diameter of the microcells is most often approximately 50 µm for both flax and wood fibre-PP microfoamed composites.

3.2 Density

Figure 4 shows a comparison of four chemical foaming agents that were used (table 2) and their influence on density and microvoid contents for flax fibre-PP microfoamed composites manufactured with a comparable gas yield. The results point out the comparable foam ability for all these chemical foaming agents. The density and the microvoid content did not differ significantly. The chemical foaming agent (T2) is used for further investigations.

With this chemical foaming agent (T2), the wood fibre-PP microfoamed composites were prepared and Fig. 5 shows that with the increase of wood fibre content, density also increases in all cases due to the different density of PP and wood fibre. Microfoamed composites showed lower density compared to the non-foamed composites and with the addition of MAH-PP5%, density reduced at most 25–30% and decreased up to 0.776 g/cm^3.

The change of the relationship from the foamed core to the compact outer skin is to be understood as a measure of the foaming grade, which changes the microvoid content and the density. The thicker core has higher density and lowers the microvoid content. A typical dependence of the density on the microvoid content of the flax fibre and wood fibre reinforced PP microfoamed composites showed that density decreases with the increase of microvoid content.

Density can be affected by the variation of the processing parameters. Figure 6 shows an example of the meaning of the fluidity of the melt (change of the flow front speed). It is evident that with the increase of the front flow speed, the density is reduced and the microvoid content is increased.

Figure 7 illustrates the influence of the mould temperature on the density and the microvoid content. An increase of the mould temperature means a reduction of the difference between the melt temperature and the mould temperature and thus enlargement of the foamed core range. An increase of the mould temperature caused by 80°C on 120°C for a 2-mm-thick plate and an acceptance of the density around 11% and an increase of the microvoid content around 172%. Both the parame-

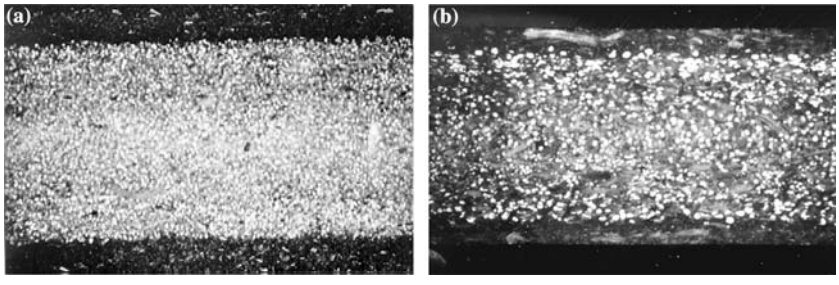

Fig. 1 a, b Typical microfoamed structure of flax fibre-PP (**a**) and wood fibre-PP (**b**) composites [fibre content. flax 30% and wood 40% by weight] [magnification 12.5.1]
Abb. 1 a, b Typische Schaumstruktur für die flachsfaserverstärkten PP-Mikroschäume (**a**) und Holzfaserverstärkten PP-Mikroschäume (**b**) Fasergehalt : Flachs 30 Gew.-% und Holz 40 Gew.-%, Vergrößerung: 12,5:1]

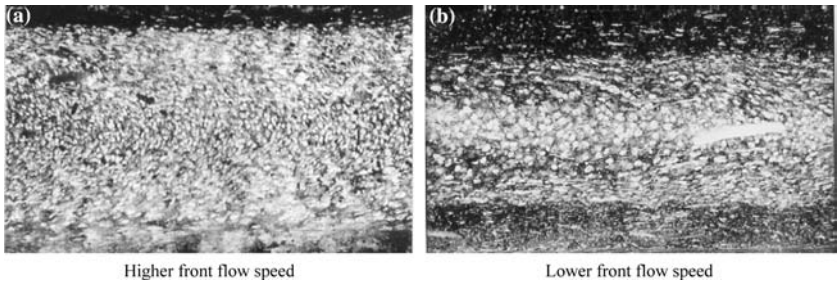

Fig. 2 a, b Flow direction in foamed structure of flax fibre-PP microfoamed composites [fibre content. 30% by weight, magnification 15.1]
Abb. 2 a, b Schaumstruktur in der Fließrichtung für flachsfaserverstärkte PP-Mikroschäume [Fasergehalt : 30 Gew.-% Vergrößerung: 15:1]

