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Investigation of the Applicability of a Hybrid Foaming System in a Mixed Plastic Waste Matrix

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Abstract
In our research, we investigated the chemical foaming of mixed plastic waste granulate (VMHD) using a combination of endothermic and exothermic foaming agents. We found that increasing the exothermic foaming agent improved cell distribution but reduced flexibility and impact resistance. The highest porosity (30.04%) was achieved with 0.5% endothermic and 3.5% exothermic foaming agents. Our results indicate that the proper ratio of foaming agents is crucial for optimizing the mechanical properties of the material.

Keywords
hybrid foaming, porosity optimization, recycled polymer composites

1 Introduction

Plastic pollution is one of the most serious environmental problems of our time, generating 300 million tons of waste annually worldwide, but only 9% of this is recycled [1, 2]. A significant portion of the remaining plastic ends up in landfills or accumulates in nature, seriously damaging ecosystems. Of particular concern is the growth of marine litter: for example, the Great Pacific Garbage Patch covers an area of 1.6 million square kilometers and contains at least 79000 tons of plastic [3]. The problem requires urgent intervention through improved waste management and stricter regulations.

The European Union has taken several steps to reduce plastic use and increase recycling. The Comprehensive Waste Directive aims for EU Member States to achieve a recycling rate of 55% by 2025, which should be further increased by 2035. The European Commission's Plastics Strategy, adopted in 2018, requires all plastic packaging to be recyclable by 2030 [3].

In our current research, we are attempting to recycle the increasingly large amount of mixed plastic waste shreds. The shredded plastic mixture consists of different polymers, which are prepared for recycling. Its composition is heterogeneous, which makes it difficult to produce a uniform quality raw material. The research is based on chemical foaming.

Polymer foams are two-phase systems in which gas bubbles of different sizes are present in the polymer matrix. Many thermoplastic polymers and some thermosetting polymers are suitable for foaming. Foaming technologies can be divided into three main categories: mechanical, physical and chemical methods. During chemical foaming, the decomposition of the added blowing agent produces a large amount of gas, which creates gas bubbles in the structure. Depending on the type of chemical blowing agent, different gases (e.g., CO, CO_2, N_2, NH_3) are released [4].

Chemical blowing agents are organic or inorganic solid compounds that decompose in the melt under the influence of high temperatures. As a result of the decomposition of the blowing agent, nucleation begins, and then gas bubbles form at these points. After the gas bubbles solidify, a cell structure is formed in the material. The formed cells are separate, and the cell density increases increasingly towards the core, thus forming the integral foam structure [5].

Among the foaming methods, the chemical blowing agent method is particularly effective for thermoplastic polymers, as the foaming is carried out using thermally reactive (endothermic or exothermic) blowing agents. This method can also be used in injection molding [6].

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To achieve adequate foaming, the volume of the injection-molded product can be increased by using the core-back technique [7]. In this process, the polymer containing the blowing agent is injected into the mold cavity, where a compact surface layer is first formed during cooling. The mold is then opened slightly, increasing the available space and reducing the pressure. Since the polymer is still molten inside the product, the internal gas bubbles can continue to expand.

In their research, Chung et al. [8] investigated the chemical foaming behavior of semi-crystalline polypropylene (PP) and amorphous polystyrene (PS), with a particular focus on the effect of processing parameters on the foaming efficiency. In addition to the azodicarbonamide used as an exothermic blowing agent, three key parameters – injection speed, melt temperature and mold temperature – were analyzed under different settings. The foaming efficiency was evaluated at the macroscopic level by measuring the mass, specific gravity and expansion ratio, while at the microscopic level by examining the cell density and cell size using a scanning electron microscope. According to their results, the crystalline structure of PP limited the foaming, while PS expanded to a greater extent. It was found that in the case of PP, low injection speed, high melt temperature and low mold temperature were more favorable to achieving the highest expansion ratio, while in the case of PS, high injection speed, medium melt temperature and low mold temperature proved to be optimal.

Akkoyun et al. [7] investigated the microstructure and mechanical properties of PP foams containing chemical blowing agents as a function of the parameters of the chemical foaming process. They specifically analyzed the effect of injection speed on the cell diameter, outer layer thickness, cell density and elastic modulus. During the tests, the injection speed was set at three different values (110 mm/s, 125 mm/s and 140 mm/s). According to their results, increasing the injection speed also increased the cell diameter, outer layer thickness and elastic modulus, which resulted in an improvement in mechanical strength.

