

Research Article

Recycled Glass Fibres from Wind Turbines as a Filler for Poly(Vinyl Chloride)

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This paper presents the method of using glass fibre with carbon deposit (GFCD), derived from the recycling of wind turbine blades, for production of composite materials based on poly(vinyl chloride) (PVC). Composite materials containing from 1 to 15 wt% of GFCD were produced by plasticising with a plastographometer and then by pressing. The processability and performance were studied. Mechanical properties in static tension, impact strength, and thermal stability were determined. Glass transition temperature was also determined by means of the dynamic mechanical thermal analysis (DMTA). The GFCD percentage of up to 15 wt% was found not to slightly affect the change in the processability, thermal stability, and glass transition temperature. PVC/GFCD composite materials are characterised by a definitely greater elastic modulus with simultaneous decrease of tensile strength and impact strength. An analysis with scanning electron microscopy revealed good adhesion between the filler and the polymer matrix.

1. Introduction

Gaining energy from alternative sources is an area of economy that has seen an increasing growth in the recent years. Particularly intense development is seen in the wind power industry with its production capacity exceeding (only in Europe) 168 GW in 2017 [1]. In the construction of rotors and other structural components of wind farm equipment, large quantities of composite materials based on chemically cured resins reinforced with glass fibres are used [2, 3]. Estimations predict that, from 2040, the quantity of disused wind turbine rotors in the world will amount to 380 thousand tonnes per annum [4].

Three main procedures for disposal of disused wind blades are in place, namely, the storage, incineration, or recycling [5, 6]. The collection and storage of this type of waste is a serious obstacle to the closed-loop economy as it prevents the reuse of raw materials. Therefore, extensive research on how to reuse such material is underway [7].

One of the ways to recycle thermosetting and chemosetting composite materials with glass fibres is their fragmentation (mechanical recycling). By doing so, a filler is obtained that can be used to modify various materials, including polymer composites [7–14].

Worn-out wind turbine blades can also be thermally recycled by incineration or pyrolysis. In the incineration process, significant amounts of energy are recovered and the remaining inorganic part is used as a charge for building materials [15–18]. In the process of pyrolytic decomposition of wind turbine waste, its polymer matrix is decomposed into liquid and gaseous organic compounds, which are then used in the chemical and power industries. Glass fibres are also a product of pyrolysis. As opposed to incineration, pyrolysis is performed at lower temperatures and in anaerobic conditions, which does not change the form of glass fibres. Therefore, these fibres can be reused for the production of polymer composites [19–21].

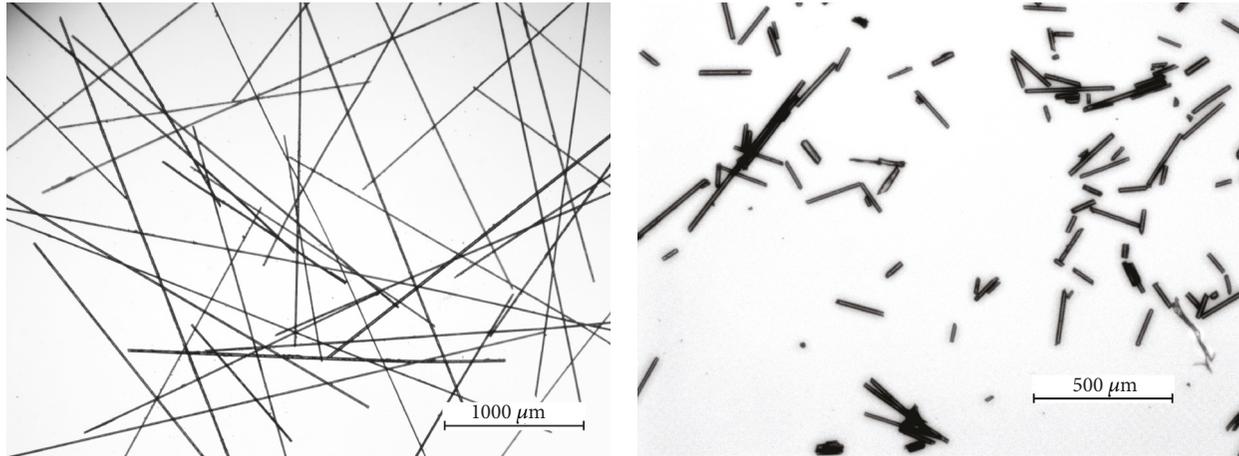


FIGURE 1: An image of GFCD, before processing (left), after processing (right), optical microscopy, and transmitted light.

The poly(vinyl chloride) composites find numerous applications in diverse branches of economy. They are irreplaceable in the production of façade elements, furniture, door and window profiles, strips, fittings, plates, and sewer pipes. Such a widespread use of PVC results from its susceptibility to various modifications of its processability and performance characteristics. This polymer is characterised by good resistance to weather conditions, chemicals, and stability of properties in the long-term use [22].

The purpose of this paper was to create and analyse the possibility of using glass fibres obtained in pyrolytic recycling of disused wind turbine components as a poly(vinyl chloride) filler.

