

POLIMERY

Structure and properties of self-extinguishing rigid polyurethane foam with inorganic filler

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Abstract: The study examined the influence of selected inorganic fillers (5 and 10% by mass) on the foaming process, physicochemical properties, and structure of self-extinguishing polyurethane foam (density of approx. 35 kg/m³) obtained by reacting polyol with isocyanate (100:110). The filler used was bentonite (B), lamellar graphite (G), dendritic copper (Cu), aluminum oxide (Al₂O₃) and silicon carbide (SiC) and Al₂O₃/SiC mixture (1:1). Stereoscopic microscopy was used to determine the structure. The functional properties were examined based on density, water absorption and fire resistance. The course of the foaming reaction depends on the content, type and physicochemical properties of the filler. The addition of bentonite increases water absorption and Al₂O₃ reduces flammability of the foam.

Keywords: polyurethane foams, inorganic fillers, dispersion, water absorption, flammability.

Struktura i właściwości samogasnącej sztywnej pianki poliuretanowej z napełniaczem nieorganicznym

Streszczenie: W pracy zbadano wpływ wybranych napełniaczy nieorganicznych (5 i 10 % mas.) na proces spieniania, właściwości fizykochemiczne i strukturę samogasnącej pianki poliuretanowej (gęstość ok. 35 kg/m³), którą otrzymano w wyniku reakcji polioliu z izocyjanianem (100:110). Jako napełniacza użyto bentonitu (B), grafitu o budowie lamelarniej (G), miedzi o strukturze dendrytycznej (Cu), tlenku glinu (Al₂O₃) i węgla krzemu (SiC) oraz mieszaninę Al₂O₃/SiC (1:1). Do oceny struktury stosowano mikroskopię stereoskopową. Właściwości użytkowe oceniano na podstawie gęstości, absorpcji wody i odporności na ogień. Przebieg reakcji spieniania zależy od zawartości, rodzaju i właściwości fizykochemicznych napełniacza. Dodatek bentonitu zwiększa absorpcję wody, a Al₂O₃ zmniejsza palność pianki.

Słowa kluczowe: pianki poliuretanowe, napełniacze nieorganiczne, dyspersja, absorpcja wody, palność.

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Polyurethane foams are often used in various industries (construction, furniture, automotive). According to data [1], it is estimated that the size of the global polyurethane foam market is 50 billion USD, and in 2032 it is expected to exceed over USD 95 billion. The wide use of foams is due to their properties – low density and high insulating properties, but they are limited by low strength and flammability [2, 3]. Solutions that combine

individual insulating characteristics with flame resistance are often sought. The properties depend on the substrates used, mixing ratios, limitations during production [2, 4–7], cell size [2, 7, 8], cell content, anisotropy of the cell structure [6, 9] and cell wall thickness [10] as well as by applied modification, e.g. chemical [5, 11, 13], organic and inorganic fillers [10, 11, 14], by surface functionalization [15]. Phosphorus-containing flame-retar-

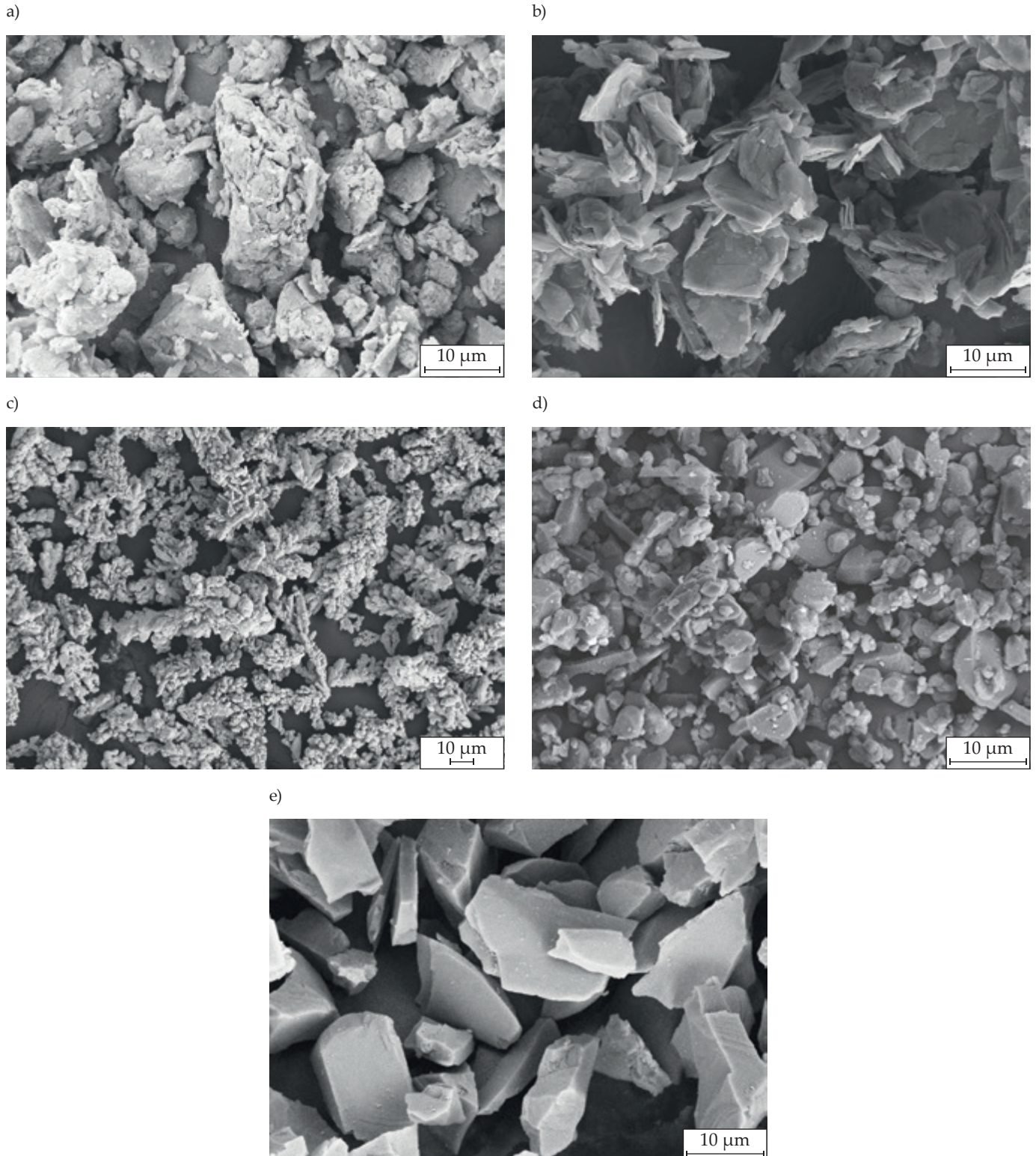


Fig. 1. SEM images: a) bentonite, b) graphite, c) copper, d) Al₂O₃, e) SiC

dant polyols are a type of reactive flame retardant that can be used as a substitute for conventional polyols in preparing rigid polyurethane foam (RPUF) to enhance its flame-retardant properties [16, 17]. In addition to phosphorus, sulfur and nitrogen compounds can be used. Using a polyol containing boron and an increased share of nitrogen in [18] resulted in flame retardancy. Zhang described the foaming reaction stages [19], who used a Bragg grating to monitor the process: cell nucleation, cell growth, microphase separation, and polymerization. Temperature changes made it possible to distinguish subsequent stages of the foaming reaction, i.e., start time, rise time, and tack-free time [20].