The combined use of endothermic and exothermic blowing agents is an innovative approach [9, 10]. This method improves the uniformity of the cell distribution and increases the number of cells due to the specific interaction of the two components. During hybrid foaming, the heat generation of the exothermic blowing agent and the heat absorption of the endothermic component create a balance in the polymer matrix, thereby optimizing the formation of the foam structure. Furthermore, the endothermic blowing agent can also have a nucleating effect, which allows for more efficient and economical foam production [11].

In Szabó et al. [12], the effect of the endothermic and exothermic hybrid foaming was investigated on the mechanical and morphological properties of recycled polyethylene terephthalate (rPET) foams. Our results showed that hybrid foaming significantly improved the homogeneity of the cell structure and increased the cell number, which contributed to the reduction of the density of the foams, while maintaining mechanical stability. In contrast, samples containing only endothermic blowing agents showed a less homogeneous cell structure. The addition of exothermic blowing agents at a concentration of 0.5% resulted in a significant improvement in the uniform distribution of cells, while at a concentration of 1% exothermic blowing agent, porosity increased without deteriorating mechanical properties. In contrast, higher exothermic blowing agent concentrations already negatively affected both the cell structure and porosity. Our research also demonstrated that higher porosity per se does not lead to mechanical weakening if the cell distribution remains homogeneous. The highest porosity level (16.6%) was achieved by using 3% endothermic and 1% exothermic blowing agent, while the mechanical properties remained favorable (Young's modulus: 1.49 ± 0.1 GPa; tensile strength: 28.94 ± 1.33 MPa; Charpy impact strength: 3.51 ± 0.46 kJ/m^2).

In this article, we aim to test the applicability of hybrid foaming on mixed plastic waste shreds.

2 Methods

The mixed plastic waste shreds used in the research were provided by Jász-Plasztik Kft. (Jászberény, Hungary). Its approximate composition is 75–85% HDPE and 15–25% PP according to the manufacturer's tests.

The foaming was done with products from Tramaco (Tornesch, Germany). Our first blowing agent was the endothermic compound Tracel IM 7200, which had a gas emission of 120 mL/g, a blowing agent content of 70%, and a decomposition temperature of 220 °C. Our second blowing agent, the exothermic compound Tracel IM 3170 MS, showed a gas emission of 50 mL/g and a decomposition temperature of 170 °C. The purpose of the combination of the two blowing agents was to increase the homogeneity of the cell structure, with particular attention to the balance of foam density and cell size. The material composition of the different samples is given in Table 1. The standard specimen production was done *via* Arburg Allrounder 420C Golden Edition Injection molding machine (Arburg,

Table 1 Composition of tested samples

	Mixed plastic waste shreds (wt.%)	Tracel injection molding (IM) 7200 (wt.%)	Tracel IM 3170 masterbatch solid (MS) (wt.%)
Ref.	100	0	0
EN40EX0	96	4	0
EN35EX5	96	3.5	0.5
EN30EX10	96	3	1
EN25EX15	96	2.5	1.5
EN20EX20	96	2	2
EN15EX25	96	1.5	2.5
EN10EX30	96	1	3
EN5EX35	96	0.5	3.5
EN0EX40	96	0	4

Loßburg, Germany). The main parameters are as follows: Screw diameter: 35 mm; Nozzle temperature: 220 °C; Mold temperature: 35 °C; Switch-over volume: 35 cm^3; holding pressure (3 stages, pressure and [time]): 650 bar [1 sec]–550 bar [8 sec]–250 bar [0.9 sec]; cooling time: 20 sec. To achieve suitable level of foaming, breathing mold technique was used, which defined as after the holding pressure, when the outer layer of the specimen cooled down, the mold is slightly opened (in this case it was opened to 0.5 mm), which allows the inner, the melted polymer to expand from the foaming agent.

The internal structure of the samples was examined using an industrial computed tomography (CT) machine. A Nikon XT H 225 ST CT scanner (Nikon, Tokyo, Japan) with a resolution of 6 μm was used to analyze the foamed mixed plastic waste shreds. The average scan time was 24 min as 1500 projections images were captured in two frames per projection mode. The scanning voltage was 165 kV and the current was 65 μA. The VG Studio Max (2023.1) software package [13] was used to analyze and visualize the data. Cell distribution was analyzed by converting voids into equivalent perfect spheres and counting the amounts between certain diameters in mm.