2. Experimental

2.1. Materials. Dry blend of poly(vinyl chloride) (PVC) with the composition of PVC S61 (Anwil SA, Poland) (100 phr), thermal stabiliser Mark 17 MOK (Acros, Germany) (4 phr), and wax Loxiol G22 (Henkel, Germany) (1phr) were used.

Glass fibres derived from the pyrolytic decomposition of wind turbine composites containing carbon deposit on their surface in the amount of 1.8 wt% \pm 0.5 wt% (GFCD) were used as a filler. The pyrolysis process is carried out in microwave reactors under nitrogen atmosphere. The microwaves generated heat the reactor up to 400–600°C, resulting in the disintegration of the chemically cured resin into oil phase and gaseous phase which is removed directly from the reactor while the recovered glass fibre is left in it. The pyrolysis process as well as the elementary analysis of fibres is presented in the papers [19, 20]. Figure 1 (left) shows optical microscope image of the fibres used. The length of individual fibres ranges from 0.15 to 4 mm (average fibre length 2.45 mm) and the diameter from 13 to 20 μm (average fibre diameter 15.3 μm). The fibres used in the composite preparation process were shortened. In order to determine the actual fibre length, PVC composite samples were dissolved in tetrahydrofuran and the filler was then separated from the solution. Figure 1 (right) shows a microscopic image of the fibres separated from the composite. The length of

fibres in the composite ranged from 0.03 mm to 0.6 mm (average fibre length 0.25 mm).

2.2. Preparation of Composites. The dry blends were mixed using a high-speed mixer with filler amounts of 1, 3, 5, 10, and 15 wt% GFCD, respectively. The blends obtained were processed by plasticising in a Brabender plastographometer chamber. The processing conditions were, respectively, chamber temperature 190°C, main rotor speed 30 min^{-1} , chamber free volume 50 cm^3 , and processing time 10 min. In this study, it has been decided to take measurements at a constant charge mass 66 g [23–26]. The cooled PVC/GFCD were ground using a laboratory mill with a hob cutter (own design) equipped with a sieve with a mesh diameter of 4 mm. The obtained materials were pressed with a hydraulic press to form plates (120 x 120 mm, thickness of 1 mm, 2 mm, and 4 mm). The pressing conditions were, respectively, temperature 190°C, preheating time 2 minutes, pressing time 2 minutes, and pressure 12 MPa. From the composite plates obtained, specimens were cut for further testing. The reference material, a PVC blend with no fibre addition, was processed in the same conditions.

2.3. Determination of Processability. Determination of processability was carried out during the processing of blends in the plastographometer chamber (methodology was described in Section 2.2) while recording changes in the torque and temperature of the kneaded material over time. An exemplary plastogram of PVC processing containing 5 wt% GFCD is shown in Figure 2. A plastographometric study was carried out, determining the specific values: M_X , maximum torque at the fusion point (X); T_X , temperature at the fusion point; t_X , time to reach the fusion point; M_e , torque at the end point; and T_e , temperature at the end point. The results are the average of 4 measurements.

2.4. Determination of Time of Thermal Stability. The time of thermal stability was determined with the Congo red method. A material specimen after processing (ca. 0.5 g) was placed in a glass test tube with an inner diameter of 4.7 mm and a

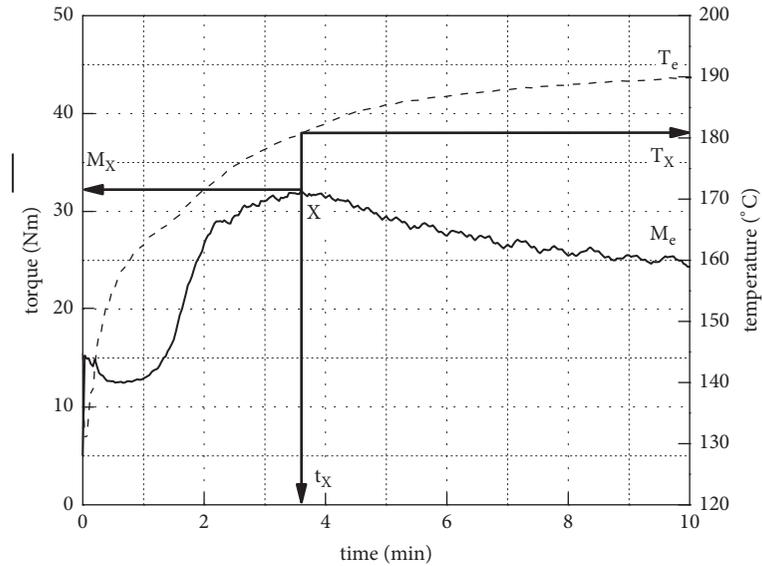


FIGURE 2: An exemplary plastogram of PVC processing containing 5 wt% GFCD.

wall thickness of 0.65 mm. In the upper part of the test tube, Congo test paper was inserted to the depth of 3 mm. The tube was placed in a stand so as to immerse its end in an oil bath heated to 200°C. The result of the test is the time after which the material is destroyed and hydrogen chloride as a PVC decomposition product [27], determined as the first visible change in the colour of the test paper, is released. The results presented are the average of three measurements.

