The application of waste toner material as a filler for the synthesis of composites based on Epidian® 5

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Abstract: The reprocessing of polymeric waste materials is an important issue from an environmental point of view. This paper presents the synthesis and characterization of epoxy resin based composites with the application of waste powder from toner (laser printer) as a filler (10 wt.%). The use of waste filler is part of a sustainable economy. For the synthesis of composites, epoxy resin (Epidian® 5) and three amines (ethylenediamine, triethylenetetramine and aniline) were applied. The presence of the amines compounds in the samples was confirmed by the ATR/FT-IR spectra on which were noted the stretching vibrations band the C-N groups around 1037 cm^{-1}. Materials were analyzed by the TG/DTG and DSC analysis, which showed the influence of kind the crosslinking agent and the presence of filler on their thermal properties. The glass transition (T_g) of materials occurs in the temperature range from 39.8 to 64.2°C. The highest values were achieved for the triethylenetetramine (TETA), which indicates the highest cross-linking of this composite. It has been shown that materials about satisfactory can be obtained, using cheap and waste substances by simple synthesis which does not require expensive polymerization initiators.

Keywords: waste materials, composites, epoxy resin, Epidian®, amines

1. Introduction

The rapid development of the economy results in an increase in the amount of produced waste. Their storage and inappropriate utilization represent huge threat for the natural environment. In addition, the limited use of fossil fuels, which are currently the main sources of energy, leads to a growing interest in recycling various types of waste (Kuity et al., 2014; Mohanty et al., 2002). All actions leading to the propagation, rational and thoughtful use of natural resources are highly advisable and fit into an economy of sustainable development (Ramos et al., 2019; Ghozali et al. 2017).

When people process secondary raw materials, the reduction of consumption the primary raw materials, is achieved (Gaikwad et al., 2017). Recovering and processing decrease the amount of waste, thus decreasing the amount of space in landfills (Sienkiewicz et al., 2017). Finally, using raw materials that are carriers of energy is an element of efficient energy management. The basic principle of recycling is the maximization of the re-use of the same materials with the least amount of raw and energetic materials needed to process them (Ruan et al., 2011).

Plastics are a commonly used material that suits very well for recycling (Kumar et al. 2017). In economic activity, plastic waste can occur in many different ways, but mainly it is used packaging or production waste. Basically, all types of plastics are recoverable and can theoretically be recycled many times. The rare exceptions that don't fit for recycling mainly go to thermal heating plants for recovery in the form of energy in communal heating systems (Zlamparet et al. 2017). Recovered plastics are used to produce, for example, rubbish bags, plastic boxes and bins, acoustic screens, flower pots and garden furniture. The use of plastic waste materials is also the subject of numerous scientific studies. Oliwa and co-workers (2020) compared the effect of type and content of fractions of

recycled glass fiber reinforced plastics on the mechanical and other properties of epoxy composites were investigated. Researchers were found that the amount and type of recyclated fraction affect the functional properties of powder-epoxy resin composites. Lamba et al. (2021) present the developments in the application of plastic waste as a constituent of construction material. The plastic waste is used e.g., as a modifier or substitute of cement, binder or fine aggregate. They are used to produce bricks, tiles, concrete and roads. The influence of plastic waste addition on mechanical and strength properties has been discussed.

Toner in laser printers is generally a very fine-ground plastic. Depending on the model of printer and the specific producer, toner can contain 85-95% of polyester (PET). In the past, other types of plastics were also used in toners, such as styrene-acrylate, which producers gradually resigned due to its low effectiveness and proven toxicity to the human body (Moffat et al. 2004; Wang et al. 2011, Yang et a. 2013). In this way, modern laser printers bake onto paper micro-particles of the same type of plastic that is used to produce PET bottles for packaged drinks. Other ingredients of toner powder are among others polypropylene wax, carbon black, other pigments, silicon dioxide and others (Fink J.K., 2013). By analysing the chemical composition of the toner, we can claim that it suits fully as a filler for polymer composites (Yordanova et al., 2014). The use of this waste filler leads to a reduction of the amount of monomers used for the synthesis of the composite.

