

# Management of waste fly ash as an epoxy resin filler

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**Abstract:** Epoxy resin composites with the addition of 20–50 wt.% of ash from municipal waste incineration were obtained. The curing kinetics of the composites (DSC) was determined, and the activation energy was calculated using the Kissinger and Ozawa method. In addition, flexural properties, and impact strength, as well as the structure of the composites (XRD, optical microscopy) were investigated. Brittles of the composites increased with increasing the filler content. The addition of ash also affected the kinetics of the resin cross-linking reaction. A change in the activation energy, degree of conversion and cross-linking time was observed.

**Keywords:** fly ash, waste management, epoxy resin, composites.

## Zagospodarowanie odpadowych popiołów lotnych jako napełniacza żywic epoksydowych

**Streszczenie:** Otrzymano kompozyty żywicy epoksydowej z dodatkiem 20–50% mas. popiołu ze spalania odpadów komunalnych. Określono kinetykę utwardzania kompozytów i stopień konwersji (DSC), a energię aktywacji obliczono metodą Kissingera i Ozawy. Ponadto zbadano właściwości mechaniczne przy zginaniu i uderności, a także strukturę kompozytów (XRD, mikroskopia optyczna). Kruchłość kompozytów zwiększała się wraz ze wzrostem zawartości napełniacza. Dodatek popiołu wpływał również na kinetykę reakcji sieciowania żywicy. Zaobserwowano zmianę energii aktywacji, stopnia konwersji i czasu sieciowania.

**Słowa kluczowe:** popiół, gospodarka odpadami, żywica epoksydowa, kompozyty.

With the dynamic development of industry and materials science knowledge, it is also important to develop methods for reprocessing and recovering raw materials or energy. This is in compliance with the principles of a Circular Economy [1, 2]. A positive research trend is the comprehensive study of the entire life cycle of a material, which in the case of composite materials is related to the characteristics of the filler and matrix, or the production and characterization of composites [3–6]. The research cycle ends with the development of methods to manage waste materials. Innovative polymer composites are often the result of this work, where waste materials can be reused as raw materials [7–12].

An example of such material is fly ash from municipal waste incinerators. Municipal waste is segregated and materially recovered in the first stage. The next stage is energy recovery through incineration. The end products are slag, boiler dust and fly ash, and solid residues from flue gas cleaning. These materials undergo stabilization

and solidification processes and are then often disposed of in landfills for non-hazardous and inert waste in accordance with current legislation [13–16]. Interest in these materials has been growing for several years. Efforts are being made to reuse them. Popular options for the management of boiler dust and fly ash include incorporation into concrete, cement, curing for roads and tile production, or the manufacture of geopolymers [17–22].

Among polymeric materials, polymer resin matrices, including epoxy, formaldehyde, and polyester resins, offer practical opportunities for the management of mineral dust materials [23, 24]. For instance, the obtained composites find an application in the construction industry. This study focuses on the development of an alternative and innovative option for the management of fly ash from municipal waste incinerators as a filler for epoxy resins.

The curing kinetics of epoxy resins are critical in determining the final properties of the cured material, influencing factors such as mechanical strength, thermal stability, and chemical resistance [25–29]. The introduction of additives into epoxy systems is an area of growing interest, as these fillers can significantly alter the polymerization process, potentially leading to enhanced or novel material properties [30]. Previous studies have

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demonstrated that the incorporation of fillers can affect the reaction kinetics by modifying the network structure and influencing the cure rate and degree of cure, which in turn impacts the physical properties of the cured resin [31–34]. The modification of epoxy resins with organic fillers has been shown to influence the cure kinetics by affecting the chemical and physical interactions between the resin and curing agents, altering the crosslink density and network morphology [35]. Differential scanning calorimetry (DSC) is a well-established technique for studying the curing kinetics of thermosetting polymers, allowing for the precise measurement of heat flow as a function of temperature and enabling the determination of kinetic parameters such as activation energy, the conversion rate, and key temperatures [36, 37].

Furthermore, particularly important is the characterization of the mechanical properties of epoxy resins filled with organic materials. The interactions between the filler and matrix have a significant impact on subsequent strength, hardness, or impact properties [38]. One of the challenges to be faced during the preparation of highly filled epoxy resin composites is the maximum amount of the filler that will allow to obtain a material with the most favorable relationship between the amount of filler used and the final properties of the composite. Numerous studies have shown that the appropriate choice of filler and its concentration allows to obtain optimal performance properties of composites, including mechanical properties [39]. In the literature, epoxy resin composites with natural fillers are well characterized, but there is still a lack of data about composites with fly ash. Soumyalata showed that composites filled with powdered coconut shells always had significantly better mechanical properties than composites filled with powdered rice husk, including impact strengths as high as 11 kJ/m<sup>2</sup> [40]. Furthermore, Salasinska *et al.* showed that the filling

degree of walnut shell waste epoxy resin providing the highest strength parameters of the composites in bending, tensile and impact tests is 30% [41].