2.5. Thermogravimetric Analysis. Thermogravimetric tests were carried out using a TG 209 F3 Netzsch Group (Germany, Selb) device, in the temperature range 30–900°C, at a rate of 10°C/min, in air atmosphere. The weight of specimens was 10–16 mg. The measurement of each material was repeated min. 3 times. A specimen for TGA tests was taken from a pressed plate prepared for testing of mechanical properties. From the analysis of thermograms, the temperature of thermal stability ($T_{5\%}$) was described. Temperature of thermal stability was defined as temperature at which 5 wt% weight loss of the specimen is observed. Undecomposed residue in 900°C after combustion (RM) was also established. From the DTG curve, the temperature was determined in which the polymer material decomposition is most intense (T_{DTG}). The TGA analysis of the filler used was performed in the same conditions.

2.6. Determination of Mechanical Properties. The mechanical properties in static tension were determined in accordance with EN ISO 527. Standardised test specimens (type IBA) were cut using a CNC milling machine. The test speed was 10 mm/min. The maximum stress σ_m and the strain in the maximum stress ε_m were determined.

Charpy impact test was carried out in accordance with EN ISO 170-1. For the tests, specimens without notches were cut from pressed plates using a CNC milling machine. A pendulum with a nominal impact energy of 4 J was used in the tests.

2.7. Determination of the Glass Transition Temperature. The glass transition temperature (T_g) was determined on the basis of changes in the value of the storage modulus (E') and the tangent of the loss angle ($\tan \delta$) depending on the temperature. The tests were carried out using a DMA Artemis device from the Netzsch Group (Germany, Selb). The determination was made in a three-point bending system (spacing of supports 20 mm, specimen width 10 mm, thickness 1 mm), with a strain of 10 μm in the temperature range 25°C–120°C with an increase rate of 2°C/min. The strain was set with a frequency of 1 Hz. The relatively small amplitude and low strain frequency allow for a measurement in a linear viscoelastic range and low value of the loss modulus [28, 29]. The range of the glass transition was characterised by the value of T_g determined as the beginning of a rapid decrease in value E' (T_g onset) and the maximum of the $\tan \delta$ peak value [30].

2.8. Statistical Analysis. To determine the significance of differences between the mean values analysed the Student t -test for independent samples has been employed. The calculations were performed using the Origin Pro 8.6 software using the Two-Sample T-Test. The comparison of mean values was carried out in pairs at the significance level of 0.05.

3. Results and Discussion

Figures 3–5 present the results of plastographometric measurements.

An addition of GFCD into PVC results in a significant reduction of fusion time of the blend being processed. What is important, acceleration of fusion of PVC/GFCD composites does not relate to changes in torque and temperature at the fusion point. The value of M_x of the processed PVC blends containing from 0 to 15 wt% of the filler changes slightly in the range of 32–35 Nm. T_x decreases by 4°C compared to unfilled material with the increase of the filler amount

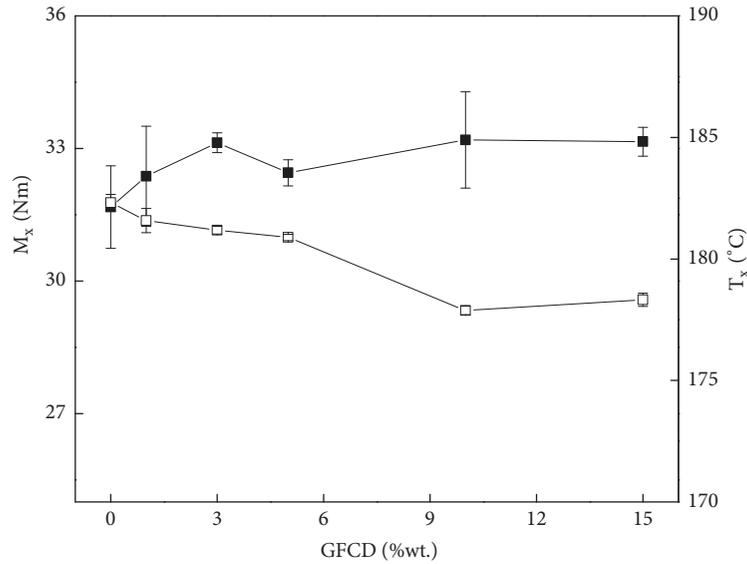


FIGURE 3: Torque at the fusion point M_x (closed symbols) and temperature of the material at the fusion point T_x (open symbols) versus GFCD content in PVC.