The main objective of our research was the synthesis of composites based on epoxy resin Epidian® 5 (bisphenol A propoxylate diglycidyl ether) crosslinked with various amines (ethylenediamine, triethylenetetramine, and aniline) with the addition of a filler in the form of a waste powder, formed in used toner. The effect of the amine used, differing in the number of amino groups, on the physico-chemical properties was evaluated. The chemical structure of the materials was confirmed by ATR/FT-IR analysis. The thermal properties of composites were tested by using DSC analysis and thermogravimetry. Additionally, the hardness of the composites was evaluated.

2.   Materials and methods

2.1. Materials

Epidian® 5 (bisphenol A propoxylate diglycidyl ether) (Ciech Sarzyna) was used as a monomer/ the main component of the polymeric matrix. Table 1 contains the main characteristics of the epoxy resin used. The three amines: ethylenediamine (EDA) (Sigma), triethylenetetramine (TETA) (Merck) and aniline (ANI) (Merck) acted as a cross-linking agent. As filler in all composites black powdered toner from a laser printer was employed.

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Table. 1. Specification data of Epidian® 5

Form:	highly viscous liquid
Color:	light yellow
Boiling point:	> 200°C
Flash point:	> 200°C
Auto-ignition temperature:	> 500°C
Epoxy number:	0.480 - 0.510 mol/100g
Density at 20°C:	1.17 g/cm^3
Viscosity at 25°C:	20,000-30,000 mPa · s
Solubility:	ketones, esters, alcohols and aromatic hydrocarbons

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2.2. Preparation of samples

Epidian® 5 (bisphenol A propoxylate diglycidyl ether) (Ciech Sarzyna) was used as a monomer/ the main component of the polymeric matrix. Table 1 contains the main characteristics of the epoxy resin used. In addition, polymerization of the epoxide was carried out, resulting in a reference three samples containing no filler. The ratio of the epoxy resin to the amine in each case was 10:1 (wt.%) (Gargol, M. and Podkościelna, B.; 2019). The chemical structures of the crosslinked resin in the

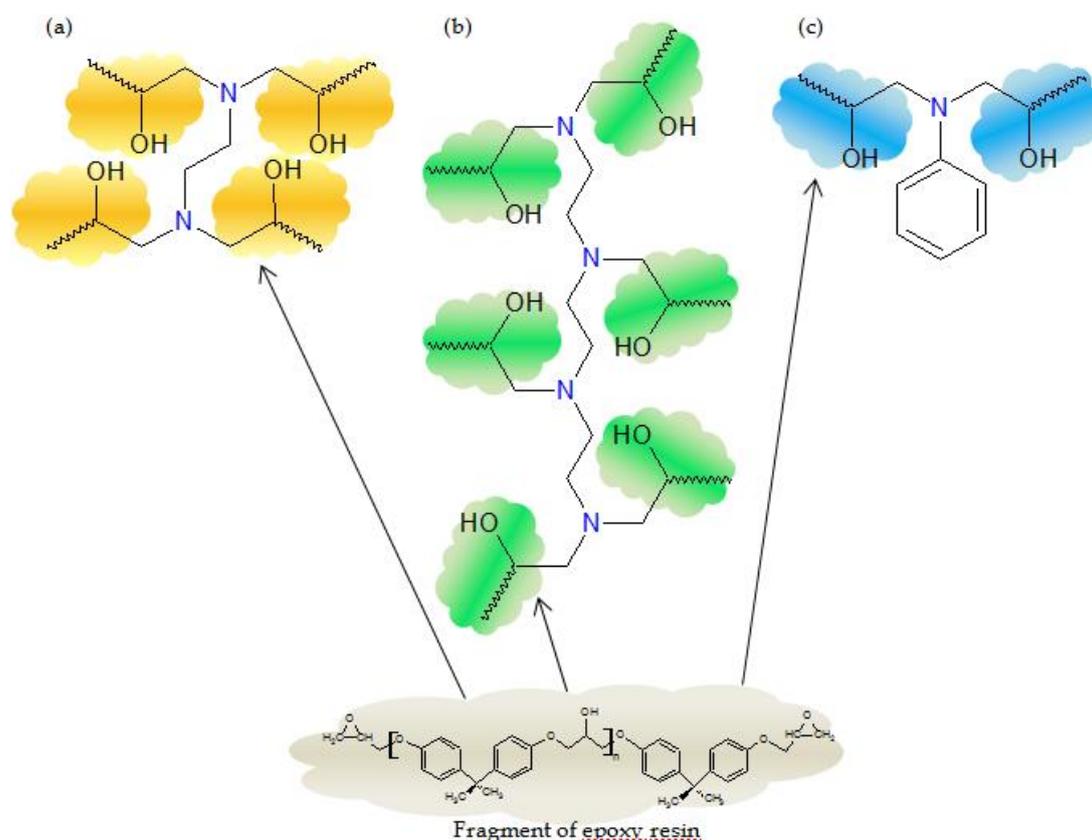composites are presented in Figure 1 and the detailed information about synthesis is presented in Table 2.