CT scanning generated a detailed 3D image of the samples, which allowed the investigation of cell structures. The volume of the cells was subtracted from the total sample volume to determine the volume of the remaining solids. The density of the sample was calculated by dividing the total mass of the material by this corrected volume, thus obtaining an accurate value taking into account the internal porosity. Density was also determined by measuring the volume of specimens used for Charpy impact strength measurement test and dividing it by its weight.

During the mechanical tests, following the guidelines of ISO 527-1:2019 standard [14], five specimens of each sample type were tested and the results were averaged. Tensile strength tests were performed using an Instron 5582 universal testing machine (Norwood, MA, USA) with a 100 mm clamp length. The test speed was set to 1 mm/min for the determination of the elastic modulus and 5 mm/min for the tensile strength measurement.

The ISO 178:2019 [15] bending tests were performed using the same universal testing machine used for the tensile tests. The bending strength and flexural modulus of the samples were measured, the crosshead speed was set to 5 mm/min, and the support was set to 64 mm.

Charpy impact strength tests were performed in accordance to the EN ISO 179-1:2023 standard [16], using a Ceast 6545 pendulum impact testing machine (Ceast, Torino, Italy), with a 15 J hammer, 62 mm support length on unnotched 80 mm long rectangle-based specimens machined from the tensile test specimens.

For analyzing the data to find significant difference among the different samples, One-way Analysis of Variance (ANOVA) with post-hoc Tukey Honestly Significant Difference (HSD) was utilized [17].

3 Results and discussion

3.1 CT results

Fig. 1 shows CT scans of the different material compositions, while Table 2 shows the number of cells and the number of cells within certain diameters when the cell is considered a perfect sphere. The cross-sectional image shows very mixed results. The endothermic and exothermic foaming agents individually achieved much smaller foaming and more collapsed cells than most mixed types. This supports our hypothesis that hybrid exothermic-endothermic foaming agents can achieve better foaming. The best foamed cross-sections were achieved by the EN35EX5, EN30EX10, EN20EX20 and EN5EX35 samples. In the