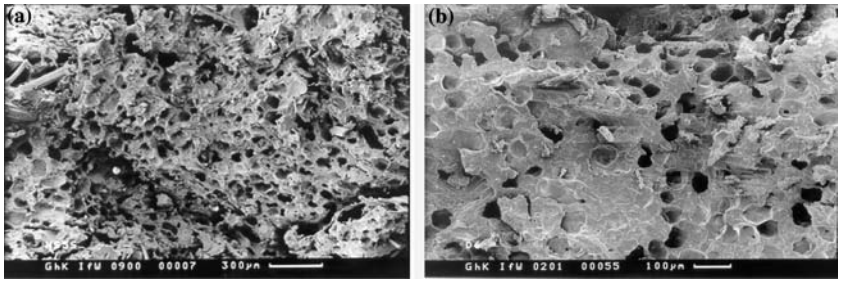

Fig. 3 a, b Cell size measurement of flax fibre-PP microfoamed composites (**a**) and wood-PP microfoamed composites (**b**) [fibre content. flax 30% and wood 40% by weight]
Abb. 3 a, b Zellgröße-Abmessung der geschäumten Flachsfaser-PP Composite (**a**) und geschäumten Holzfaser-PP Composite (**b**) [Fasergehalt : Flachs 30 Gew.-% und Holz 40 Gew.-%]

ters influenced the nucleation and growth of the microcells which affect the mechanical properties of the composites' dependence on the density.

3.3 Mechanical properties

The characteristic values of various types of PP with non-foamed flax fibre and wood fibre reinforced differed only slightly. Tensile strength and tensile modulus of flax-PP and wood-PP non-foamed composites were in the range of 26–29 MPa and 2800–3800 MPa, respectively. Also the flexural strength and the flexural modulus lay in the comparable range.

The mechanical characteristic values of the flax fibre-PP microfoamed composites are affected by microvoid content. With the increase of microvoid content of 0–20 vol% (density reduction around 20%), a reduction of the flexural modulus takes place only around 10–15% and the flexural strength around 25% because of the fine cell structure, which is illustrated in Fig. 8. These characteristic values for the two other PP sorts remain in the comparable range. In the case of wood fibre-PP composites, specific flexural strength follows a similar trend, that is flexural strength reduced proportionately in the microfoamed composites, which is illustrated in Fig. 9.

Specific flexural strengths for all wood fibre-PP composites samples were calculated by taking the ratio of flexural strength to its density. With the addition of the coupling agent MAH-PP(5%) in the foamed com-

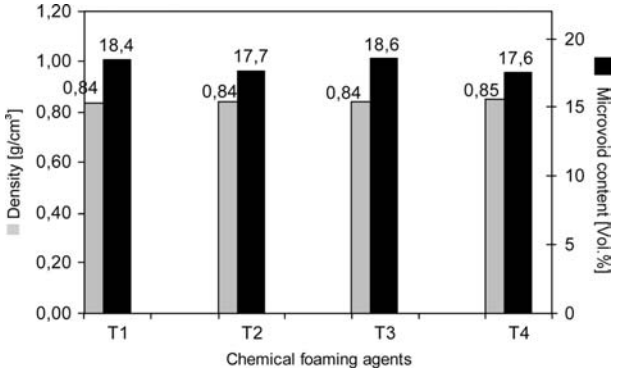

Fig. 4 Influence of chemical foaming agent on density and microvoid content of flax fibre-PP microfoamed composites [fibre content.30% by weight, temp. 190°C]
Abb. 4 Einfluss der chemischen Treibmittel-Typen auf die Dichte und Mikroporengehalt der geschäumten Flachsfaser-PP Composite [Fasergehalt : 30 Gew.-% Temp. 190°C]