Fig. 1. Simplified structure of the epoxy resin matrix cured by: (a) ethylenediamine, (b) triethylenetetramine, (c) aniline

Table. 2. Composition of the polymeric samples cured by amines

Number of sample	Type of sample	Mass of Epidian® 5 (g)	Mass of amine (g)	Name of amine	Quantity of filler (wt.%)
1	EP-EDA+T10%	10	1	ethylenediamine	10
2	EP-TETA+T10%	10	1	triethylenetetramine	10
3	EP-ANI+T10%	10	1	aniline	10
4	EP-EDA	10	1	ethylenediamine	0
5	EP-TETA	10	1	triethylenetetramine	0
6	EP-ANI	10	1	aniline	0

2.3. Characterization of the products

Attenuated total reflection-Fourier transform infrared (ATR/FT-IR) spectra were obtained using a Bruker FT-IR spectrophotometer TENSOR 27 (Bruker GmbH, Mannheim, Germany). Spectra were recorded in the frequency range of 3600 – 600 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans in the absorbance mode and using the powdered samples.

The dynamic thermogravimetric analysis (TG/DTG) was performed in helium atmosphere (flow 25 cm^3/min) with the use of a thermal analyzer STA 449 F1 Jupiter (Netzsch, Selb, Germany). The 12.0 ± 0.05 mg samples (placed in Al$_2$O$_3$ crucibles) were heated in the temperature range of 25 – 600°C at a heating rate of 10°C min^{-1}. As a reference, an empty Al$_2$O$_3$ crucible was used.

The DSC thermograms were obtained with the use of a Netzsch 204 calorimeter (Germany). The 10.0 ± 0.05 mg samples were placed in the aluminum pans with pierced lids, whereas an empty

aluminum crucible with a pierced lid was used as a reference. Dynamic scans were performed in the temperature range from -20 to 250°C in the nitrogen atmosphere with a flow rate of 30 cm³/min.

The hardness of the analyzed samples was measured using a Shore D method using a Zwick 7206/H04 hardness apparatus (Germany) at 25 °C. The indenter of the Shore apparatus was immersed in different places of the sample. The readings were taken after 15 s at the temperature of 22 °C. Five measurements were made for all the samples and the average values were calculated.