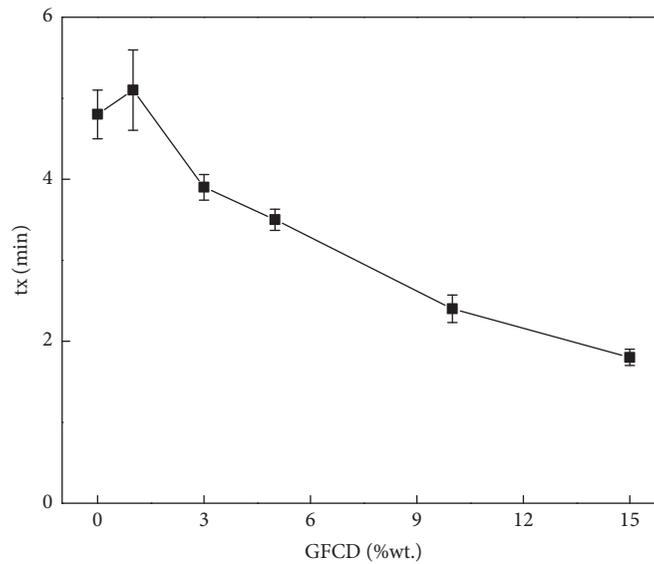


FIGURE 4: The relationship between the fusion time t_x and GFCD content in PVC.

to 15 wt%; however, this change should be explained by a shorter material processing time, rather than by the changes in processability. This is proven by the analysis of the values of M_e and T_e . It was found that the increase in GFCD percentage in PVC of up to 15 wt% did not significantly affect both the recorded torque at the end and the final temperature of the processed blend. Based on the plastographometric analysis, it can be concluded that the addition of the filler favours the disintegration of PVC grains [31], which determines its faster fusion, but this filler does not cause substantive changes in the processability of PVC.

For a proper production of composites based on a PVC matrix, it is necessary to determine their thermal stability. The time of thermal stability determines the approximate possible

time of PVC processing at a given temperature, while the temperature of thermal stability determines the temperature above which the PVC decomposes rapidly. The relationship between the time of thermal stability of PVC/GFCD composites and the filler percentage is shown in Figure 6. Adding the filler in the amount of 3% results in a reduction of the time of thermal stability by 2 minutes compared to unmodified PVC. No significant change in the time of thermal stability of composites containing 3% to 15% GFCD has been found.

Also a slight reduction in the thermal stability (determined by TGA method) of PVC foams with glass fibres was pointed out in [32]. The authors explain that this is caused by a greater thermal conductivity of glass fibres which may accelerate the penetration of heat into the PVC matrix

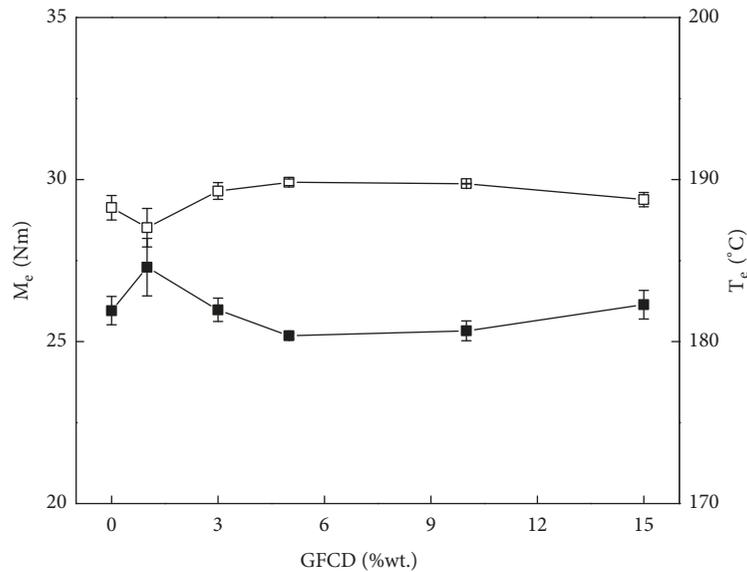


FIGURE 5: Torque at the end point M_e (closed symbols) and the temperature at the end point of the material T_e (open symbols) versus GFCD content in PVC.

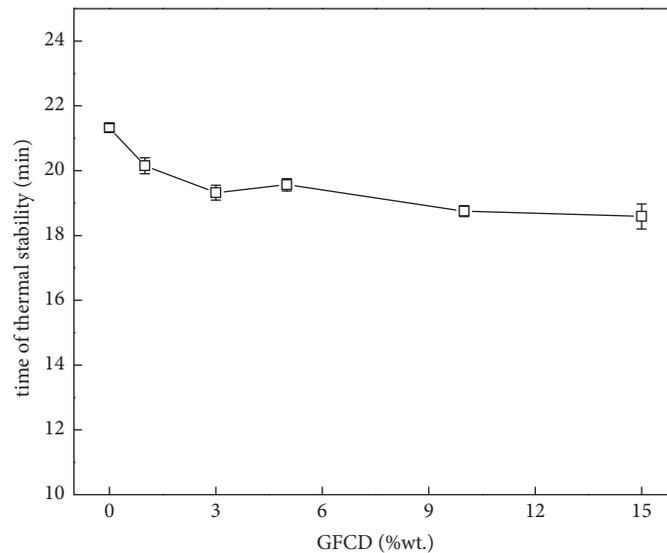


FIGURE 6: The relationship between the time of thermal stability of composite materials and the GFCD content in PVC.

and its faster degradation. However, it should be noted that accelerated fusion of composites (in the case of a 15% GFCD content by more than 3 minutes) results in a longer processing time of the composite at a higher temperature and higher shear forces. This undoubtedly affects the thermal stability of the composites obtained. Nevertheless, the time of the thermal stability of composites in the range of 17–21 minutes at 200°C allows proper processing under the same conditions as in the case of the unfilled PVC.