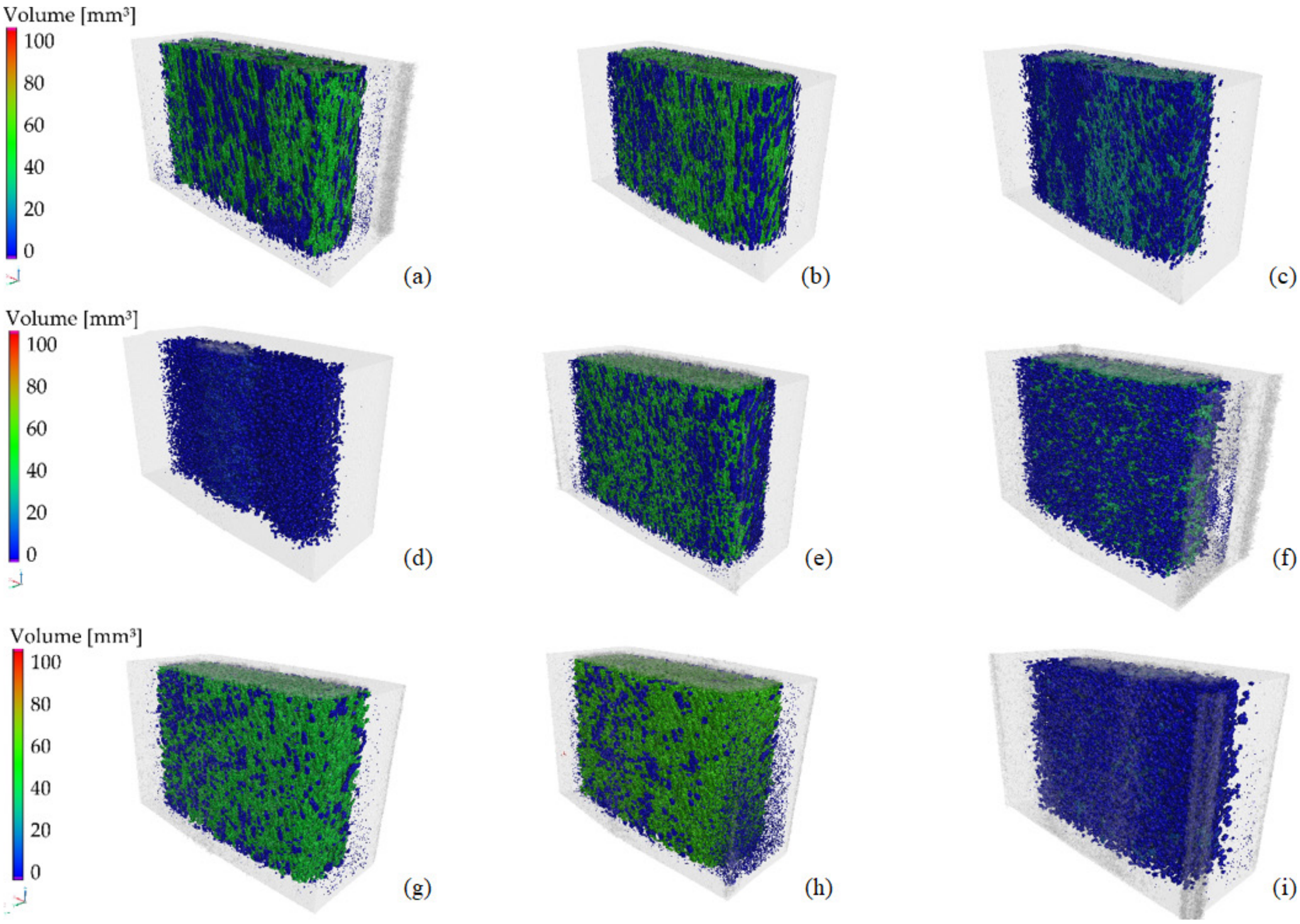


Fig. 1 Cross-section CT scans of foamed samples: (a) EN40EX0; (b) EN35EX5; (c) EN30EX10; (d) EN25EX15; (e) EN20EX20; (f) EN15EX25; (g) EN10EX30; (h) EN5EX35; (i) EN0EX40

Table 2 Number of cells and number of cells within certain diameters in mm if the cells are considered perfect spheres

	Number of cells	<0.03	[0.03, 0.06]	[0.06, 0.09]	[0.09, 0.12]	[0.15, 0.18]	[0.18, 0.21]	[0.21, 0.24]	[0.24, 0.27]	[0.27, 0.3]	>0.3
EN40EX0	4704	1502	1689	481	334	246	158	118	55	34	24
EN35EX5	4441	532	1581	981	654	342	165	92	42	23	15
EN30EX10	5301	232	768	1389	1165	776	492	220	124	61	38
EN25EX15	4779	40	226	634	890	768	771	589	323	222	121
EN20EX20	7083	934	2903	1559	918	423	175	90	45	14	13
EN15EX25	8543	1233	2500	1160	1581	1164	512	221	94	41	21
EN10EX30	3976	1335	1302	341	388	333	157	61	35	15	2
EN5EX35	9797	3603	5023	311	321	273	155	64	32	10	3
EN0EX40	5264	406	439	311	784	1126	929	561	316	146	87

case of the other samples, a smaller foamed core with much larger cells was formed. Based on the images, it can be seen that the ratio of the foaming agents greatly influences the size and density of the formed cells. Kharbas et al. studied thermoplastic polyurethane foaming, for which they used chemical and physical foaming agents. According to their results, the combination of 3% microcellular and supercritical fluid foaming gave the densest foamed cell structure [18]. When analyzing the cell size distribution, it is clear, that the proper ratio of endothermic-exothermic foaming agent can significantly increase the amount of cells forming in the foamed samples. The highest amount of cells were achieved in the EN5EX35 samples, however, it was paired with large amount of cells equivalent of sphere diameter between 0.03 and 0.06 mm. A large shift in cell size distribution is also observable in the case of EN25EX15 sample, where the cells sizes are distributed in an almost Gaussian distribution between 0.03 and 0.3 mm.