3.   Results and discussion

3.1. ATR/FT-IR analysis

Characterization of the chemical structure of all tested materials was made by Attenuated Total Reflection-Fourier Transform Infrared spectroscopy (ATR/FT-IR). Characteristic bands of the functional groups are visible on the curves. The courses of spectra are similar because the main component of these compositions was cured epoxy resin (Fig. 2.).
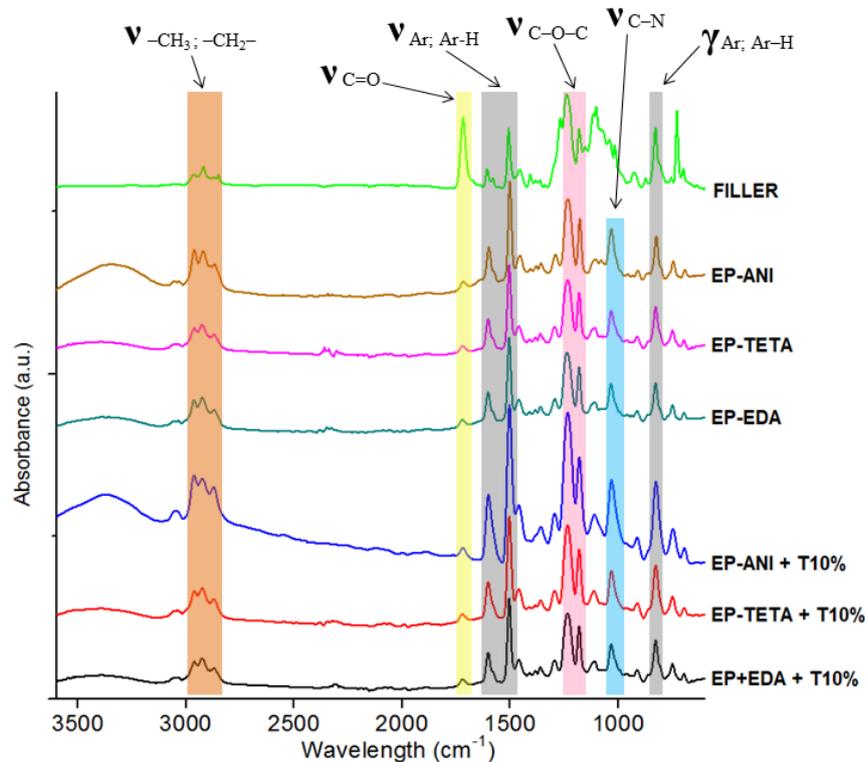

Fig. 2. The ATR/FT-IR spectra of the composites and three reference samples without the filler

The Epidian® 5 has aromatic rings in its structure. For this reason, the vibrations of these groups are visible in all spectra. The signals from 1505 to 1603 cm⁻¹ correspond to the symmetric and asymmetric stretching vibrations in the aromatic ring. Additionally, around 828 cm⁻¹, the medium signal of deformation vibrations of Ar and Ar-H is notable. Its location indicates that aromatic carbon atoms number 1 and 4 are substituted. In the 3363-3391 cm⁻¹ range, the maxima of wide bands derived from the stretching vibrations of the residual -OH groups or the adsorbed water are found. This signal can be more intensive due to the presence of amine and a strong tendency towards water absorption by these molecules. Additionally, after curing of the epoxy resin chains, -OH groups also exist (formed after opening the epoxy ring). These bands are greatest in the case of samples containing of the aniline. The signals from 1458 to 1461 cm⁻¹ can be attributed to the deformation vibrations of methyl and methylene groups. Characteristic bands of both types of groups are visible also as two signals: the first peak from 2962, to 2966 cm⁻¹ and the second in the range: 2924-2926 cm⁻¹. These peaks correspond to the symmetrical and asymmetric stretching vibrations of these structure fragments. The presence of the amines contained in the samples is confirmed by the valence band of stretching vibrations in the C-N bonds. This signal is observed on spectra around 1037 cm⁻¹ Moreover the appearance of the

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characteristic vibrations of the carbonyl group (range: 1719 and 1723 cm^{-1}) was observed. The presence of these molecular fragments and C-O-C groups (dublets located near 1182 and 1237cm^{-1}) indicates the existence of ester groups in filler molecular structure. (Gargol et al., 2021, Silverstein et al., 2005).

3.2. TG/DTG analysis of the obtained composites

Thermal stabilities and degradation behavior of the polymeric materials were studied by means of thermogravimetry. The curves obtained from the TG and DTG analyses for all samples are presented in Fig. 3. For each material there were determined the mass loss factors: Initial Temperature

Fig. 3. The TG and DTG curves of the analyzed materials

decomposition (ITD), $T_{10\%}$ (temp. at 10% weight loss), maximum temperatures of individual decomposition steps (T_1, T_2, T_3) and the residual masses (RM) for each sample. The measurements data are summarized in Table 3. The system is considered stable until 2% of its mass is lost.