The aim of the research was to obtain a highly filled epoxy resin with waste ash originating from industrial waste incineration and to characterize the properties of the obtained composites. This is one of the potential solutions for using the above-mentioned waste raw material within the framework of secondary waste management. The relationship between the amount of filler added and the properties of the composites was determined. According to the authors' knowledge, highly filled systems based on epoxy resins with ash from waste incineration have not been obtained so far. The effect of the degree of resin filling with ash on the curing kinetics, structure and mechanical properties of the composites was studied. The optimal filler concentration was also determined, at which the highest strength parameters were obtained, thus presenting the potential of the obtained material for applications as low-loaded elements used or working in conditions to which epoxy resin is resistant.

## EXPERIMENTAL PART

### Materials

ES R90 epoxy resin and CES H71A hardener (Connector, Niepruszewo, Poland) were used. The mixture consisted of component A and component B, which contained:

- Component A: epoxy resin based on bisphenol A and epichlorohydrin with an average molecular weight of <700; mono[(C12-14-alkyloxy)methyl]oxirane derivatives (C.E.S. R90 product catalog sheet valid as of 10.01.2024),
- Component B: benzyl alcohol, isophoronediamine, 4,4'-isopropylidenediphenol, oligomeric reaction pro-

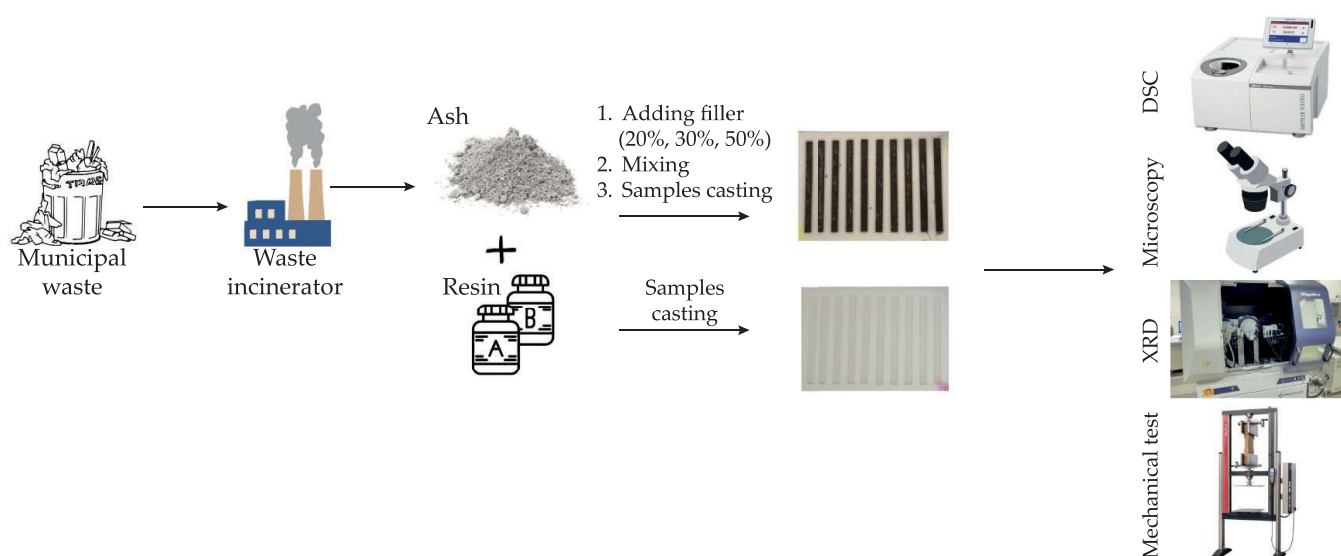


Fig. 1. Scheme of the work done

ducts with 1-chloro-2,3-epoxypropane, reaction products with 3-aminomethyl-3,5,5-trimethylcyclohexylamine, m-xylenediamine; salicylic acid (Safety Data Sheet C.E.S. H-71A valid as of 1/10/2024).

The resin was filled with ash, which is one of the fractions of residues from the incineration of industrial waste. The ash was dried before being added to the resin (for 10 hours at 80°C).

### Composites preparation

The composites were obtained by casting. The filler was used in the amount of 20, 35 and 50 wt (Fig. 1). The filler content is also the sample designation. Reference samples were also obtained from pure resin (without filler). Ash was distributed in component A by intensive mixing for 60 s, then component B was added to the mixture and the whole mixture was mixed for 60 s. The final mixture was poured and distributed in silicone molds 100 × 10 × 4 mm. The samples were cross-linked for at least 48 h before testing according to the resin manufacturer's recommendations.