Fig. 2 shows the porosity based on the CT test results in contrast to their density. The reference material had a density of 0.89 g/cm^3. Based on the results, the porosity of the specimen made with the pure endothermic chemical foaming agent was 26%, which seems better compared to the pure exothermic, which showed a porosity of 15.5%. The density of the two is 0.71 g/cm^3 and 0.73 g/cm^3 respectively, which does not appear to be a huge difference. With minimal addition of the endothermic foaming agent, the EN5EX35 sample reached 30% porosity, the highest value among the other samples, which is also visible on the CT scan itself. In the opposite case, in the case of the EN35EX5, the porosity did not change compared to the pure endothermic foaming agent, but the density jumped to 0.79 g/cm^3, the highest point amongst all. When the exothermic ratio further increased, the porosity decreased to 18 and then 13%, and the density remained high for the EN30EX10 (0.78 g/cm^3).

In the case of a half-and-half endothermic-exothermic ratio, the porosity jumped back to 24.5%, but still did not reach the level of the pure endothermic chemical foaming agent, and by further increasing the exothermic ratio, we get 21 and then 24% porosity. Regarding density, it seems with mixture of endothermic and exothermic foaming agent, while the endothermic is in dominance, the density is much higher, suggesting less than desired weight decrease. The results support the importance of selecting the appropriate blowing agent ratio and highlight the significant difference in the effect of the two blowing agents.

Kmetty et al. [19] compared the effect of endothermic and exothermic blowing agents for the foaming of Natureworks Ingeo 2003D polylactic acid and concluded that the exothermic blowing agent achieved a higher porosity of 55%, while the endothermic blowing agent achieved only 34%. The large difference was attributed to the processing temperature, which is consistent with our results, which indicate that the selection of the appropriate processing temperature is essential for the endotherm.

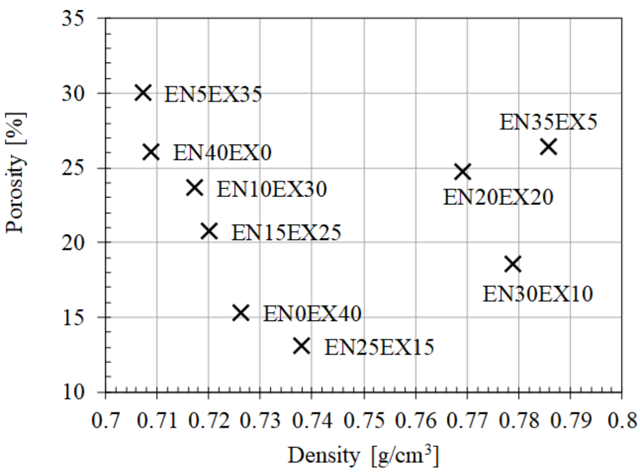


Fig. 2 The porosity of the different foamed materials as a function of density

3.2 Tensile test

The results of the tensile test are shown in Fig. 3. The reference, non-foamed samples had a tensile strength of 21.22±1.36 MPa and a Young's modulus of 0.84±0.04 GPa. The Tukey HSD test failed to find significant difference among the tensile strength of the samples. When the reference is removed, significant difference was found between the EN0EX40 paired with the reference and EN5EX35. In case of Young's modulus, similar result was found. Compared to the reference and the EN5EX35, the EN0EX40 samples are significantly different. The highest tensile strength value was achieved by the sample prepared with only exothermic foaming agent, with a value of 16.73 MPa, while the lowest was achieved by the EN5EX35 sample. The tensile strength of the sample prepared with a purely endothermic chemical foaming agent was determined to be 15.05 MPa, which shows a low deviation with increasing exothermic foaming agent ratio. In the case of the EN20EX20 sample, we can see a more serious spike, in the form of a tensile strength of 15.68 MPa, which remains stable with a decrease in the endothermic foaming agent ratio up to the EN10EX30 sample. It is difficult to write a trend for the tensile strength values; in both cases the individual foaming agents performed