Comparing the course of the whole TG and DTG curves it can be stated that behavior at high temperature of composites was a bit different than the samples without toner. The initial decomposition temperatures of the analyzed materials range from 260.5 to 306.4ºC. Analyzing the DTG curves one can see that in the case of the materials doped by toner the curves contain three separate signals related to the degradation stages (T_1, T_2 and T_3). Whereas the thermal decomposition of the cross-linked epoxy resins proceeds in a two-step process (T_1 and T_2). On the DTG curves, the first main peak (T_1) takes place between 339.2°C (EP-TETA+T10%) and 376.1°C (for EP-ANI). It is related to the main step of thermal degradation of the polymer matrices (destroying of polymer network) (Sobiesiak et al. 2017, Podkoscielna, 2014). Additionally, a separate and small decomposition stage for each material can be also found on the DTG curves (T_2). The maximum rate of this step appears at the temperature range of 523.5 to 536.9°C and this suggests the degradation of the epoxy matrix follows a complicated mechanism. The addition of toner to the polymer network results in an additional stage of weight loss of the sample (T_3). The range of these effects was from 358.3 to 404°C. Further heating of the analyzed materials above ~540ºC led to their complete thermal degradation. The residual mass of crosslinked samples assessed at the final temperature is in the range from 1.73% (EP-ANI+T10%) to 3.73% (EP-EDA), while for filler RM is 25.24%. The TG and DTG curves for the filler show two stages of thermal decomposition (maxima at: 382.7 and 434.8ºC). It shows that the extent of filler degradation partially overlaps with that of the epoxy resin. This fact causes that the course of TG / DTG curves for composites in the range from about 380 to 430ºC is slightly changed. The high thermal resistance of the filler proves that it is not a compound belonging to the group of polyolefins. Additionally, due to the FT-IR analysis, it can be assumed that we are dealing with polyester.

The above data shows the different effects of used amine on the behavior of obtained samples in higher temperatures. At the earliest, the beginning of thermal decomposition was recorded for samples cured by aniline. The presence of this amine caused, that TG curve slopes are softer than for samples containing TETA and EDA. Two aliphatic amines used to crosslink resin caused, that the residual mass of samples (detected at the end of measurements) was higher than the final masses of materials with aniline.

Table. 3. Results of the TG/DTG analysis of the samples

Type of sample	ITD (ºC)	$T_{10\%}$ (ºC)	T_1 (%)	T_2 (%)	T_3 (%)	RM (%)
EP-EDA+T10%	303.1	339.7	20.3	91.2	29.4	1.79
EP-TETA+T10%	298.2	334.8	13.9	89.7	31.4	2.28
EP-ANI+T10%	294.1	325.2	34.8	84.6	54.8	1.73
EP-EDA	306.4	340.1	12.5	82.9	-	3.73
EP-TETA	313.5	336.1	27.6	83.9	-	2.10
EP-ANI	260.5	302.9	41.9	82.3	-	1.91
FILLER	288.6	357.3	19.8	50.2	-	25.24

In Figure 4, the proposed mechanism of polymer network fragmentation is presented. The suggested mechanism is based on literature data (Salasinska et al., 2019). In the case of natural fillers, their decomposition leads to environmentally safe, small aliphatic hydrocarbons, alcohols, and then H_2O and CO_2. While crosslinking the epoxy resins, the main products of thermal decomposition, (apart from carbon dioxide and water) are phenol (and its derivatives), benzene, toluene and amino derivatives.