### Methods

#### Dynamic differential scanning calorimetry

Dynamic differential scanning calorimetry (DSC) measurements were conducted using DSC 6000 instrument (Perkin–Elmer, Waltham, MA, USA) at four different heating rates: 5, 10, 15, and 20°C/min from 0 to 220°C in inert atmosphere (argon, 20 mL/min). Samples were placed in aluminum pans covered by aluminum lids and sealed using a crimper. The total heat of the curing reaction ( $\Delta H_T$ ) was estimated by integrating the area under the peak.

#### X-ray diffraction

XRD analysis was performed using a TUR M-62 (Germany) horizontal diffractometer (CuK $\alpha$  radiation, at 30 kV and anodic excitation 25 mA) with an HZG-3 goniometer. All diffractograms were recorded for angles in the 2 $\theta$  range: 10°–80° with a step of 0.04° (Cu K $\alpha$  radiation, after Ni filtration).

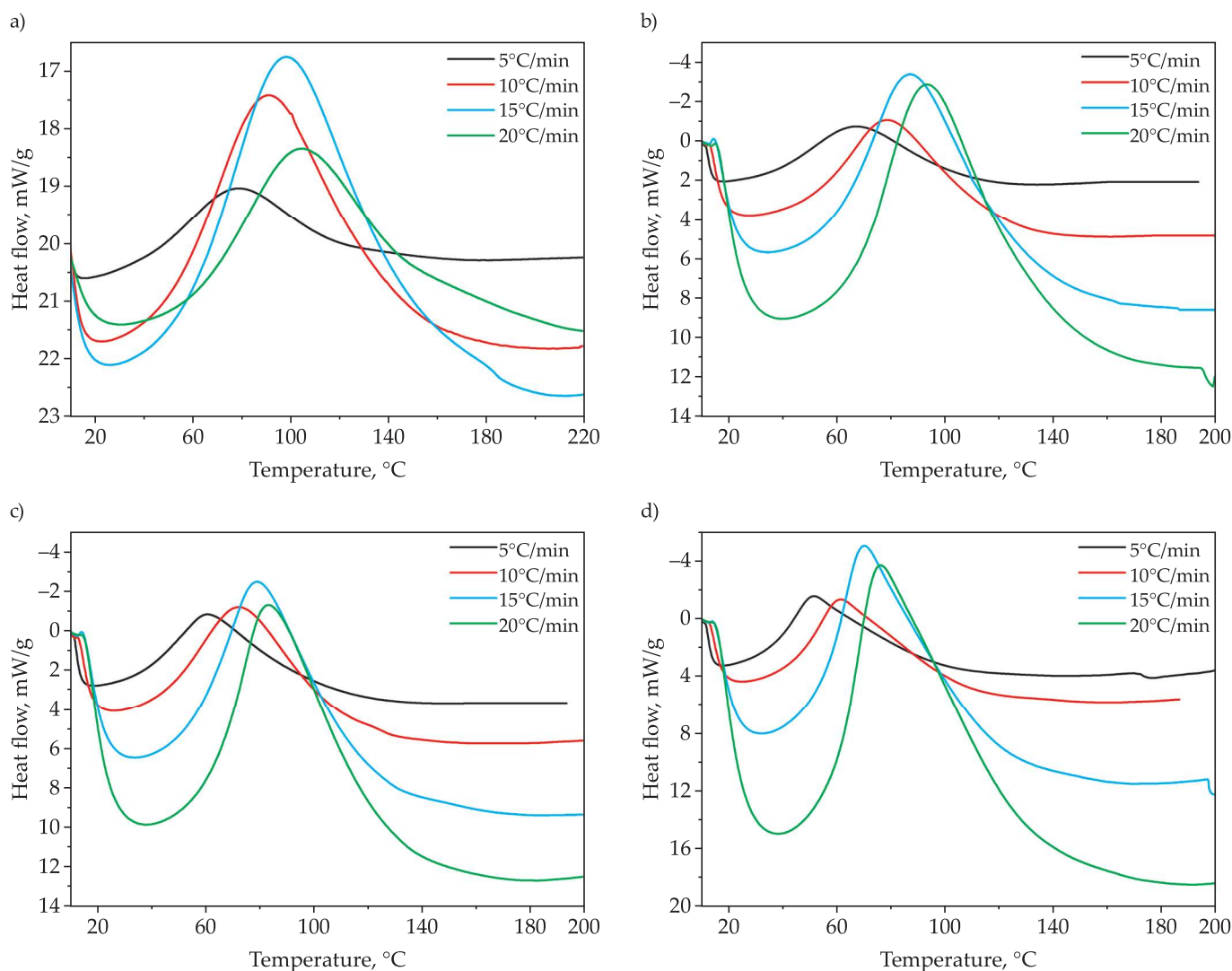


Fig. 2. DSC curves of composites with different ash contents: a) 0 wt%, b) 20 wt%, c) 35 wt%, 50 wt%

## Mechanical properties

Mechanical properties such as flexural properties and unnotched Charpy impact strength were evaluated in accordance with ISO 178 and ISO 179, respectively. Flexural properties were conducted using a Zwick Z020 (Zwick Roell, Ulm, Germany) machine with the load capacity of 20 kN and a crosshead speed of 1 mm/min until a deflection of 6 mm was reached or the sample broke. Impact strength was evaluated using Charpy impact machine Zwick 5120 (Zwick Roell, Ulm, Germany). In each analysis, 10 samples were examined by determining the average and standard deviation.