The main limitations of the application are that it is low in fire resistance and is environmentally nuisance. Reducing flammability is possible using fillers and appropriate chemical modifications, which also affect other properties. In work [21], nano-SiO₂, peanut shells, and pine bark were introduced, resulting in the structure's fragmentation. The flammability of polyurethane foams can be reduced by adding expanded graphite [22], carbon nanotubes [7], and aluminosilicates [23]. Flame retardancy mechanisms depend on the filler used [2, 7, 23, 24]. Silicon carbide is used as a filler in both flexible and rigid polyurethane matrices to increase the TC value in electronic applications [25]. Inorganic and organic fillers do not affect the state of hydrogen bonds in composites [26].

Therefore, the objective of the current research is to study the impact of selected inorganic fillers on the foaming process, physicochemical properties, and structure of self-extinguishing polyurethane foam. A two-component polyurethane system consisting of polyol and isocyanate mixed in a ratio of 100: 110 (by weight) was selected. Bentonite (B), lamellar graphite (G), copper with a dendritic structure (Cu), aluminum oxide (Al₂O₃) and silicon carbide (SiC), as well as the mixture of Al₂O₃/SiC (1 : 1) were used as fillers. The structure of the obtained foams was examined using a stereoscopic microscope. Moreover density, water absorption and flammability were evaluated.

EXPERIMENTAL PART

Materials

The polyurethane system consisting of polyol (component A) and isocyanate (component B) mixed in a ratio of 100:110 (by weight) was purchased from Polycore (Swidnik, Poland). The foam used for modification was self-extinguishing and had an apparent density of approximately 35 kg/m³. Special bentonite (B) with 75% montmorillonite content, bulk weight 0.73 g/cm³ (Zakłady Gorniczo-Metalowe "ZEBIEC" JSC, Starachowice, Poland), lamellar graphite (G) MG 394 with grain size < 45 μm (Sinograf, Torun, Poland), specific fraction 90% (carbon content 94%), bulk density 0.4 g/cm³, dendritic copper Lt16 (Cu) with grain sizes from 32 to 150 μm (Stanchem, Niemice, Poland), bulk density 1.5–1.6 g/cm³, aluminum oxide (Al₂O₃) > 98% purity and bulk density 1.51–2.07 g/cm³ (Warchem, Warsaw, Poland) and silicon carbide (SiC) > 97% purity and bulk density 1.3–1.5 g/cm³ (PPH REWA, Nowy Redzien, Koluszki, Poland) were used as inorganic fillers. The fillers were pre-dried at temperature of 80±2°C for 4 hours in a Pol-Eko SLW 53 STD forced air dryer (Wodzislaw Slaski, Poland). The morphology of the fillers was examined using a Zeiss Supra 35 scanning electron microscope (Carl Zeiss AG, Oberkochen, Baden-Württemberg, Germany) and is shown in Fig. 1.

Composites preparation

The composites were obtained using a high-speed dissolver Dispermat LC30 mixer (VMA-Getzmann GmbH, Reichshof, Germany). The fillers were incorporated into the matrix at concentrations of 5 and 10 wt%. A mixture of aluminum oxide and silicon carbide (1:1) was also used as a filler. The filler was added to polyol while mixing at 900 rpm for 30 s, then isocyanate was added while continuing mixing for 10 s at 150 rpm. The foams were cast into polyethylene molds.

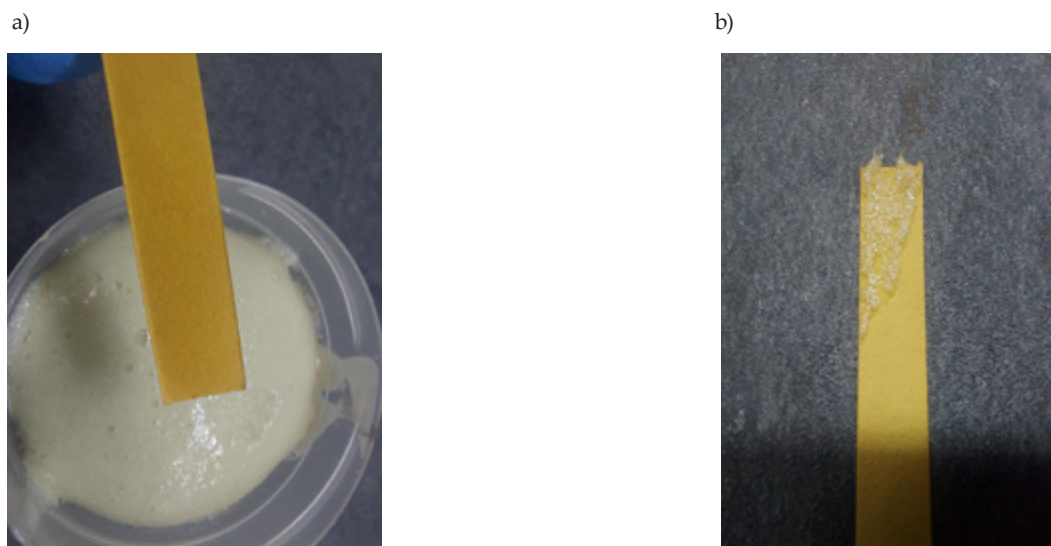


Fig. 2. Verification of the foam surface using tissue paper: a) dry foam, b) wet foam (unfinished reaction)

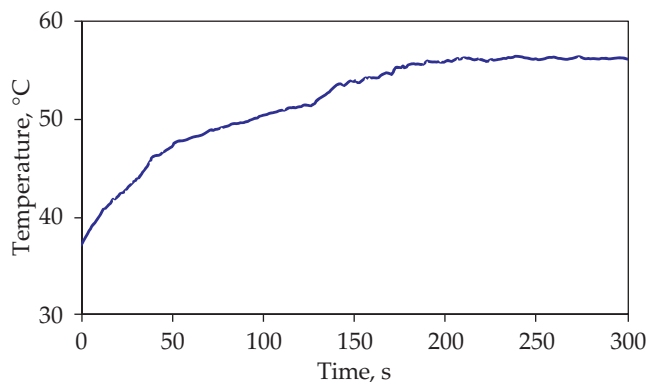


Fig. 3. Temperature as a function of time during the foaming process

Methods

The impact of fillers on the foaming process was determined by measuring the start time of reaction, understood as the moment when the mixture temperature begins to increase, the rise time (temperature stabilization) and the dry time (time to reach a dry foam surface, checked with tissue paper) (Fig. 2).