3.2. Differential Scanning Calorimetry

The thermal behaviour of the materials was also studied by means of DSC analysis. The DSC curves for these materials in the range -20 to 250°C are presented in Figure 5.
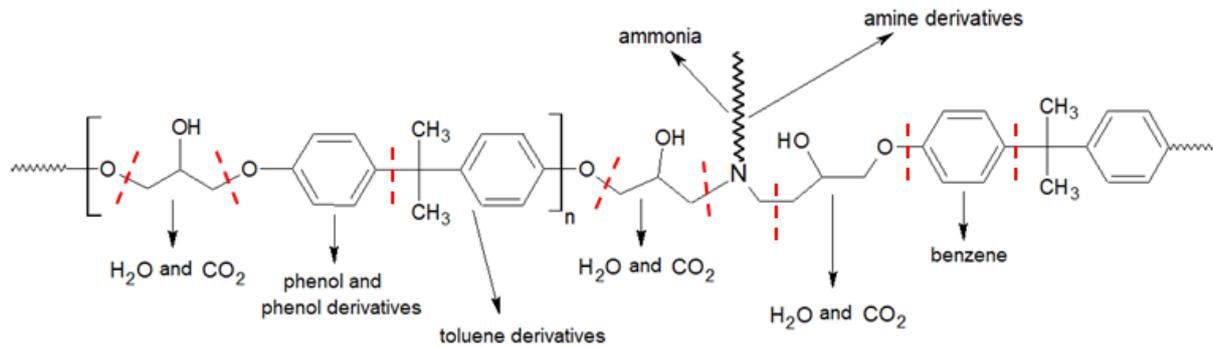
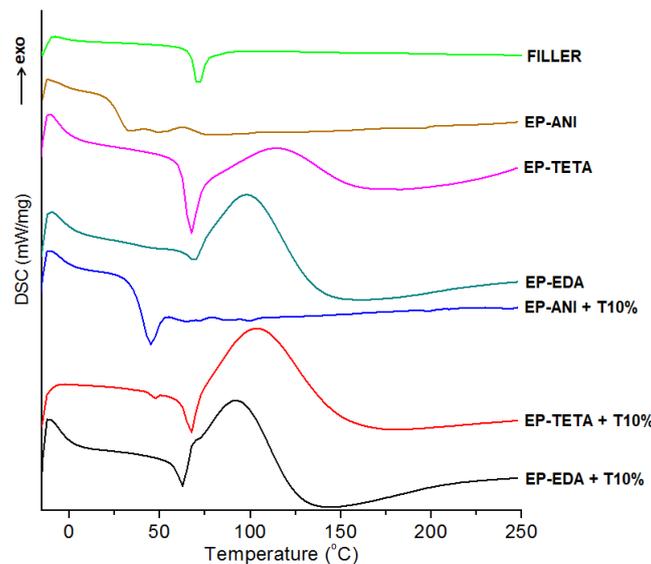Fig. 4. Proposal mechanisms of polymer fragmentation under heating

Fig. 5. DSC curves of the obtained samples

The DSC curves of the composites with toner had a similar course as suitable curves of the reference samples. Table 4 contains numerical data determined from the DSC curves after the heating cycles. It can be seen that, for six samples, a change of the curing agent type of the sample has a significant effect on the shape of the DSC curves. Some differences can be seen related to the course of curves for materials cured by aniline. On the curves, the one endothermic effect could be seen. These signals were connected with the glass transition (Tg) of polymer materials and used filler. These peaks occur in the temperature range from 39.8 to 72.1°C. The values of this parameter are lower for materials with aniline. On the DSC thermograms was detected exothermic effect peaks (exo $_{MAX}$), which maxima occur in the temperature range from 66.4 to 109.8°C. This signal wasn't visible on DSC curve for filler, so this effect is probably a result of the thermally crosslinking of non-cured resin fragments during their heating by residue amounts of unreacted amines. Also, in this case the type of applied amine has an important effect on the shape and intensity of this peak.