## Optical microscopy

Microscopic images of the samples were taken using an optical microscope (Optica Microscopes, Ponteranica, Italy). The microscope was equipped with a Toupcam optical camera. Photographs were taken for breakthroughs after mechanical testing.

## RESULTS AND DISCUSSION

### Characteristics of the resin curing process

The DSC curves of the tested composites, measured at four heating rates: 5, 10, 15 and 20 °C/min in argon atmosphere, are shown in Fig. 2. The onset of curing temperature ( $T_o$ ), maximum peak temperature ( $T_p$ ) and endset temperature ( $T_{end}$ ) are summarized in Table 1. The cure enthalpy at each heating rate for each sample was quantified by integrating the area under the exothermic peak ( $dH_T$ ) and is also given in Table 1.

As shown in Fig. 2, the exothermic peaks shift towards higher temperatures with an increasing heating rate. Similar observations were reported in previous works by Thanki and Parsania [42] and Achilias *et al.* [43]. The same change was observed in the case of compositions with fillers, which is also in agreement with the literature reports. Yarahmadi *et al.* [44] showed that the addition of graphene oxide and starch-functionalized graphene oxide resulted in a shift of the peak temperature ( $T_p$ ) towards higher values. In the study of Cruz-Cruz *et al.* [45], a shift towards higher  $T_p$  was observed after the addition of carbon fiber. Analogous results were observed by Ferdosian *et al.* [21] for bio-based epoxy systems comprising DGEBA and different percentages of lignin-based epoxy resin. Saad *et al.* [46] reported that modification of brominated epoxy resin (brominated DGEBA) with organically modified clay (Cloisite 25A) resulted in the shift of  $T_p$  to a lower value with the increase of heating rate, which may suggest the catalytic effect of Cloisite 25A.

The addition of ash caused a decrease in  $T_p$ , which increased with the increase in filler content. For epoxy resin  $T_p$  ranged from 78.1 to 104.6 °C with increasing heating rate, and for the composite with 50 wt.% ash  $T_p$  was in the range of 49.8–69.5 °C (Tab. 1). Kovaleva and Savotchenko [47] showed that the addition of mineral fillers leads to an increase in heat release. However, Ton-That *et al.* [48] reported that modification of epoxy resin with organo-nanoclay (Nanomer I30E) has no effect on  $T_{onset}$  and  $T_p$ . Ferdosian *et al.* [35] found that the maximum curing temperature of lignin-based epoxy resin with BPA-based epoxy resin decreases with increasing the amount of lignin-based epoxy resin.

The initial curing temperature and the peak temperature increase with increasing heating rate. This shift in  $T_p$

**Table 1. Thermal properties of composites determined by DSC during curing**

Filler, wt%	Heating rate, °C/min	$T_o$ , °C	$T_p$ , °C	$T_{end}$ , °C	$dH_T$ , J/g
0	5	17.3	78.1	169.4	343.1
	10	24.6	90.8	204.1	310.3
	15	26.1	98.8	208.7	328.1
	20	33.8	104.6	223.4	278.3
20	5	19.8	65.5	125.6	351.2
	10	29.7	75.4	158.7	332.5
	15	36.8	82.3	186.8	322.3
	20	40.3	86.8	188.7	298.9
35	5	20.1	59.2	139.3	311.5
	10	26.7	69.4	154.7	330.7
	15	36.3	74.3	177.5	301.6
	20	39.3	76.7	177.8	319.3
50	5	19.4	49.8	136.8	289.8
	10	26.2	58.1	154.3	284.5
	15	32.8	64.9	166.5	295.0
	20	39.3	69.5	185.3	287.6



allows for the determination of energy activation using the methods of Kissinger and Ozawa. These methods allowed the analysis of peak temperatures obtained from non-isothermal measurements for the epoxy resin at different heating rates (Fig. 3).

### The Kissinger method

In Kissinger's method, the peak temperature is used to calculate the activation energy of the process ( $E_a$ ). It is assumed that the curing rate is maximum at  $T_p$  [49]. Using the peak temperature data, the  $E_a$  can be determined according to Eq. 1.

$$\ln\left(\frac{\beta}{T_p^2}\right) = \ln\left(\frac{Q_p AR}{E_a}\right) - \frac{E_a}{RT} \quad (1)$$

where  $Q_p$  is calculated using Eq. 2:

$$Q_p = -\left(\frac{df(\alpha)}{d\alpha}\right) = \alpha_p \quad (2)$$

The plot of  $\ln \beta/T_p^2$  against  $1/T$  should be a straight line with slope equal to  $-E_a/R$ .