Figures 7 and 8 show exemplary TGA and DTG curves of poly(vinyl chloride), filler used, and composite containing 5 and 15 wt% of GFCD filler. The analysis results are given in Table 1.

On the TGA thermogram of fillers in the temperature range of 450–650°C, the c.a. 2 wt% loss of the initial sample

mass was observed. It was connected with the decomposition of carbon residues after the pyrolysis. The decomposition of PVC and PVC/GFCD composites take place in two stages. In the first, in the temperature range of about 260–340°C, the autocatalytic dehydrochlorination of PVC macromolecules and the release of the decomposition product in the form of HCl in an amount of approx. 53 wt% related to PVC mass occurs [27, 33, 34]. At the same time, in the structure of macromolecules in reactive points, after separation of the Cl^{\bullet} and H^{\bullet} radicals, formation of double bonds and creation of polyene groups take place. The released by-product HCl additionally catalyses and accelerates the destruction of PVC macromolecules. In the next stage of decomposition in the temperature range 340–600°C, the secondary crosslinked polyene structure oxidises and also the carbon residue is

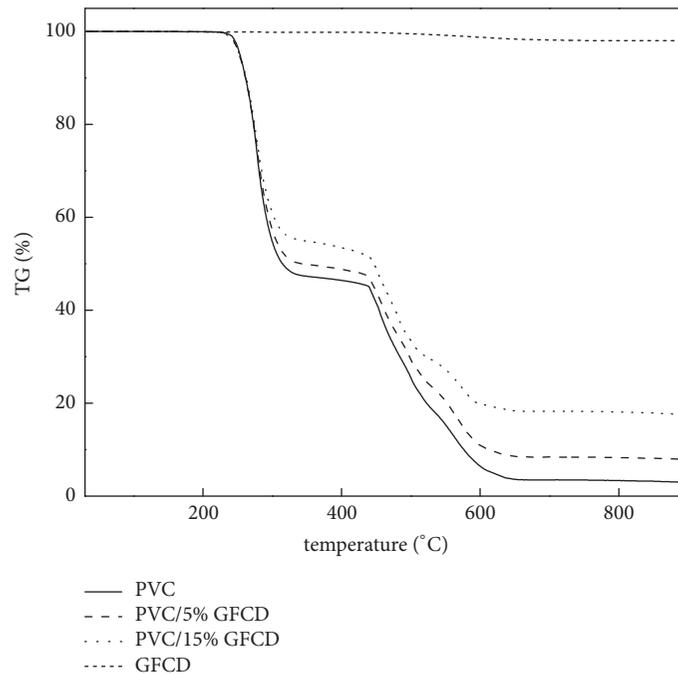


FIGURE 7: Sample thermograms of TGA of PVC, PVC/GFCD composites and filler used.

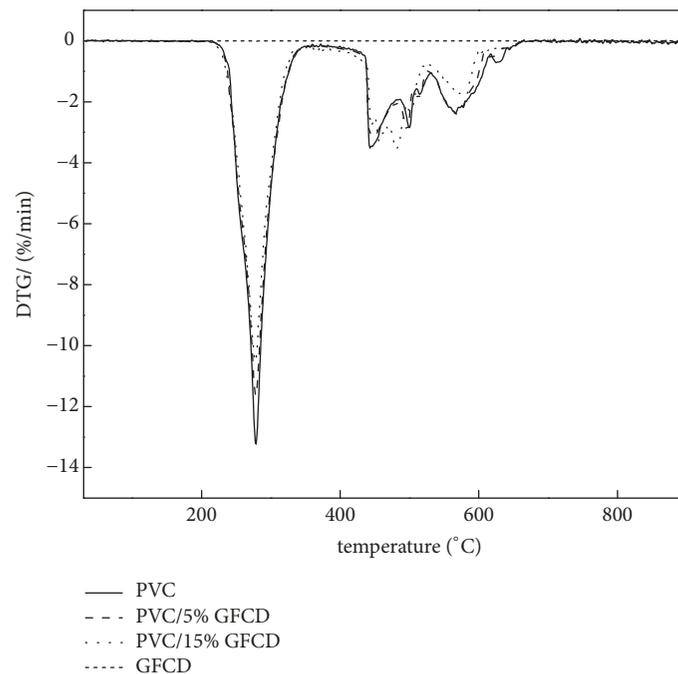


FIGURE 8: Sample thermograms of DTG of PVC, PVC/GFCD composites and filler used.

burnt to form CO_2 [33, 34]. The burning residue determined at 900°C (RM) shows the amount of the remaining noncombustible substance during the TGA tests. The residue after the TGA testing is proportional to the filler percentage in the composite. It consists of inorganic residues after PVC burning and mineral parts of the filler.