Table. 4. DSC data of the samples

Type of sample	Tg (°C)	exo $_{MAX}$ (°C)
EP-EDA+T10%	59.9	91.9
EP-TETA+T10%	63.6	102.9
EP-ANI+T10%	45.4	70.1
EP-EDA	64.2	97.6
EP-TETA	58.3	109.8
EP-ANI	39.8	66.4
FILLER	72.1	-

3.3. The Shore hardness measurements

Measurements of hardness consisted in vertical immersion of the indenter into the materials surface. The hardness values of the obtained materials are presented in Fig. 6. Numerical values of these parameters are expressed in the D scale.
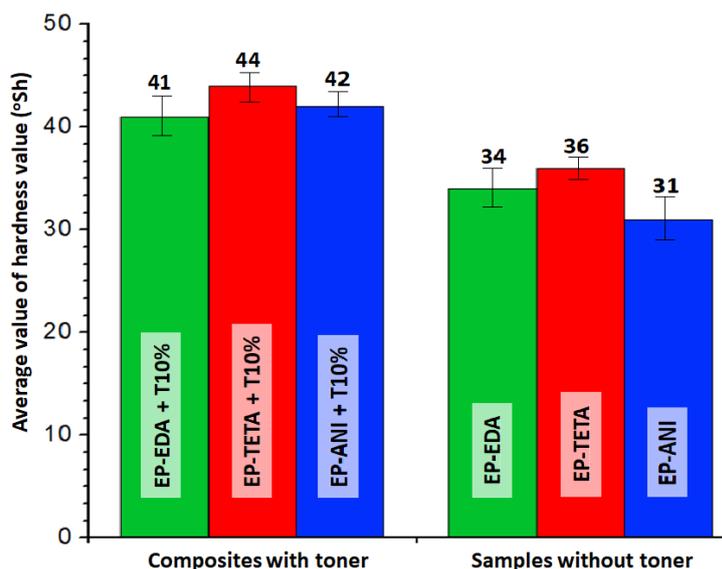

Fig 6. Values of hardness test of synthesized samples

The hardness of the composites was in the range of 41 – 44 units, whereas the values of the hardness of the reference samples were in the range from 31 to 36 units. These results show slight changes with the type of amine used. The highest values of hardness were recorded for the materials cured by TETA. The above results show that the amount of filler (toner) in the samples increases the hardness of the synthesized materials slightly. This parameter is comparable for all composites.

4. Conclusions

In this paper, the synthesis of epoxy resin derived composites is presented. As a filler the waste black powder from toner of laser printer was applied. Six samples with different amounts of filler (0 wt.% and 10 wt.%) were synthesized based on the commercial epoxy resin (bisphenol A propoxylate diglycidyl ether). These composites were cross-linked at room temperature using three different crosslinking agents: ethylenediamine, triethylenetetraamine and aniline. All amines caused the curing of the resin, while the fastest reaction occurred for TETA.

The spectroscopy analysis (ATR/FT-IR) confirms the presence of characteristic groups in composite structures like methyl and methylene groups, aromatic rings or amine fragments. The TGA analysis demonstrated that for the analyzed composites, the TGA and DTG curves had almost the same trajectory. The range of ITD was from 260.5 to 306.4°C. The thermal decomposition of the cross-linked resins proceeds in a two-step process, whereas on DTG curves for the materials doped by filler the curves contain a third additional stage is related to the destruction of filler. The largest thermal residue mass (RM) assessed at the final temperature was for EP-EDA sample. The thermal stability of the samples without used filler are to a small extent more thermally stable, because these materials are characterized by a higher degree of crosslinking. It can be seen that a change of the curing agent type of the sample has significant effect on the shape of the DSC curve. These curves for the obtained samples show one endothermic signal corresponding with the thermally crosslinking of non-cured resin fragments during their heating by residue amounts of unreacted amines. The glass transition region (endothermic effect) spreads over a similar temperature range for materials containing EDA and TETA. The values of this parameter are lower for composite and reference samples cured with aniline.

The hardness results of the obtained materials show that the addition of filler increasing of their hardness. Moreover, the type of amine determines about values of this parameter for these samples. The above results indicate that the waste powdered toner can be used as an eco-filler for the synthesis of cheaper and mechanical resistant polymeric composites based on the epoxy resin, but the quantity of this filler should be selected for their future application needs. These composites could be used for filling cavities in polymer coatings, wood or other surfaces.

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