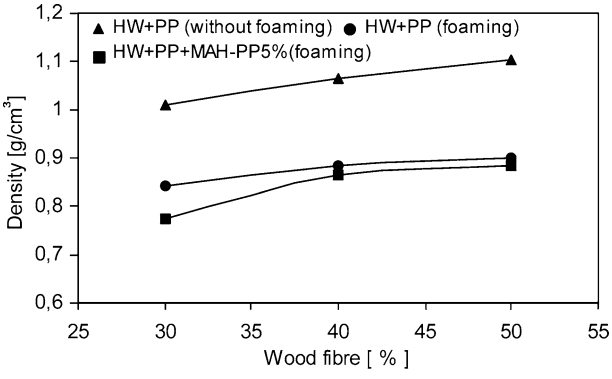

Fig. 5 Density of non-microfoamed and microfoamed wood-PP composites with and without coupling agent
Abb. 5 Dichte der ungeschäumten und geschäumten holzfaserverstärkten PP-Composite mit und ohne Haftvermittler

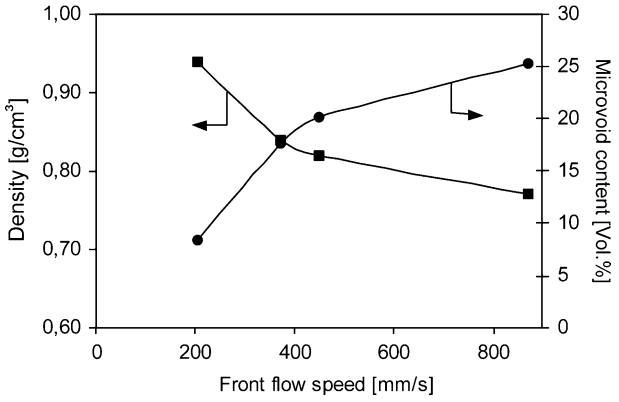

Fig. 6 Effect of front flow speed on density and microvoid content of flax fibre-PP microfoamed composites [fibre content. 30% by weight.]
Abb. 6 Einfluss der Fließfrontgeschwindigkeit auf die Dichte und den Mikroporengehalt der geschäumten Flachsfaser PP- [Fasergehalt: 30 Gew.-%]

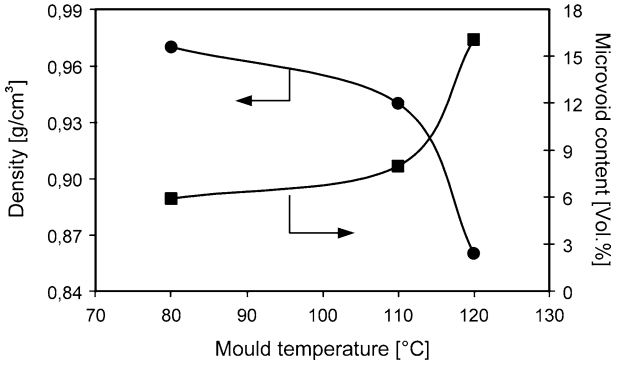

Fig. 7 Effect of mould temperature on density and microvoid content of flax fibre – PP microfoamed composites [fibre content. 30% by weight.]
Abb. 7 Einfluss der Werkzeugtemperatur auf die Dichte und den Mikroporengehalt der geschäumten Flachsfaser PP- [Fasergehalt: 30 Gew.-%]

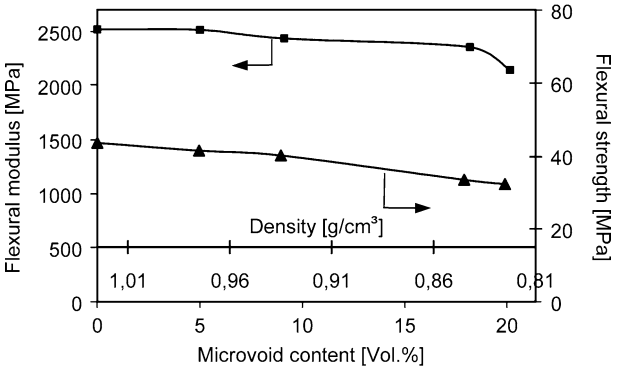

Fig. 8 Flexural properties of flax-PP microfoamed composites dependence on density and microvoid content [fibre content. 30% by weight.]
Abb. 8 Einfluss der Dichte bzw. des Mikroporengehalts auf die Biegefestigkeit und den Biege-E-Modul der geschäumten Flachsfaser PP- [Fasergehalt: 30 Gew.-%]