Both composites and unmodified PVC demonstrate similar temperature of thermal stability, defined as the

temperature at which a loss of 5% of the original sample weight occurs ($T_{5\%}$). According to the data analysis, an addition of the filler in the tested concentration range does not significantly change the temperature of thermal stability of the material. The maximum DTG peak, which determines the temperature of the most intense PVC decomposition, also occurs at a similar temperature for all analysed materials and differences in its intensity

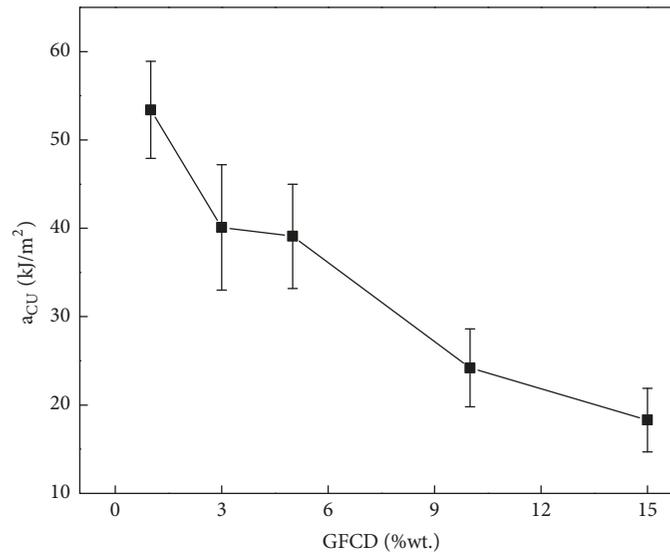


FIGURE 9: The relationship between the composite impact strength a_{CU} and the GFCD content in PVC.

TABLE 1: TGA results of PVC and PVC/GFCD composites.

GFCD content [%]	Temperature of 5wt% mass loss [°C] $T_{5\%}$	Maximum temperature of DTG peak [°C] T_{DTG}	Residual mass in 900°C [%] RM
0	253,6	278,9	2,9
1	252,1	277,6	3,8
3	252,3	278,2	6,1
5	252,6	278,4	7,9
10	252,1	277,6	12,0
15	252,8	277,9	17,5

result only from the amount of polymer matrix in the composite.

An addition of GFCD to PVC causes the reduction of both impact strength and tensile strength (Figures 9 and 10). The specimens of unmodified PVC were not broken in the impact test. PVC/GFCD composites with 1 wt% demonstrated the impact strength of 53 kJ/m², while the increase in filler content of up to 15 wt% caused the impact strength reduction to 18 kJ/m². The reduction in tensile strength is observed only above 5% of GFCD content in the composite. Materials containing 15 wt% filler are characterised by 23% lower value of σ_m compared to PVC with slight changes of ϵ_m .

The method of production of composite materials used in this paper affects the random arrangement of fibres which are also located across the acting forces transmitted through the specimen; it is additionally visible in the cryogenic fractures of the studied materials (Figures 13 and 14), thus strengthening effect is not visible [35, 36]. The reduction of impact strength and tensile strength of PVC composites with fibrous fillers is described in works [32, 37]. However, in most cases, the addition of glass fibres in thermoplasts influences increase in the discussed mechanical properties [38–42]. The

reduction of impact strength and tensile strength may be the result of insufficient adhesion between GFCD and PVC or deterioration of the mechanical characteristics of fibres in the pyrolysis process.

Figure 11 shows the course of change of the modulus E' during bending depending on the temperature. The characteristic values of DMTA analysis were presented in Table 2. A significant increase in the material rigidity was noted along with the increase in the GFCD content in the polymer matrix. At 25°C, the value of E' increased by 37 % with a 15 % share of the filler in relation to PVC. Increased rigidity of composites is also preserved in the glass transition area.

Based on the DMTA analysis, the glass transition temperature values of the tested materials were determined. The value T_g determined from the changes in the value E' and the value $\tan \delta$ (Figure 12) do not depend on the content of GFCD in the polymer matrix. Differences in the values T_g are not more than 1°C and should be considered as insignificant, and their values are close to those describing the unfilled PVC. These results indicate that the filler applied does not cause important changes in the interaction at the molecular level of the polymer matrix.

TABLE 2: Summarising the glass transition temperature values and storage modulus values of the PVC/GFCD composites.

GFCD content (%wt.)	T _g (°C)			E' at 25°C (MPa)
	E' Onset		tan δ	
0	84,9		94,1	3465
1	85,4		94,2	3561
3	85,4		94,3	3738
5	85,4		94,1	3913
10	85,9		95,1	4220
15	85,6		94,7	4735