### The Ozawa method

In this method [50],  $E_a$  is calculated from dependence of the logarithm of the heating rate and the inverse of the exothermic peak temperature, according to the Eq. 3:

$$\ln \beta = \ln \ln\left(\frac{AE_a}{R}\right) - \ln f(\alpha) - 5.133 - 1.052 \frac{E_a}{RT} \quad (3)$$

where  $f(\alpha)$  is a constant function (Eq. 4):

$$f(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} \quad (4)$$

The plot of  $\ln \beta$  against  $1/T$  should be a straight line with slope equal to  $-1.052 E_a/R$ .

In Fig. 3, the inverse peak temperature ( $1/T_p$ ) is plotted against the logarithm of the heating rate ( $\ln \Theta$ ) for the Ozawa method, and ( $\ln \Theta/T_p^2$ ) is plotted against  $1/T_p$  according to the Kissinger equation. The  $E_a$  values derived from the slopes of these plots are shown in Tab. 2. Although these values are closely aligned, the  $E_a$  calculated using the Ozawa method is slightly higher than that obtained via the Kissinger method, a difference observed by other researchers as well [51].

The addition of fillers causes an increase in  $E_a$  in composites with 20 wt% and 35 wt% filler content compared to pure epoxy resin.  $E_a$  of the epoxy resin increased from 58.6 kJ/mol (Kissinger method) and 54.8 kJ/mol (Ozawa method) to 68.5 kJ/mol and 70.5 kJ/mol, respectively, at an ash content of 35 wt%. This has the effect of hindering the cross-linking process of the polymer matrix, which directly affects the structure of the composite and its properties. On the other hand, increasing the content of fillers to 50 wt% causes an unexpected decrease in  $E_a$ . This suggests an acceleration of the curing process by adding ash. This may be due to the presence of functional groups that can facilitate a faster curing rate.

Lem and Han [52] observed a significant reduction in the activation energy of unsaturated polyester resin, decreasing from 83 to 37.5 kJ/mol, upon addition of 50 wt%  $\text{CaCO}_3$ . In the study of Saad *et al.* [46],  $E_a$  showed a tendency to reduce with Cloisite 25A content from 57.9 kJ/mol to 55.7 kJ/mol at 10 wt% Cloisite 25A. Other researchers reported no change in the activation energy of epoxy resin containing 8 wt% carbon black [53]. Saeb *et al.* [54] showed, however, that  $E_a$  increased with the addition of  $\text{Fe}_3\text{O}_4$  and chitosan-modified  $\text{Fe}_3\text{O}_4$  compared to the unmodified resin. Similar results were obtained by Yarahmadi *et al.* [44] in the case of graphene oxide and starch-functionalized graphene oxide and Ferdosian *et al.* [35] for a lignin-based epoxy resin.

The addition of ashes to the resin caused a slower reaction, as evidenced by the decrease in the conversion degree at the maximum polymerization temperature (Table 3).

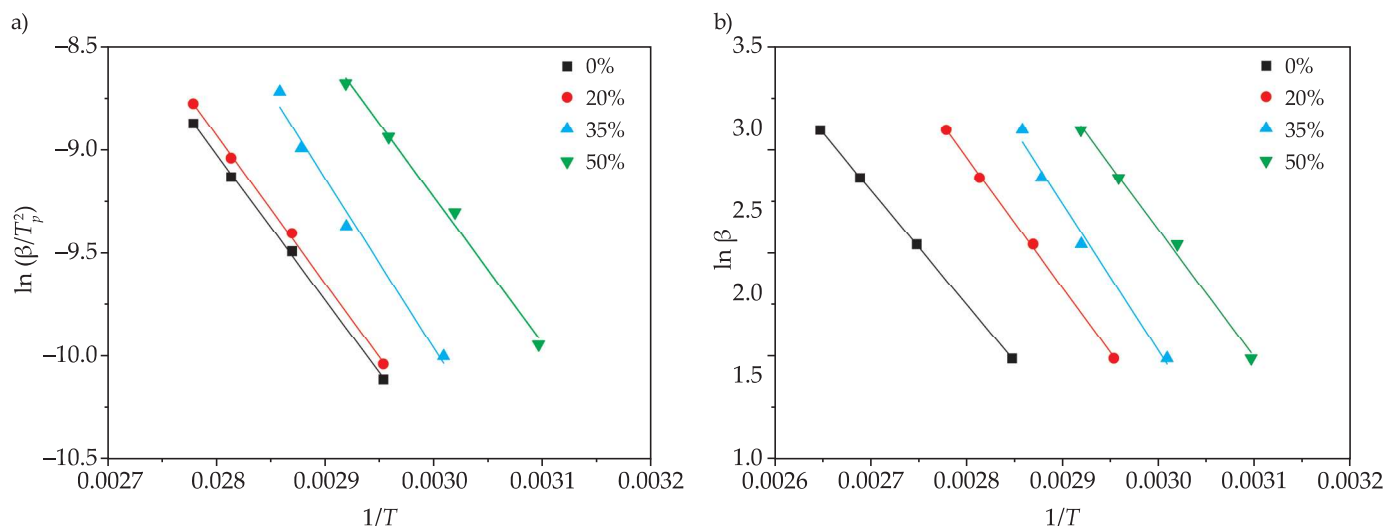


Fig. 3. Plots for pure resin and composites: a) Kissinger method, b) Ozawa method