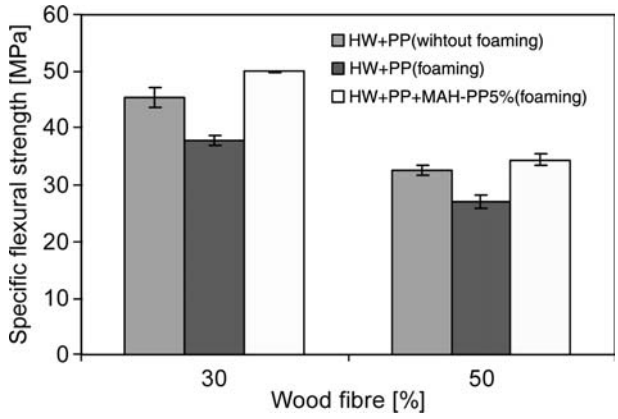

Fig. 9 Specific flexural strength of microfoamed and non-foamed wood-PP composites with and without MAH-PP5%
Abb. 9 Spezifische Biegefestigkeit der ungeschäumten und geschäumten holzfaserverstärkten PP-Composite mit und ohne MAH-PP5%

posites, specific flexural strength increased to 33% at wood fibre contents 30% by weight. In our previous work (Bledzki et al. 2002), it was investigated that the coupling agent MAH-PP showed best performance in the concentration of 5% with the wood fibre-PP composites.

Figure 10 shows the increase of the microvoid content of 0–20 vol% causes with the tensile characteristic values that the tensile strength and the tensile modulus decrease only 5–15% maximum.

The specific tensile strengths for both microfoamed and non-foamed wood fibre-PP composites are summarised in Fig. 11 and these are parallel to the specific flexural strengths just described. Again, specific tensile strength decreased with the increase of wood fibre content and similarly specific flexural strength decreased in microfoamed composites and increased 50% with the

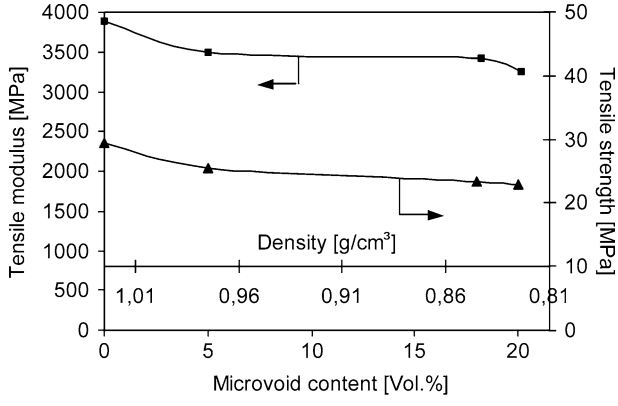

Fig. 10 Tensile properties of flax-PP microfoamed composites dependence on density and microvoid content [fibre content. 30% by weight.]
Abb. 10 Einfluss der Dichte bzw. des Mikroporengehalts auf die Zugfestigkeit und den Zug-E-Modul der geschäumten Flachsfaser PP-Composite [Fasergehalt: 30 Gew.-%]

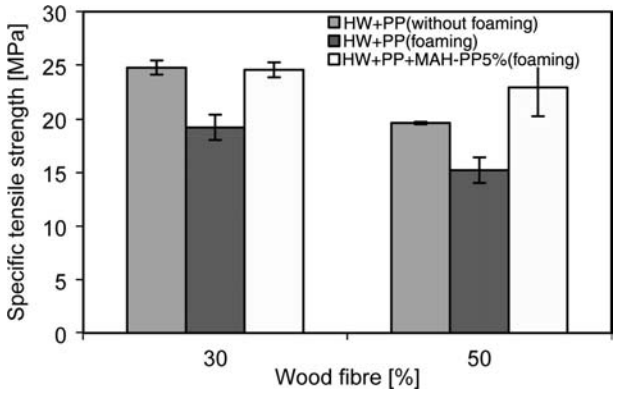

Fig. 11 Specific tensile strength of microfoamed and non-foamed wood-PP composites with and without MAH-PP5%
Abb. 11 Spezifische Zugfestigkeit der ungeschäumten und geschäumten holzfaserverstärkten PP-Composite mit und ohne MAH-PP5%