The temperature change during growth was also monitored using the thermal imaging method (Flir A615 camera working with IrControl software, Teledyne Technologies, Thousand Oaks, CA, USA). The temperature change as a function of time is shown in Fig. 3. The adopted thermal

imaging research methodology assumed measurement of the foam surface temperature (Fig. 4). Changes in foam core temperature were not evaluated.

All tests were performed on samples of the same volume. The shape factor (S_f) is calculated as the quotient of the foam height after tack-free time (H) to the diameter of the foam (D):

$$S_f = \frac{H}{D} \quad (1)$$

The tests were conducted at $20 \pm 2^\circ\text{C}$ and 35% humidity.

Stereomicroscopy

Leica stereomicroscope DVM6 (PIK Instruments Ltd, Piaseczno, Poland), equipped with a camera with a resolution of 10 Mpx and a magnification of 16:1 was used to examine the structure of the foams cross-section.

Fourier-transform infrared spectroscopy

Fourier infrared spectroscopy (FT-IR) (Shimadzu Corporation, model IRSpirit FTIR, Kyoto, Japan) was used to analyze the chemical structure. The spectra were recorded using at least 20 scans with 4 cm^{-1} resolution, in the spectral range of $3600\text{--}400\text{ cm}^{-1}$. The ATR diamond crystal was cleaned with ethanol between the tests to avoid obtaining false spectra.

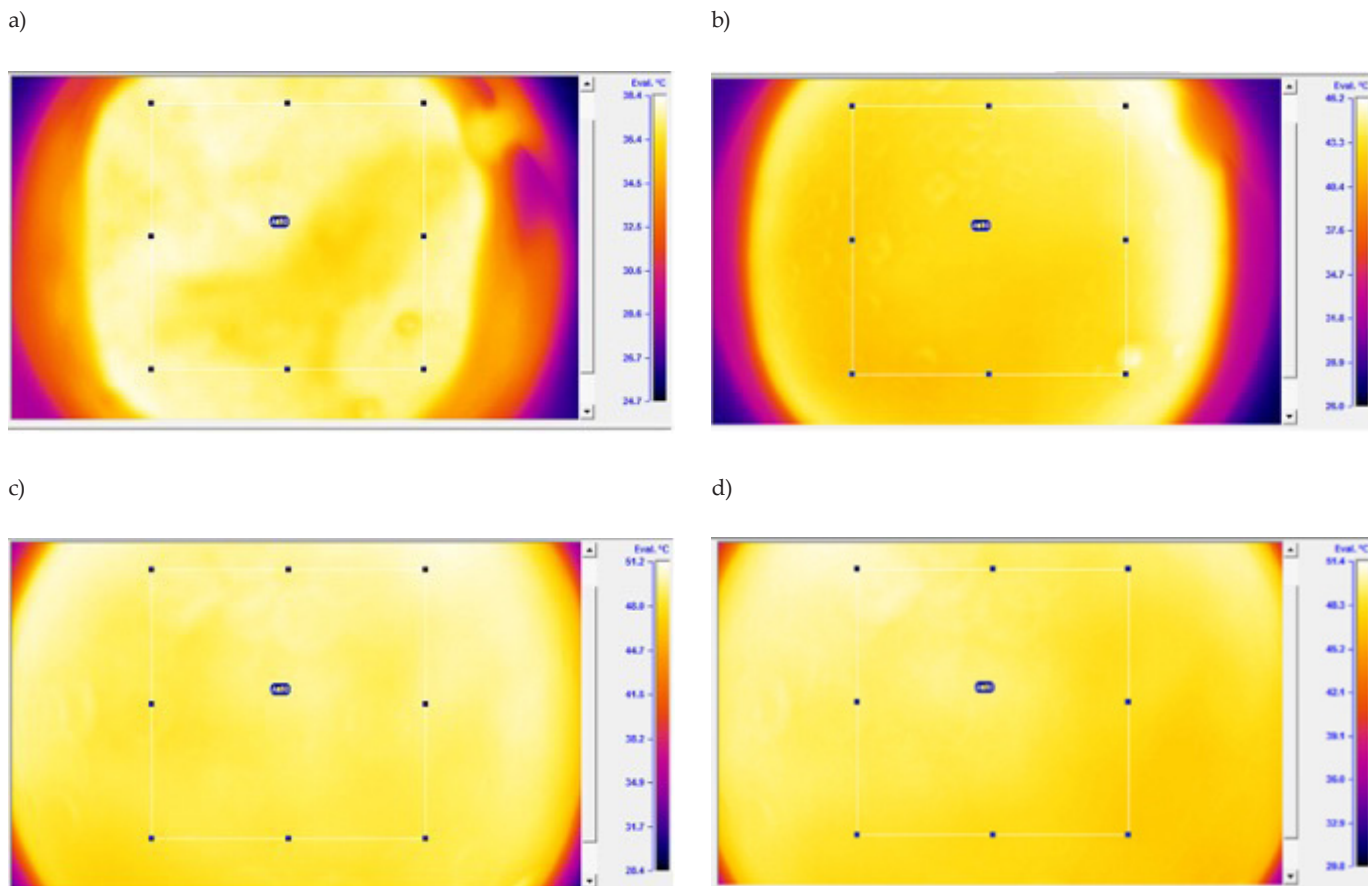


Fig. 4. Thermograms of the foam surface: a) beginning of registration, b) after 40 s c) after 100 s d) after 120 s

Density

The density was determined for 5 samples cut from the obtained foams with a diameter of 20 mm and a height of 20 mm. Measurements were conducted in accordance with the ISO 845 standard.

Water absorption

Absorption tests were conducted in accordance with the ISO 62 standard. Samples with a diameter of 20 mm and a height of 20 mm were dried at $40 \pm 20^\circ\text{C}$ to constant weight, placed for 24 hours in a water bath at a temperature of $20 \pm 2^\circ\text{C}$, and then weighed. The water absorbency was determined according to the following formula:

$$W = \frac{m_w}{m_s} \quad (2)$$

where: W – water absorption (%), m_w – mass of the sample after removing from the water bath (kg), m_s – sample mass after removal from the dryer (kg).