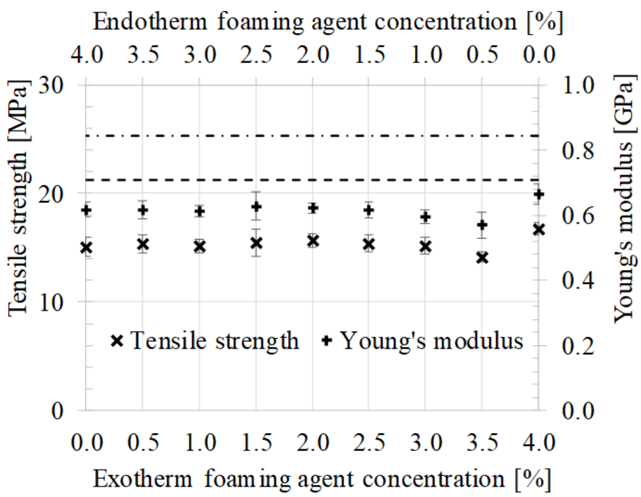
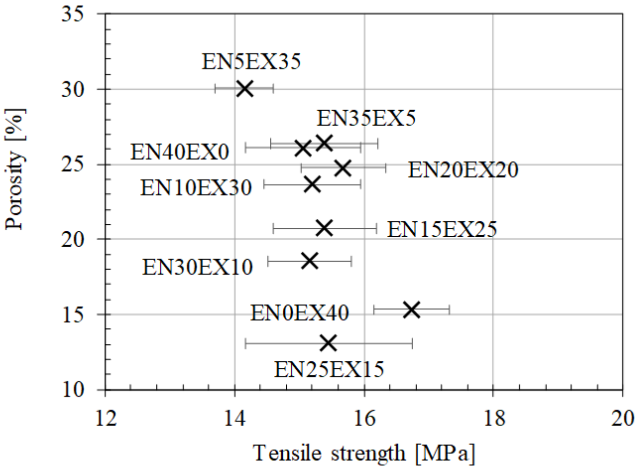


Fig. 3 Tensile strength and Young's modulus of the different foamed samples

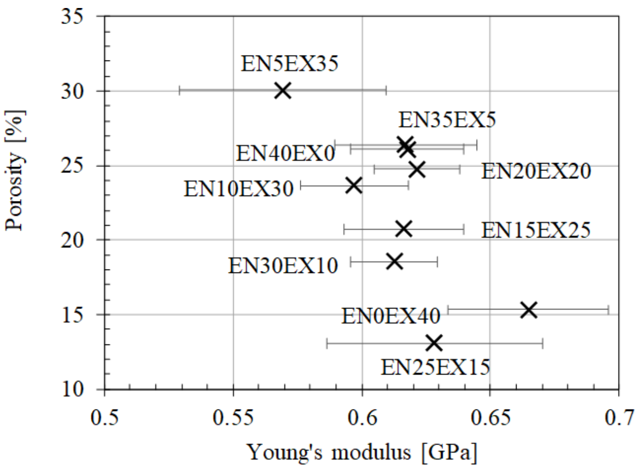
better compared to the mixed types, but the deterioration is lower in the case of endothermic mixtures than in the case of exothermic ones. Since EN35EX5 has one of the highest porosity, it can be said that the higher cell density leads to better resistance to uniaxial tension, which is also supported by Heim and Güzel [20].

If we observe the Young's modulus values, their change with the change in the ratio of different blowing agent materials changes in the same way as the tensile strength. The highest modulus value was the purely exothermic foamed sample (0.66 GPa), while the lowest was the EN5EX35 sample (0.57 GPa). This is explained by the degree of foaming between the different samples, just like for tensile strength.

Fig. 4 showcases the correlation between porosity and the tensile test results, with Fig. 4 (a) the tensile strength, Fig. 4 (b) the Young's modulus. It can be observed, that the EN0EX40 sample had the highest tensile strength and modulus with the second lowest porosity, While the EN5EX35 with the highest porosity had the lowest resistance to one-axis tension.



(a)



(b)

Fig. 4 Correlation of tensile properties to porosity of the foamed samples: (a) Tensile strength; (b) Young's modulus

3.3 Flexural test

The results of the flexural test are summarized in Fig. 5. The reference was measured to be 25.89±0.63 MPa in terms of maximum flexural stress and the flexural modulus was determined to be 0.84±0.03 GPa. The Tukey HSD found that even with the reference in the data, the flexural strength of the EN10EX30 and EN5EX35 samples are significantly different than EN35EX5 and EN30EX10, meaning that the ratio of the foaming agent has an effect on the flexural strength. In case of flexural modulus, the EN10EX30 showed difference compared to EN35EX5 and EN30EX10. The trend in both strength and modulus values is almost identical to the tensile test, but it highlights the differences between the individual samples much more. The samples produced with purely endothermic and exothermic blowing agents showed higher modulus and strength values during flexure than their mixed counterparts, with the exception of the EN35EX5 sample, which has the highest flexural strength of 24.59 MPa and a flexural modulus of elasticity of 0.87 GPa. It can be seen that the higher proportion of endothermic chemical blowing agent favored the flexural properties much more, while the exothermic one less so. The negative effect of exothermic foaming agents on flexural properties was also observed by Dou and Rodrigue [21], when the flexural strength of foamed PP decreased from ~1 GPa to ~0.37 GPa when using 0.5% rotational plastic molding technology.