**T a b l e 2. Activation energy of the tested samples calculated using the Kissinger and Ozawa method**

Method	$E_a$ , kJ/mol			
	0 wt%	20 wt%	35 wt%	50 wt%
Kissinger 58.6		59.7	68.5	58.9
Ozawa	54.8	62.2	70.5	61.2

**T a b l e 3. Conversion degree at the maximum polymerization temperature of the tested samples at different heating rates**

Filler, wt%	Degree of conversion at different heating rates			
	5°C/min	10°C/min	15°C/min	20°C/min
0	0.39	0.37	0.38	0.39
20	0.38	0.29	0.17	0.26
35	0.23	0.23	0.18	0.27
50	0.20	0.21	0.17	0.20

An increase in the activation energy and a decrease in the maximum temperature and degree of conversion at the maximum polymerization temperature may lead to a deterioration of the mechanical properties due to a lower degree of cross-linking and a lower density of the polymer network, as already discussed.

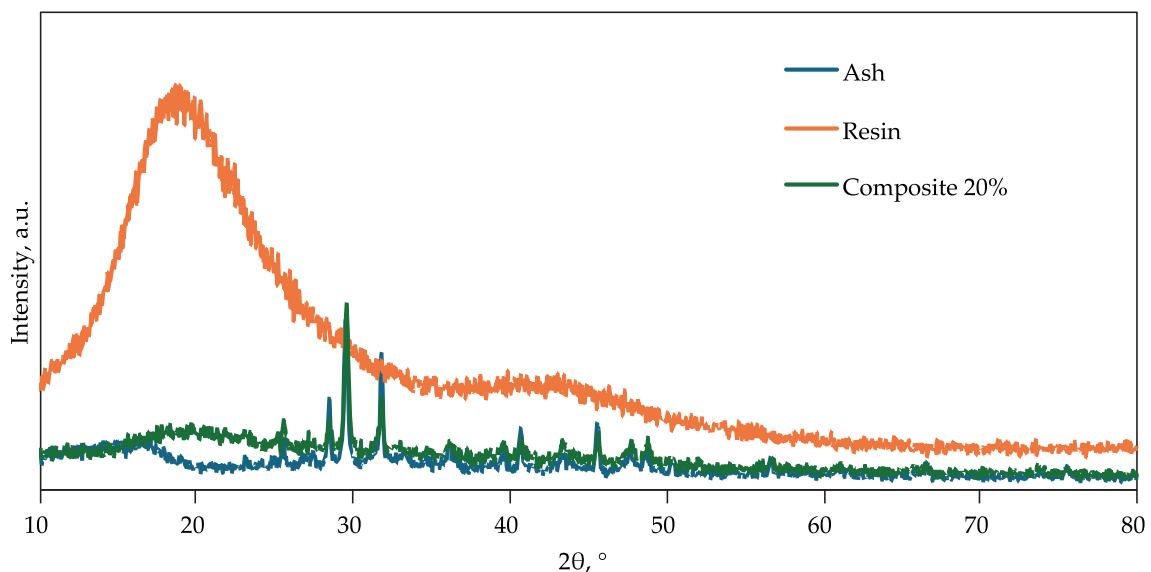
### Structure analysis

The results of XRD analysis are shown in Fig. 4. Measurements are presented for the matrix, filler and composite containing 20 wt% filler. The matrix was found to be amorphous. During the analysis of the structural studies of the filler, maxima of compounds with crystalline structure were observed. After qualitative analysis using XRayan software, maxima were found for compounds such as  $\text{CaCO}_3$ ,  $\text{SiCl}_4$  and  $\text{NaCl}$ . These maxima are also visible in the X-ray patterns of the composites. The presence of such compounds is characteristic of fly ash from waste incineration plants. Bruder-Hubscher also found the presence of these elements and compounds in the ash [55].

Fig. 5 shows optical microscopy images of composites after mechanical tests at the fracture site. The addition of filler affects the morphology of the composites. The micrographs clearly show a change in fractured behavior from more ductile in the case of resin to brittle in the case of composite. During fracture of the samples, the filler was detached from the matrix and left visible voids. This indicates poor adhesion of the filler to the polymer matrix. In addition, the composites are characterized by a porous structure and visible ash aggregates, which adversely affect the transfer of dynamic loads, acting as stress concentration points and facilitating crack propagation. With the increase of the filler amount, more filler aggregates can be observed in the material. Nevertheless, it should be noted that the impact resistance of composite samples is comparable regardless of the amount of ash introduced [39].