Flammability

Flammability was conducted using a cone calorimeter according to ISO 5660-1. The tests were conducted on samples cut from polyurethane foams in the form of cuboids with dimensions of 100×100 mm and a thickness of 20 mm to 30 mm. The temperature during the test was 21°C , humidity was approximately 35%, and atmospheric pressure was approx. 97.1 kPa. The tests were conducted using an automatic dual cone calorimeter from Fire Testing Technology Ltd (East Grinstead, Great Britain). The tests involved placing the samples horizontally concerning the conical radiator and initiating the combustion reaction with a spark igniter. External thermal radiation of 50 kW/m^2 was used, affecting the sample with thermal energy with a surface density of 50 kW/m^2 . The heat release rate (HRR), total heat release (THR), time to ignition (TTI), smoke production (TSP), carbon monoxide and carbon dioxide concentration, MARHE (Maximum Average Rate of Heat Emission), and ARHE (Average Rate of Heat Emission). The cone calorimeter allows for gas analysis, i.e., a detailed examination of combustion products, including CO and CO_2 emissions.

RESULTS AND DISCUSSION

The foaming process analysis

Figure 5 shows the results of reaction start times, measured from the time of isocyanate addition to the temperature increase while always maintaining the same volume.

The results presented in Fig. 5 clearly indicate that fillers accelerate the reaction start time. In most cases, the higher the filler content, the shorter the reaction start

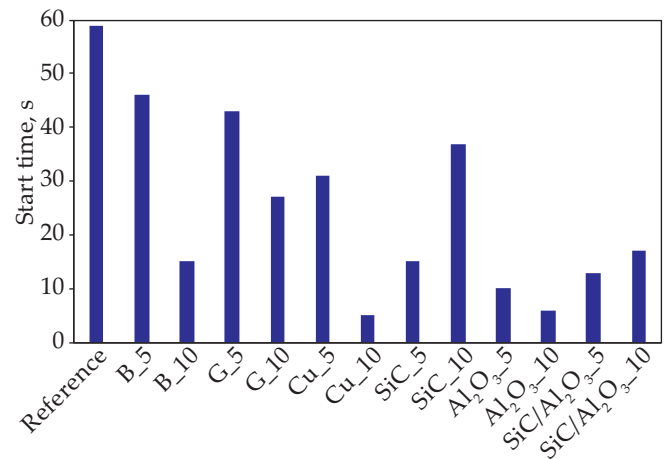


Fig. 5. Effect of the type and fillers content on start time

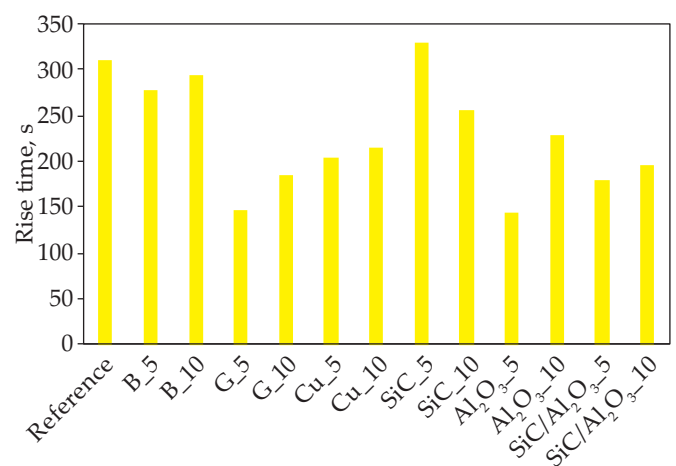


Fig. 6. Effect of the type and fillers content on rise time

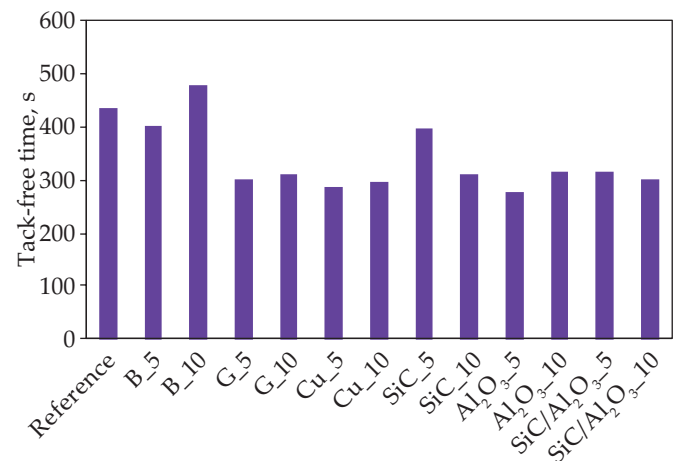


Fig. 7. Effect of the type and fillers content on tack-free time

time. The opposite behavior is valid only for SiC and SiC/Al₂O₃; for a content of 10 wt%, the reaction start time is longer than for 5 wt%. This is due to the influence of silicon carbide on the reaction; the higher the SiC content, the faster the reaction starts.

The results of reaction completion times, presented in Fig. 6, clearly indicate that fillers shorten the time, except for composites with 5 wt% SiC content. This confirms that SiC influences the course of the reaction in obtaining