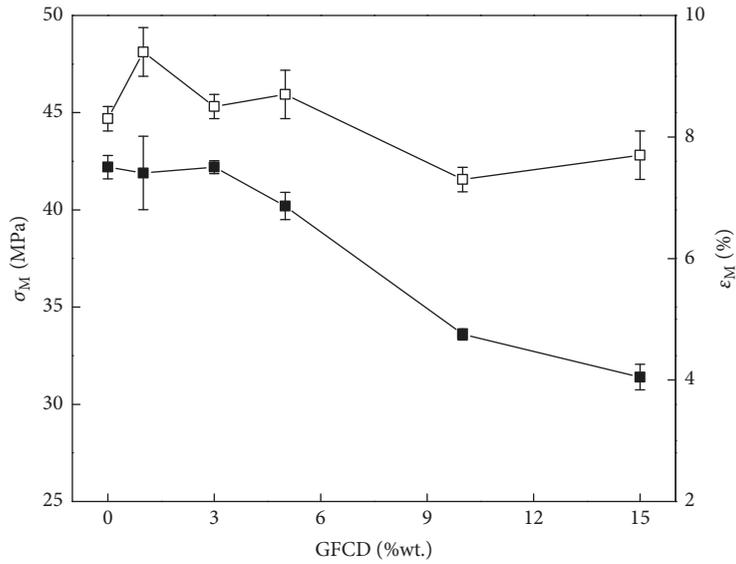


FIGURE 10: Tensile strength σ_M (closed symbols) and elongation at maximum stress ϵ_M (open symbols) versus GFCD content in PVC.

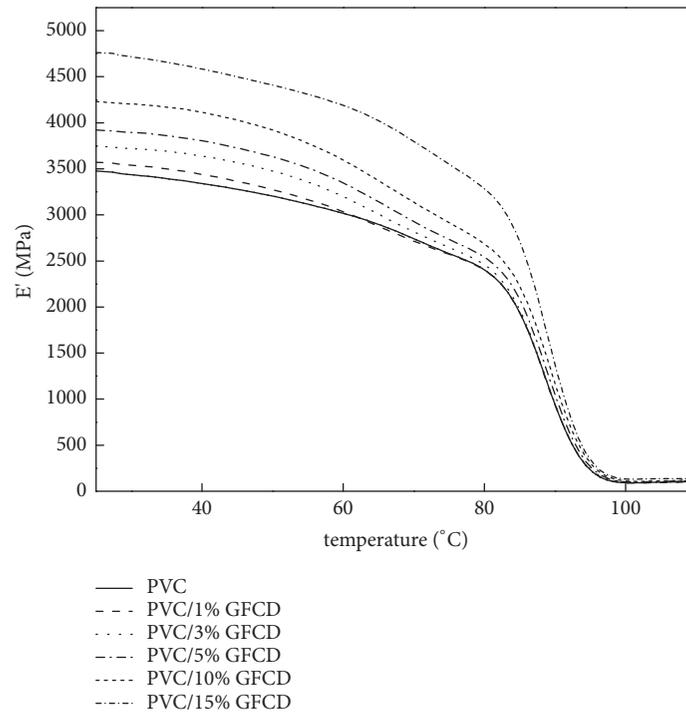


FIGURE 11: The relationship between the storage modulus E' versus the temperature of PVC and PVC/GFCD composites.

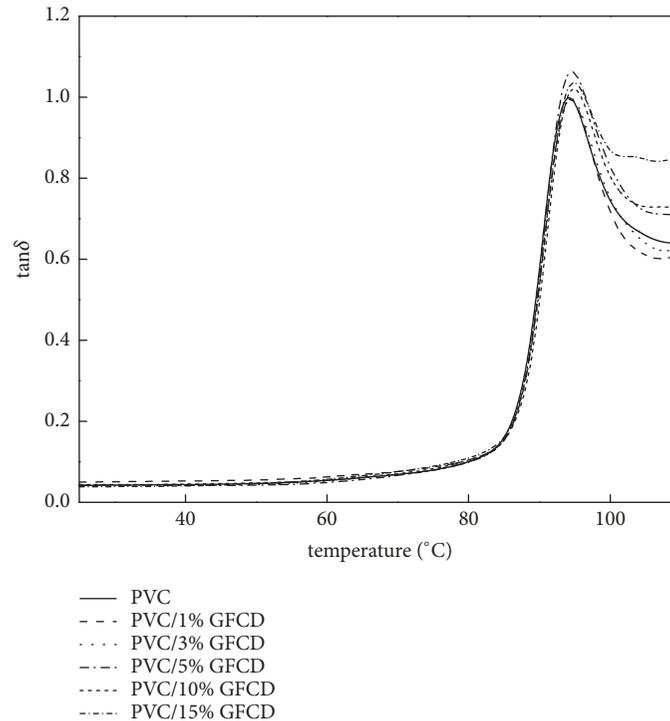


FIGURE 12: The relationship between the loss factor $\tan \delta$ versus temperature of PVC and PVC/GFCD composites.