### Mechanical properties

Mechanical properties of composites are summarized in Tab. 4. The addition of 20 wt% ash caused a slight


**Fig. 4. XRD patterns of ash, resin and composite filled with 20 wt% ash**

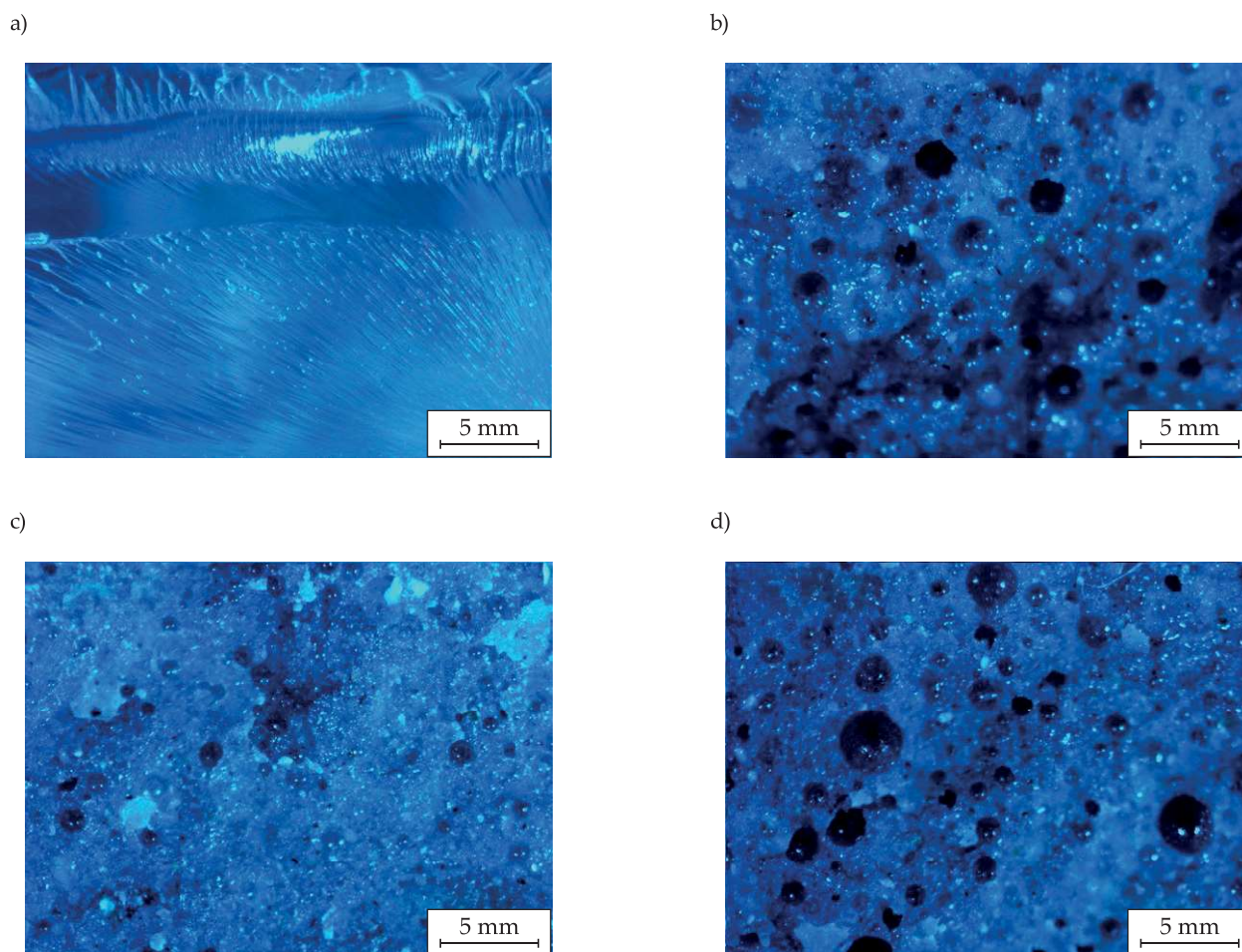


Fig. 5. Optical microscopy images: a) pure epoxy resin, b) 20 wt% ash, c) 35 wt% ash, d) 50 wt% ash

decrease in flexural strength (3 MPa) while maintaining flexural modulus. Further increase in the ash content resulted in an increase in stiffness, as evidenced by a twofold increase in flexural modulus for the 50 wt% filler compared to the 20 wt% filler and a decrease in flexural strength by about 23%. The improvement in stiffness is also reflected in the smaller deflection arrow. The pure resin and the composite containing 20 wt% of ash did not crack at a deflection of 6 mm, while the composite containing 50 wt% of filler cracked at a deflection of 2.4 mm. The dynamic properties differ significantly from the static properties. After adding ash to the resin, regardless of its content, impact strength decreased by about 7.5 times, and in the case of the 50 wt% filler content even by 10 times.