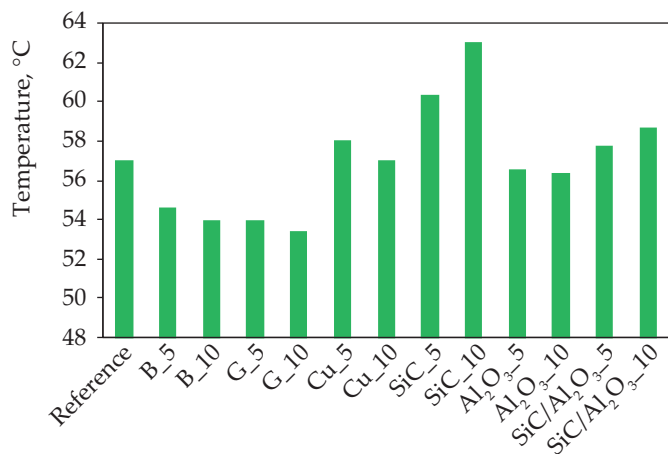


Fig. 8. Effect of the type and filler content on maximum process temperature

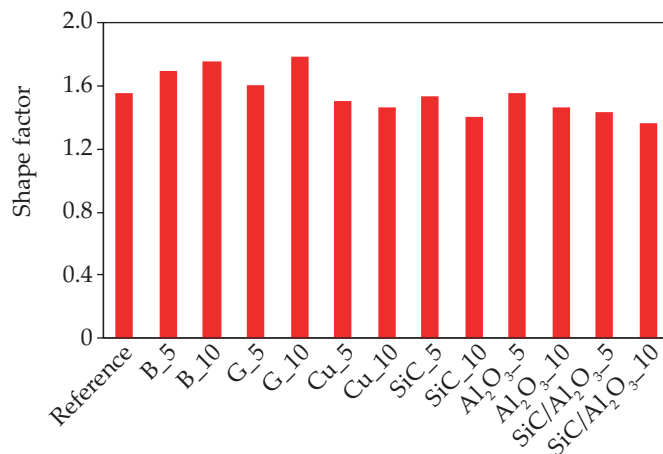


Fig. 9. Effect of the type and filler content on shape factor

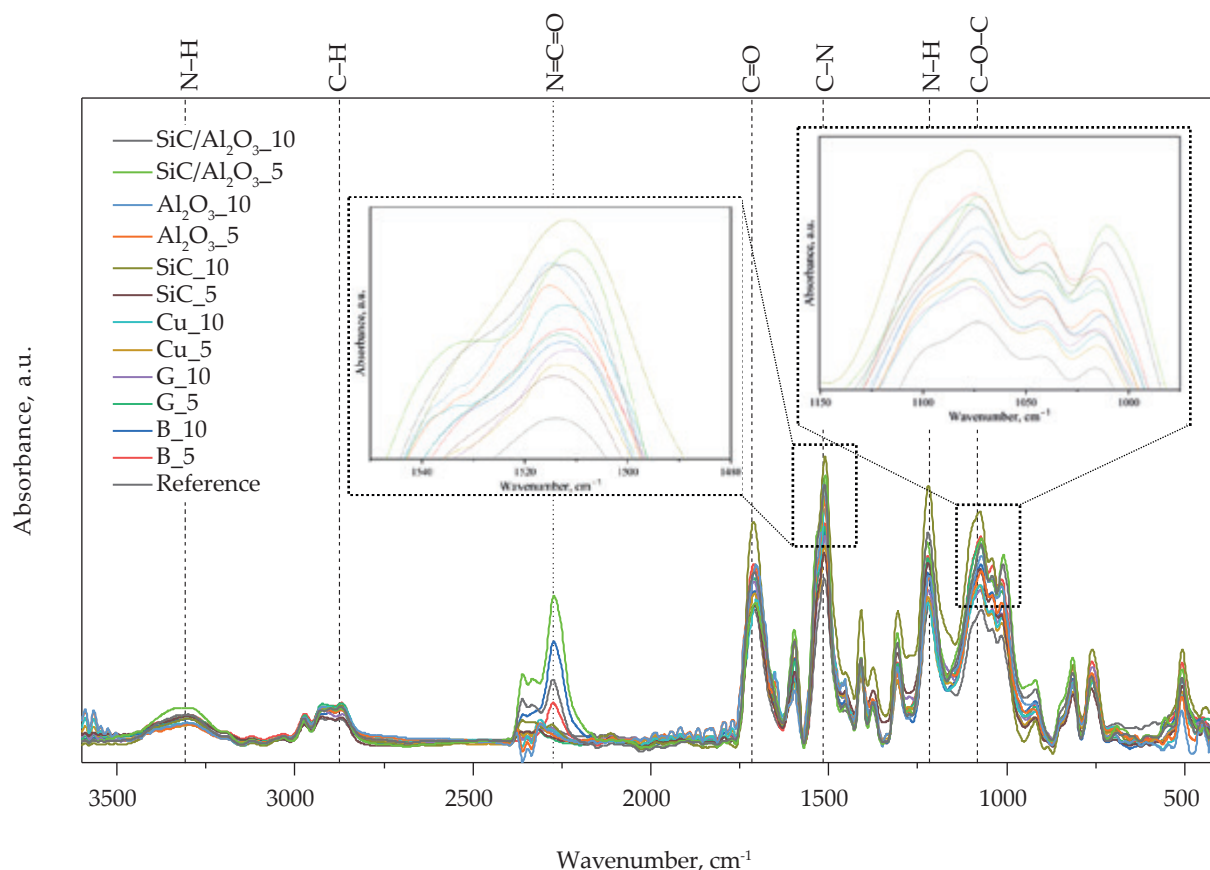


Fig. 10. IR spectra of tested polyurethane foams

polyurethane foams. Compared to the reference foam, the SiC₅ composite shows a longer reaction growth completion time than SiC₁₀. The lower the SiC content, the longer it takes to complete the reaction. It should be noted that foams modified with SiC/Al₂O₃ show a relationship like other composites, i.e., the higher the content, the longer the reaction completion time. The most significant difference of approximately 60% was observed for foams modified with 5 and 10 wt% Al₂O₃, and the slight

test difference of approximately 5.5% for composites with bentonite and copper.

Figure 7 shows the influence of the type and content of fillers on the tack-free time. The proposed foam modification shortens the time to obtain a tack-free time. Most composites have a much shorter time to obtain a tack-free time, approximately 300±20s. The exception is the B₁₀ composite, which may result from water absorption (Fig. 13).