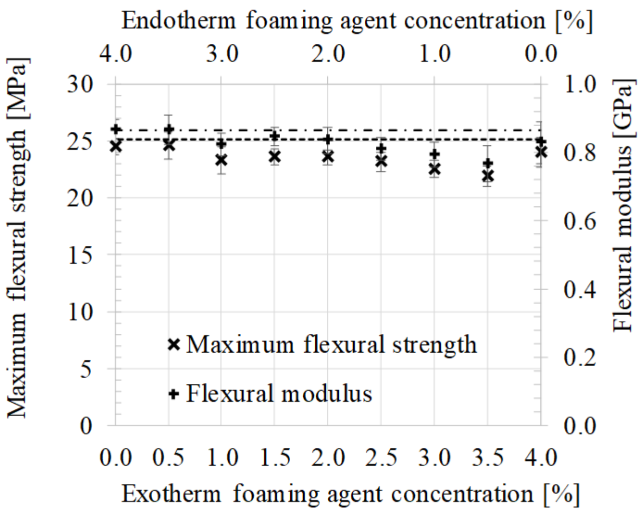


Fig. 5 Flexural strength and flexural modulus of the different foamed samples

Fig. 6 displays these results with porosity. Fig. 6 (a) for maximum flexural strength and Fig. 6 (b). Among the samples, EN35EX5 had the flexural properties with 26.4% porosity. It has hard to draw a trend to the dataset, but based on the porosity results, with dominant endothermic foaming agent better porosity and flexural strength can be achieved.

3.4 Impact strength

The results of the impact strength tests are summarized in Fig. 7. The samples without foaming agent was measured to be 97,57 kJ/m^2. The foamed samples impact strength was measured to be between 25–45 kJ/m^2. Tukey HSD test confirmed that there is no significant difference between any samples, with the exception of the reference. Even excluding it the test was unable to find significant difference. The highest impact strengths were shown by samples dominated by exothermic foaming materials, with specific impact works of 38.24, 42.16 and 35.94 kJ/m^2 for the EN15EX25, EN10EX30 and EN5EX35 samples. The impact strength of the samples containing pure endothermic blowing agent was 34.13 kJ/m^2, which gradually decreased to 31.07 and then 27.99 kJ/m^2 with increasing proportion of exothermic blowing agent. In the case of samples dominated by exothermic blowing agent, the opposite was true, with mixtures yielding higher impact strength, while the EN0EX40 sample showed one of the lowest results, 31.87 kJ/m^2. Zhou and colleagues conducted experiments with PP-bamboo blends with different exothermic-endothermic blowing agents and measured similar increasing and then decreasing impact energy. In the case of 0% blowing agent, a specific impact energy of ~5 kJ/m^2 was measured for notched specimens, at 1% it increased to over 7 kJ/m^2, and then decreased again with the use of 2% exothermic blowing agent. This supports the importance of choosing the appropriate exothermic foaming ratio [22].

Fig. 8 showcases the connection between porosity and impact strength. According to this, the endothermic foaming agent dominant samples can achieve high porosity and impact strength, however, the porper ratio of exothermic