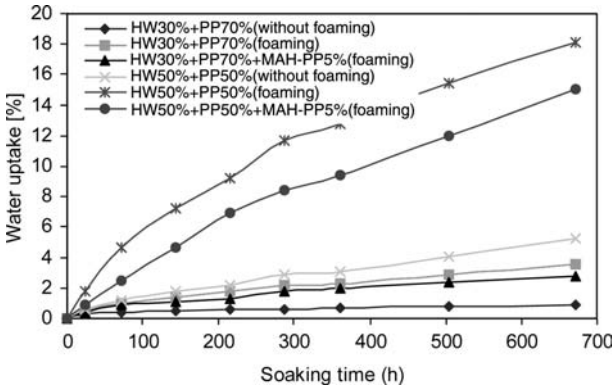

Fig. 13 Water absorption of microfoamed and non-foamed wood-PP composites with and without MAH-PP5%
Abb. 13 Wasseraufnahme der ungeschäumten und geschäumten holzfaserverstärkten PP-Composite mit und ohne MAH-PP5%

addition of coupling agent MAH-PP5% at wood fibre content 50% by weight.

3.4 Water absorption

Polypropylene is water-rejecting and shows no swelling in aqueous environment (Domininghaus 1988). The investigations from a flax- PP group (fibre content of 50 vol. %) showed that after 16 h a water absorption effected from 4,38% and a swelling takes place from around 4,28% (Mieck 2001). Also with the flax fibre reinforced PP microfoamed composites showed a water absorption effect which is observed in Fig. 12.

The water absorption takes place nearly proportionally in the examined range. With the foamed specimens, the water content and the thickness swelling were approximately two times higher than the non-foamed composites after the storage duration of 300 h.

For wood fibre-PP composites, water absorption results are plotted in Fig. 13 by water uptake versus soaking time. The microfoamed wood fibre-PP composites without coupling agent showed remarkably higher water absorption than did the non-foamed wood fibre-PP composites due to the presence of void content which is similar to flax fibre-PP foamed composites and other wood-PVC composites (Bledzki et al.1998). On addition of the coupling agent, the composites show lower water absorption when compared to non-addition of coupling agent wood fibre-PP composites which indicate that the coupling agent plays an important role in repelling the water molecules.

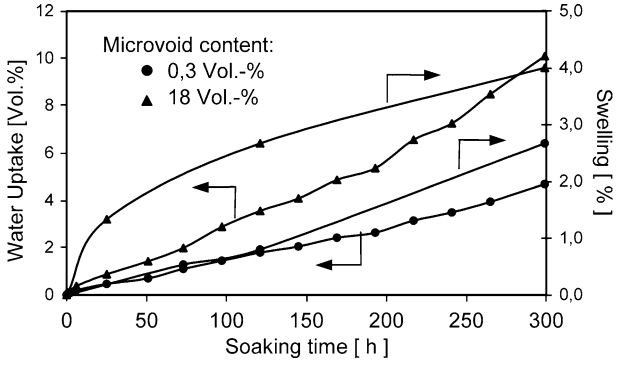

Fig. 12 Water absorption and swelling of flax-PP microfoamed and non-foamed composites [fibre content. 30% by weight.]
Abb. 12 Wasseraufnahme und Dickenquellung für flachsfaserverstärkte PP-Mikroschäume [Fasergehalt: 30 Gew.-%]

4 Conclusion

The flax and wood fibre reinforced polypropylene microfoams have hardly been described so far in the literature and a new trend with natural and wood fibre strengthened plastics racks, in the injection moulding process was prepared. The advantages of this processing technology lies in the fact that by changing of the processing parameters with a usual injection moulding machine, with a certain chemical foaming agent content, sandwich-structure shaped parts can be manufactured. From this study it can be concluded that.

- The microfoam structure depends on flow way.
- Front flow speed and mould temperature affect the density and microvoid content of flax fibre reinforced PP microfoamed composites.
- Density reduced around 20–25% for flax fibre-PP and wood fibre-PP foamed composites, respectively, and decreased up to 0.77 g/cm^3 for wood fibre and 0.81 g/cm^3 for flax fibre reinforced PP microfoamed composites.
- The increase of microvoid content within the measured ranges causes, with the firmness and the rigidity, only limited changes of the physicomechanical characteristic values for flax fibre-PP foamed composites.
- With the addition of the coupling agent, physicomechanical properties of wood fibre-PP microfoamed composites improved up to 50%.

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