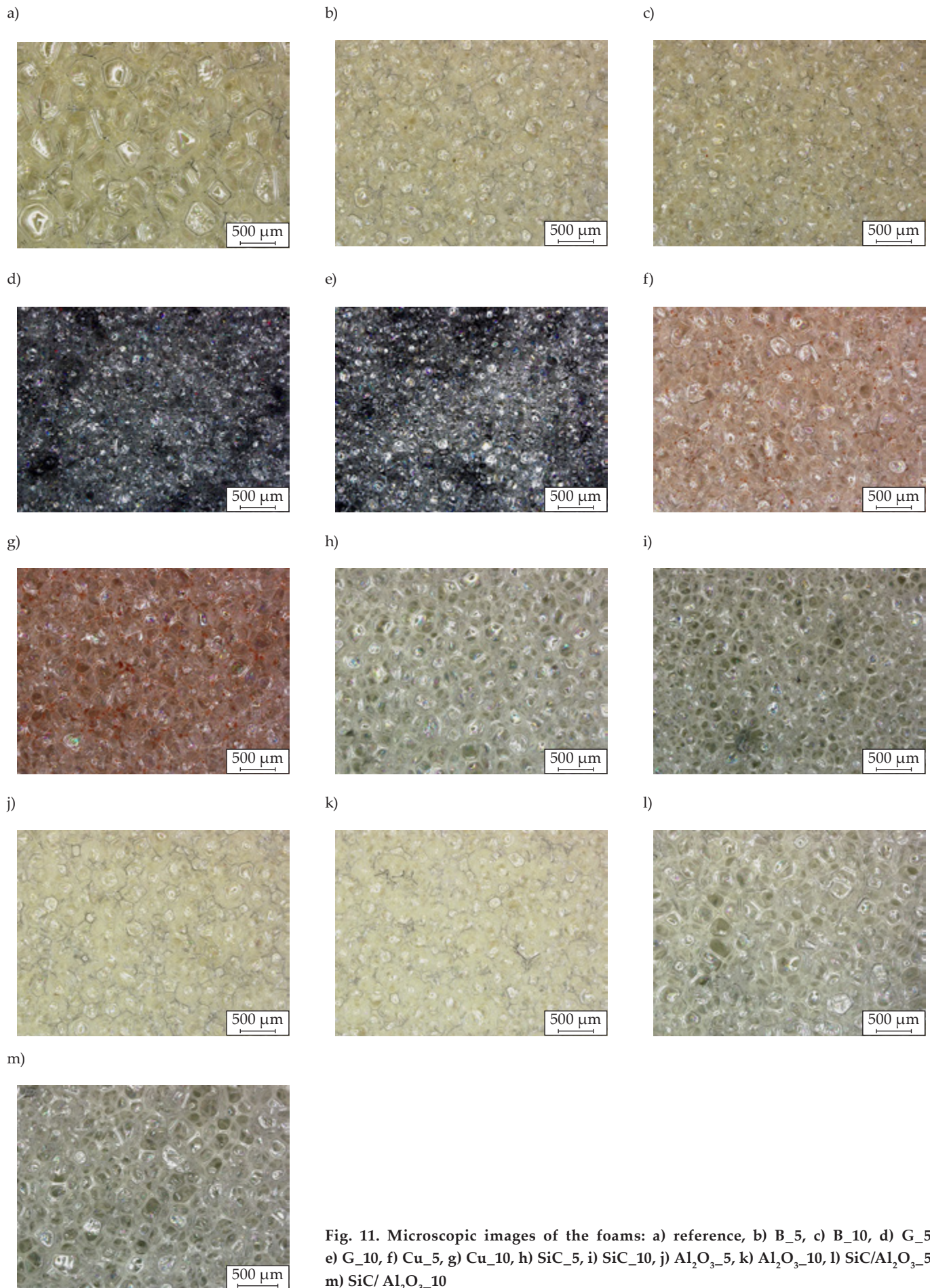


Fig. 11. Microscopic images of the foams: a) reference, b) B_5, c) B_10, d) G_5, e) G_10, f) Cu_5, g) Cu_10, h) SiC_5, i) SiC_10, j) Al₂O₃_5, k) Al₂O₃_10, l) SiC/Al₂O₃_5, m) SiC/ Al₂O₃_10

The highest temperatures observed result from the physicochemical properties of the fillers themselves, their distribution on the surface, and the fineness of the structure, as indicated in Fig. 8. The course of the foaming reaction strictly depends on the properties and content of the fillers used, which also significantly affects the shape factor (Fig. 9) and the foams structure and morphology (Figs. 10 and 11). As can be seen, in the case of the reaction start time, the introduction of the tested fillers reduces the aspect ratio, i.e., the modification used affects the degree of expansion. For bentonite and graphite, an increasing dependence of the content on the shape factor is observed, and the composites B_5, B_10, G_5, and G_10 show a greater increase than the reference foam. The rest of the composites show a lower shape factor than the reference foam. For these composites, the higher the content, the lower the coefficient.

Structure and morphology

Figure 10 shows FT-IR spectra of the reference and modified foams indicating typical stereoscopic fingerprints of polyether polyol [27]. Moreover, all tested spectra exhibit a broad peak of N-H stretching vibration in 3323 cm^{-1} as well as C=O stretching vibrations in urethanes in 1726 cm^{-1} . The absorption peaks in 1547 cm^{-1} and 2253 cm^{-1} indicate the presence of an isocyanate component. The addition of fillers altered the intensity of characteristic peaks depending on the filler. However, there was no unequivocal dependence on the filler type and its incorporated quantity. The greatest changes are observed for foams filled with 5 and 10 wt% of SiC and SiC/Al₂O₃. These composites exhibit the highest absorption peak in 1099 cm^{-1} and 1513 cm^{-1} , which are characteristics of polyurethane. The absorption band in 1513 cm^{-1} is attributed to NH₂ group, whose growth is related to polyurethane synthesis [28]. This could be caused by embedding carbon found in silicon carbide into the chemical backbone of polyurethane foam.

Figure 11 shows microscopic images of the reference and modified foams. The foams have a closed-cell struc-

ture, and the fillers only affect its fragmentation. The greatest fragmentation was observed for composites with copper. The fillers are built into the bridges (ribs) between the pores, and their distribution is not even. The observed agglomerates are the result of the processing. It would be necessary to verify the mixing time of the polyol with the filler before adding the isocyanate. Specific properties and observation of the microstructure clearly indicate that the course of the foaming reaction depends on the content, type and physicochemical properties of the filler.

Density

Figure 12 shows the average results from measuring the density of foams modified with fillers related to the reference sample. All fillers increase the apparent density compared to the reference sample by approximately 30% on average. The greatest changes were recorded for graphite, copper, and silicon carbide composites.

In the case of graphite (0.4 g/cm^3), a uniform distribution of the filler across the thickness is observed (Fig. 11d). In the case of copper and silicon carbide, the factor influencing the density of the foams was bulk density. The introduced fillers significantly influenced the fragmentation of the structure. It can also be noticed that the scatter of results for the mentioned composites is larger (larger standard deviation). This is related to the area from which the samples were taken. The apparent density depends on the fillers' bulk density and content in the composites.

Water absorption

The average results of water absorption tests are shown in Fig. 13. The highest water absorption was observed for composites with bentonite, which is related to its hygroscopicity. B_5 has almost 50% higher absorbency than the reference foam, and B_10 absorbs twice as much. The difference between B_5 and B_10 was approximately 50%. An important factor influencing absorbency seems to be