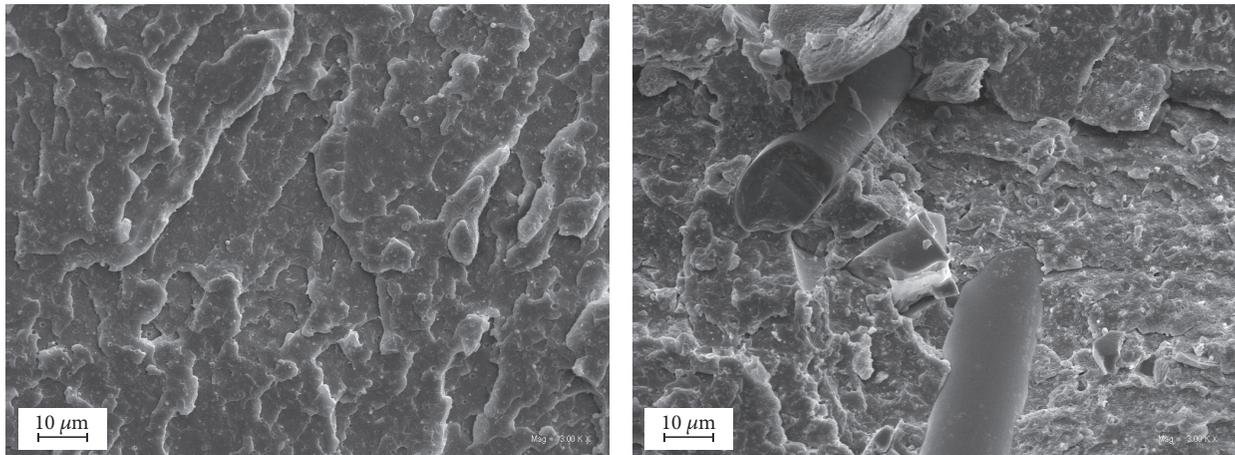


FIGURE 13: SEM images of cryogenic fracture of PVC (left) and PVC/5% GFCD composite (right).

Figures 13 and 14 present examples of SEM images of cryogenic fractures of the studied materials. The fibres in the composite are mostly homogeneously distributed in the polymer matrix; however, the presence of a few clusters of fibres in the composites with a filler content above 10 wt% was observed. Due to the production method of the materials, the arrangement of the fibres does not show orientation; i.e., the fibres are arranged at various angles. There were no clear caverns at the filler-polymer matrix interface. Compared to other thermoplastic composites with glass fibre, no open spaces were found after the elongated fibres, and the broken single GFCD fibres protruded only slightly beyond the plane of the polymer matrix fracture. At the fracture surface, the

remaining fibres, set across the crack boundary, are also observed, although they have been broken.

Based on these observations, it can be concluded that there is a relatively adhesion between PVC and the glass fibre used containing a carbon deposit on its surface [39, 43]. The no visible reinforcement effect with simultaneously a good adhesion confirms the deterioration of the properties of the fibres used, also described in [16, 19].

4. Conclusions

Based on the tests conducted, it was found possible to obtain PVC composites with glass fibres with a carbon deposit,

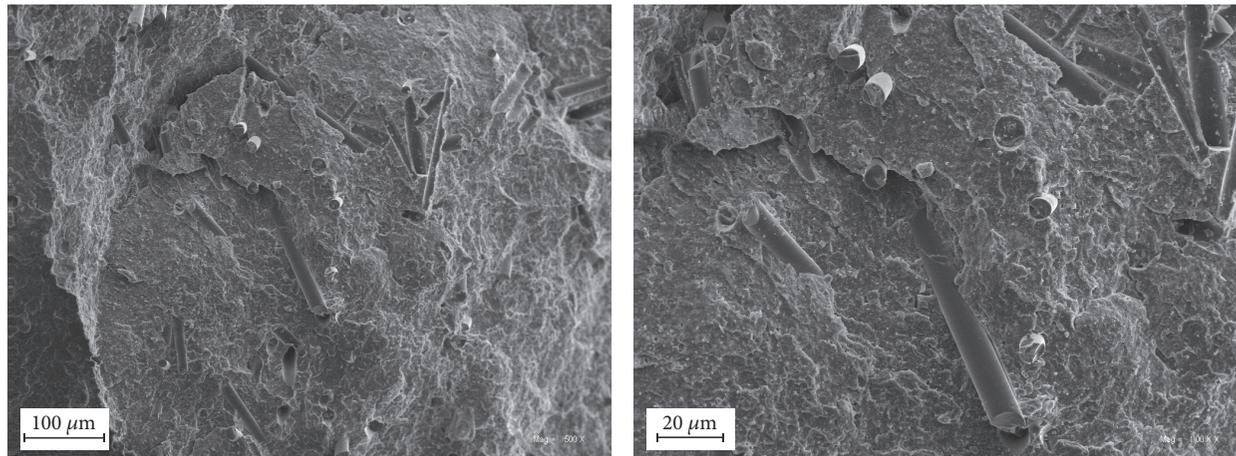


FIGURE 14: SEM image of the cryogenic fracture of a PVC/10% GFCD composite.

which are formed during the pyrolytic recycling of wind turbines.

Obtained composites demonstrate very similar processability compared to PVC and the manufacturing process should not involve difficulties compared to conventional PVC processing. The percentage of GFCD affects neither the temperature of thermal stability nor the time of thermal stability which is extremely important in the case of poly(vinyl chloride) composites. The increase in the percentage of fibres used in PVC causes a clear increase in the modulus of elasticity of the obtained composites while reducing the value of tensile strength and impact strength. This type of change in performance is typical for many thermoplastic composites containing inert fillers. Therefore, the properties obtained do not exclude the application of the proposed PVC/GFCD composites.

In the case of development of pyrolytic recycling of wind turbine blades or other similar products made of chemically cured composites, a large amount of glass fibres will be created which, according to the study presented, can be successfully used to modify poly(vinyl chloride).

Data Availability

Data will be made available on request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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