The deterioration of mechanical properties is a direct result of the influence of ash on the resin curing process, as shown above. In addition, the incompatibility between the ash particles of complex chemical composition and the epoxy resin also has an effect, preventing efficient load transfer between the matrix and the filler [39]. Ash particles function as notches during the impact test, reducing the energy required to break the sample, which is also confirmed by the significant deterioration of impact resistance of epoxy resin samples filled with 15–35 wt% different types of renewable fillers [56]. It should be noted, however, that during bending, samples filled with 20 wt% ash achieved properties comparable to those of the pure resin. The study [57] showed that flexural strength was highest for composites filled

Table 4. Selected mechanical properties of examined samples

Filler, wt%	Flexural modulus GPa	Flexural strength MPa	Deflection arrow mm	Impact strength kJ/m <sup>2</sup>
0	0.9±0.1	22.0±2.7	–	42.2±4.1
20	0.9±0.1	19.4±2.5	–	5.6±0.6
35	1.1±0.2	17.6±2.5	5.0±0.6	5.6±0.5
50	2.0±0.3	14.9±1.2	2.4±0.4	4.1±0.1



with powdered plant-based materials with a filler concentration of 15–30 wt%, depending on the selected raw material, which is in line with the results obtained in this work. Similarly, Abakel *et al.* [58] showed that the properties determined in the static tensile test were also optimal for composites filled with 20 wt% walnut shells modified with carboxylic acids. Improving the compatibility of the matrix with the filler through appropriately selected ash modification can have a beneficial effect on impact strength, increasing the application potential of the obtained materials towards low-load components.

Particle size can also affect the mechanical properties of composites. In this work, the filler was used without any modification. Hence, one of the directions of development of the presented research problem may be the mechanical or chemical modification of the filler.

## CONCLUSIONS

In this work, the effect of municipal waste incinerator waste ash on the curing process, structure, thermal and flexural properties, and impact strength of epoxy resin was investigated. The addition of ash affects the curing process of epoxy resins by lowering the peak cure temperature ( $T_p$ ) and enthalpy of curing, increasing the activation energy ( $E_a$ ), and reducing the conversion, which indicates a slower curing process and reduced cross-linking. XRD analysis identified crystalline compounds in the ashes, which remained in the composite and influenced the thermal properties. The addition of 20 wt% ash had no effect on the flexural properties of the epoxy resin. However, the addition of a larger amount of ash (35–50 wt%) resulted in increased stiffness and a significant decrease in flexural strength and impact strength due to the stress concentration around the ash particles. The obtained results confirm the possibility of using waste ash as a filler for epoxy resins.

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## Authors contribution

J.Sz. – methodology, investigation, writing-original draft, writing-review and editing; D.T. – methodology, investigation, writing-original draft, writing-review and editing; G.P. – methodology, investigation, writing-original draft, writing-review and editing; S.B. – conceptualization, supervision.

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## Conflict of interest

The authors declare no conflict of interest.

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## IX Ogólnopolskiej Konferencji Naukowej

### „Biopolimery – źródło nowych materiałów”

online, 3 lipca 2025 r.

**Biopolimery** ze względu na swoje atrakcyjne właściwości, cieszą się coraz większym zainteresowaniem. Wykorzystywane są w przemyśle, medycynie, kosmetologii czy farmakologii.

**Celem Konferencji** jest wymiana aktualnej wiedzy, najnowszych doniesień oraz odkryć związanych z biopolimerami. Podczas Konferencji zostaną poruszone tematy związane z metodami syntezy, analizy, modyfikacji, jak również możliwości bieżącego i potencjalnego wykorzystania biopolimerów.

Do udziału w Konferencji zapraszamy pracowników naukowych z krajowych ośrodków naukowo-badawczych, specjalistów z zakresu ochrony środowiska, inżynierii materiałowej i tkankowej, biochemików, biotechnologów, jak również lekarzy oraz farmaceutów.

#### Tematyka konferencji:

- Nowoczesne biopolimery
- Biopolimery jako biomateriały w inżynierii tkankowej
- Polimery w produkcji inteligentnych materiałów
- Nanotechnologia polimerów
- Polimery biodegradowalne
- Biopolimery w problemach środowiskowych
- Potencjalne zastosowania biopolimerów

#### Ważne terminy:

Zgłoszenie udziału:

**I etap** – 27 marca 2025 r., **II etap** – 8 maja 2025 r., **III etap** – 17 czerwca 2025 r.

Przysłanie streszczenia wystąpienia – 26 czerwca 2025 r.

Przysłanie pełnego tekstu wystąpienia – 14 lipca 2025 r.

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**Miejsce konferencji:** platforma ClickMeeting – online

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