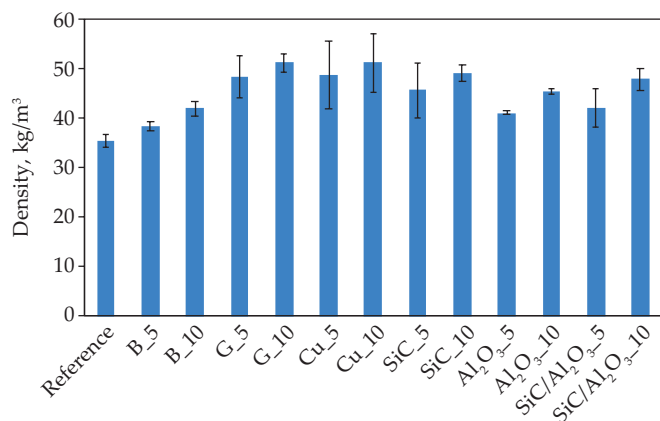


Fig. 12. Effect of the type and fillers content on foam density

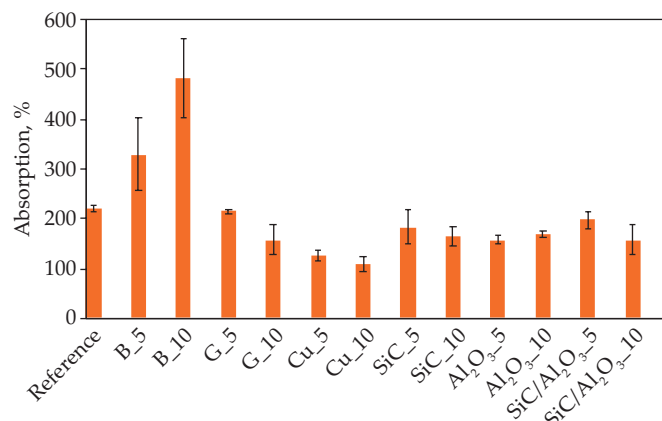


Fig. 13. Effect of the type and filler content on water absorption

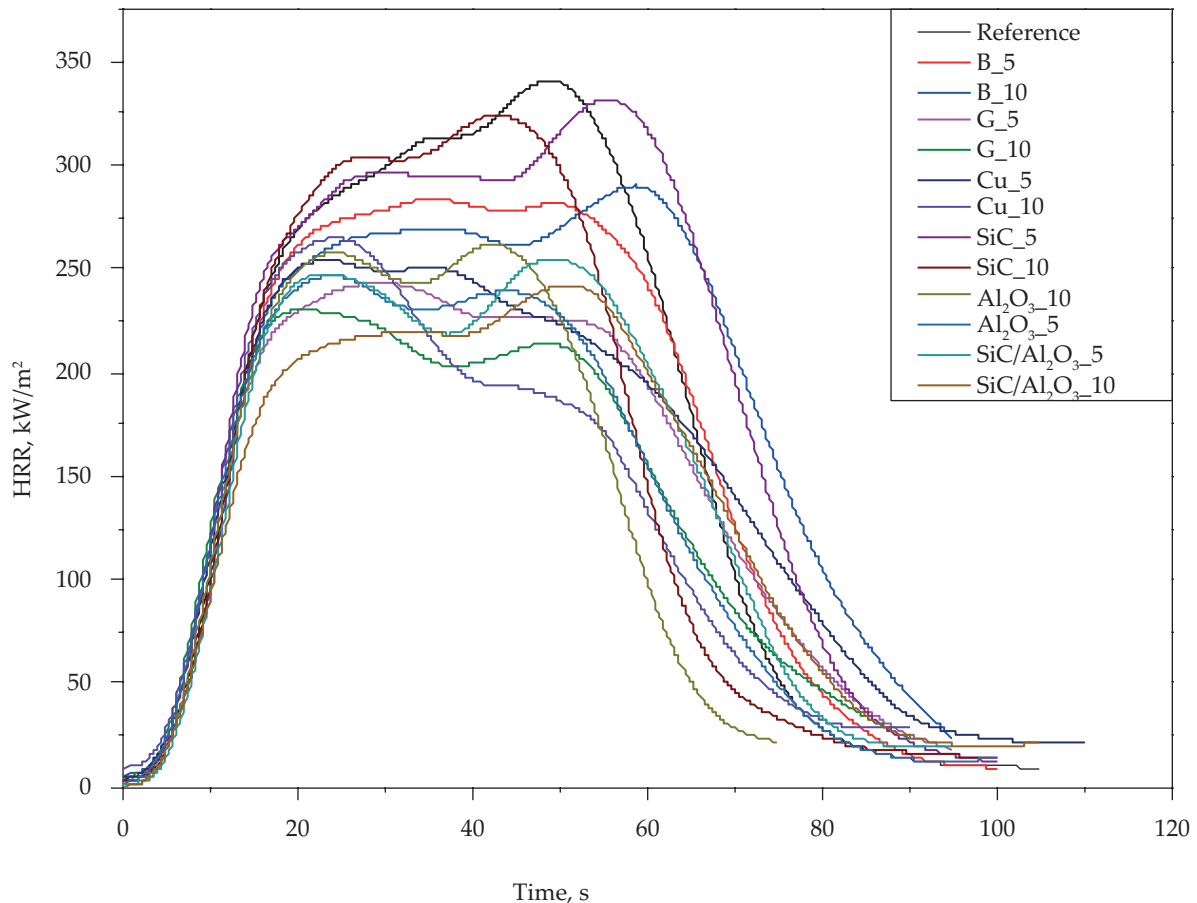


Fig. 14. Effect of the type and filler content on heat release rate (HRR)

the hygroscopicity of the fillers themselves. The remaining composites show values lower than the reference foam. If fillers do not absorb water, they reduce the water absorption of the foams. The lowest absorbency was demonstrated by the C_5 and C_10 composites, which is also related to the microstructure of the composites (smallest pores).

Flammability

The flammability properties are presented in Table 1 and Fig. 14. The effect of fillers on the properties of polyurethane foams was analyzed according to the methodology described above, i.e., time to ignition, total heat release, smoke release, mass loss rate, heat of combustion, and toxic gas production. The release of smoke in fire conditions may limit visibility, complicate evacuation, and cause additional toxicological threats. As can be observed (Table 1), foams containing bentonite showed increased smoke emission values, which may increase the risk level in the case of a fire. In terms of safety, fillers that limit smoke production are preferred.

Total heat release is critical because it reflects the material's potential contribution to fire development. Composites filled with bentonite showed greater heat release, which suggests that this filler may contribute to more intense combustion. Copper and graphite have

a neutral effect on heat release, indicating the potential stability of these composites during fire.

The intensity of the burning of material is related to the rate at which the fire spreads. The lower the combustion intensity, the longer the reaction time. Composites filled with graphite showed moderate combustion intensity, which may indicate potentially better fire characteristics.

Foams modified with Al_2O_3 and SiC showed similar values of mass loss rates to the unfilled sample, suggesting that these fillers do not positively affect combustion in terms of mass loss rates. However, foams filled with copper showed moderate heat efficiency, which may indicate stable fire properties compared to other fillers. Cu_5 and Cu_10 composites showed lower CO (toxic gas) efficiency values, which is beneficial from the point of view of fire toxicology. SiC_5 and SiC_10 composites were characterized by increased combustion intensity, which indicates that they may contribute to faster fire spread.