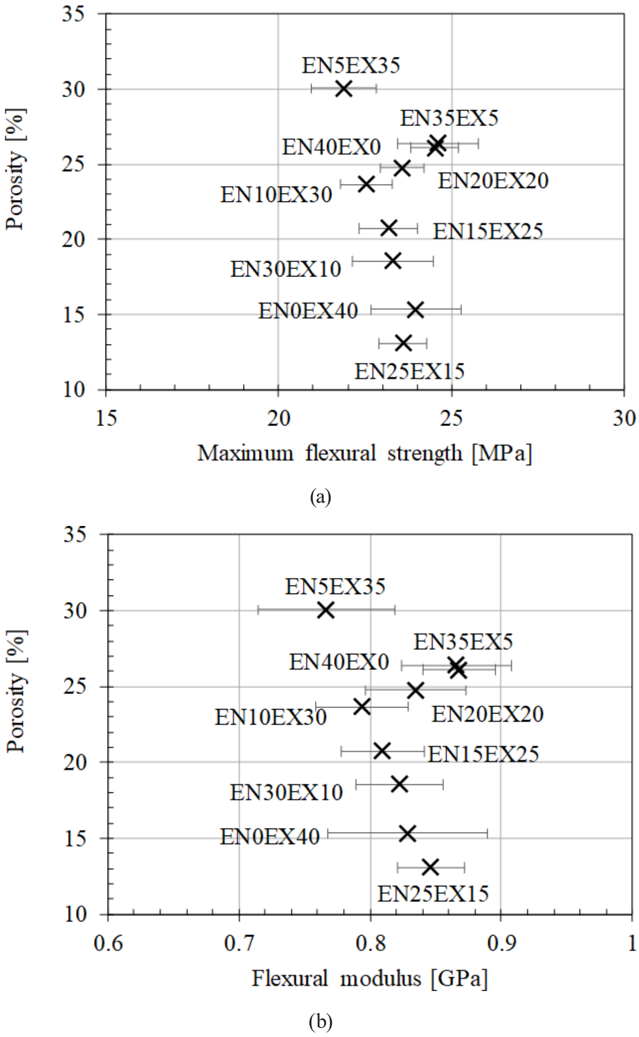


Fig. 6 Correlation of flexural properties to porosity of the foamed samples: (a) maximum flexural strength; (b) flexural modulus

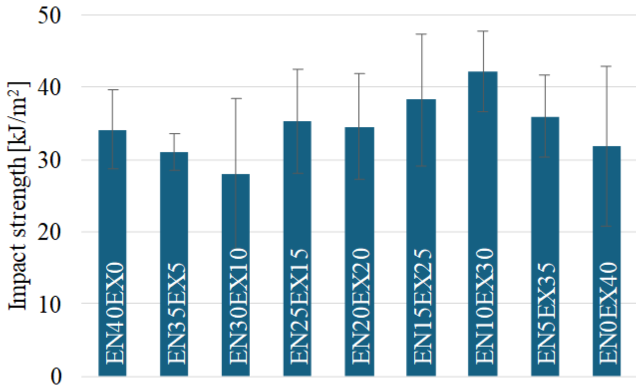


Fig. 7 Impact strength of the different foamed samples

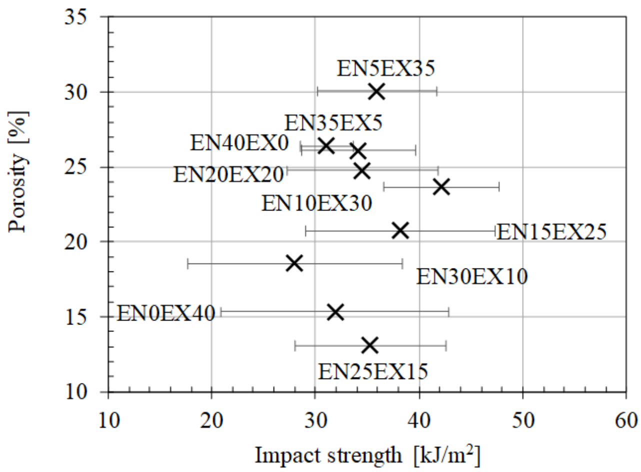


Fig. 8 Impact strength of the different foamed samples with the correlation to porosity

foaming agent is still impactful to the dynamic mechanical resistance of the foamed material.

4 Conclusion

The aim of this research was to investigate the applicability of hybrid endothermic–exothermic foaming in the recycling of mixed plastic waste (VMHD). The results showed that the appropriate blowing agent ratio plays a key role in the formation of the homogeneity of the foam structure and the optimization of the mechanical properties. Based on the CT tests, the highest porosity value was achieved by the EN5EX35 sample, which, taking advantage of the combination of endothermic and exothermic foaming, resulted in a more uniform cell structure. Mechanical tests confirmed that a higher ratio of the endothermic blowing agent is more advantageous in terms of flexural strength and elastic modulus, while the dominance of the exothermic blowing agent had a lesser weakening effect on the tensile strength and impact strength. Overall, the results support that the optimally chosen endothermic–exothermic ratio allows maintaining the balance of mechanical strength and porosity, thus enabling the efficient recycling of mixed plastic waste. The application of hybrid foaming may be a promising solution for the production of lightweight yet mechanically stable foamed materials that can potentially be used in the automotive industry and other industrial fields.

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