The choice of filler significantly impacts the fire safety of polyurethane foams. Analysis of the behavior of the tested materials allows for a deeper understanding of their reaction to fire and their potential impact on fire safety. Each of the fillers - aluminum oxide, silicon carbide, bentonite, copper, and graphite cause changes in the value of the composite properties, which translates into its overall flammability. Foams filled with Al_2O_3 show moderate behavior in terms of time to ignition. SiC composites

Table 1. Flammability properties

Property	Content, wt%												Reference		
	Al ₂ O ₃		SiC/Al ₂ O ₃		B		Cu		G		SiC				
	10	5	10	5	10	5	10	5	10	5	10	5			
Ignition time	s	3	2	1	2	3	2	1	2	1	2	1	2	3	3
Flameout time	s	81	85	71	66	87	74	82	83	84	69	79	69	79	69
Total heat release	MJ/m ²	14.1	14.2	13.4	12.1	18.1	12.6	15.6	12.9	14.4	15.5	19.1	17.5	19.1	17.5
Average specific MLR	g/(sm ²)	13.27	16.78	15.19	17.37	15.63	14.97	14	14.7	13.52	19.9	17.92	19.24	17.92	19.24
Total smoke release	m ² /m ²	724.2	698.3	632.7	605.5	810.2	507.2	685	615.1	738.9	712.7	886.1	809.3	886.1	809.3
Total smoke production	m ²	6.4	6.2	5.6	5.4	7.2	4.5	6.1	5.4	6.5	6.3	7.8	7.2	7.8	7.2
MARHE	kW/m ²	181.9	194.6	192.4	197.3	223.8	185.2	197.5	180.2	191.5	236.7	248.5	246.6	248.5	246.6
Heat release rate mean	kW/m ²	140.56	157.91	140.8	173.46	200.36	147.82	148.67	151.81	159.33	162.46	200.99	174.47	200.99	174.47
Heat release rate peak	kW/m ²	244.29	257.73	250.49	263.5	295.1	268.82	254.7	231.47	243.78	325.73	336.43	347.02	336.43	347.02
Effective heat of combustion means	MJ/kg	17.76	16.24	17.92	16.95	18.29	18.09	18.09	17.74	17.73	18.19	18.47	17.49	18.47	17.49
Effective heat of combustion peak	MJ/kg	52.73	77.81	47.84	66.24	74.52	54.82	38.45	70.72	76.67	74.48	44.91	58.4	44.91	58.4
Mass loss rate mean	g/s	0.069	0.086	0.069	0.09	0.096	0.0871	0.073	0.075	0.079	0.079	0.096	0.088	0.096	0.088
Mass loss rate peak	g/s	0.177	0.202	0.173	0.18	0.179	0.165	0.192	0.145	0.155	0.202	0.188	0.223	0.188	0.223
Specific extinction area means	m ² /kg	884.88	776.57	816.1	809.96	803.52	693.01	765.13	810.43	878.29	815.68	838.33	797	838.33	797
Specific extinction area peak	m ² /kg	1503.28	1186.47	2759.13	1669.25	2633.77	1344.75	2150.26	1561.07	1139.97	2840.13	2491.58	3023.29	2491.58	3023.29
Carbon monoxide yield mean	kg/kg	0.0836	0.0758	0.0868	0.081	0.0657	0.0857	0.0855	0.0771	0.0779	0.0877	0.0719	0.0741	0.0719	0.0741
Carbon monoxide yield peak	kg/kg	0.1996	1.7139	0.6297	1.1186	0.7585	0.3793	0.3503	0.4038	0.1641	3.302	1.4451	827.48	1.4451	827.48
Carbon dioxide yield mean	kg/kg	1.16	1.04	1.16	1.07	1.22	1.18	1.19	1.15	1.18	1.19	1.2	1.14	1.2	1.14
Carbon dioxide yield peak	kg/kg	3.35	20.04	16.56	17.37	29.79	3.95	10.54	6.12	5.91	15.96	23.29	22773.26	23.29	22773.26

have a similar profile, which may indicate their stability in the initial phase of fire. Foams with bentonite are particularly noteworthy regarding heat release, especially at 10 wt% content. Copper, especially in lower concentrations, can shorten the time to ignition, which is important for quick response to fire. However, the mere presence of copper does not necessarily translate directly into higher combustion intensity or higher heat release, making it potentially beneficial in reducing fire risk. Graphite does not seem to significantly impact the time to ignition initiation or total heat release, but it may contribute to stabilizing the flammability of foams; however, its smoke release or exhaust toxicity requires additional analysis.

After conducting comprehensive analyses of the flammability properties of polyurethane foams with various fillers, aluminum oxide is the most promising material in reducing fire risk. Foams filled with Al_2O_3 were characterized by a moderate time to ignition and did not show a significant increase in heat release. Neither Al_2O_3 nor SiC showed any significant effect on the total smoke emission. Composites with Al_2O_3 do not seem to affect the toxicity of exhaust gases (reduction of CO and CO_2 emissions).

Considering all the analyzed parameters, aluminum oxide is the filler that can best contribute to increasing the fire safety of polyurethane foams. Its ability to limit heat generation, not significantly increase smoke generation, and shorten the time to ignition makes it a prime candidate for applications requiring materials with a reduced fire risk.

CONCLUSIONS

Based on the conducted research, it can be concluded that the course of the cross-linking reaction depends on the content and properties of the filler. Important parameters are density, structure and thermal properties of the filler. The highest temperature of the foaming process was recorded for composites modified with copper and silicon carbide. The shape factor is the highest for composites with bentonite (0.73 g/cm^3) and graphite (0.4 g/cm^3). The lower the bulk density, the higher the degree of expansion. The shape factor is lower for higher density fillers due to the inhibition of the foaming process. The highest water absorption was recorded for composites with bentonite, which is related to the absorption of bentonite itself. The bentonite selected for testing due to its montmorillonite content is characterized by a high swelling coefficient. Flammability tests show that Al_2O_3 has the best fire safety properties. This is due to limited heat released, smoke production and shortened ignition time. This is probably because the fillers are incorporated into bridges (ribs) between individual cells or form a compact layer on the surface. An important factor influencing the obtained results, especially the physicochemical characteristics, is the preparation of the samples and the area from which they were taken.

Authors contribution

M.S. - writing-original draft, conceptualization, writing review and editing, methodology, investigation, references, supervision, validation, project administration; M.C – writing-original draft, writing review and editing, methodology, investigation, visualization, references, validation; S.S. – writing-original draft, writing review and editing, methodology, investigation, visualization, references, validation; B.C. - writing, flammability research; I.G. - flammability research.

All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare no conflict of interest.

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