Thermal Regeneration and Reuse of Carbon and Glass Fibers from Waste Composites

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Abstract

This article aims to develop a method for regenerating and reusing carbon and glass fibers extracted from unrecyclable scraps of carbon plastics, printer parts, and laminating coating. A comparison of known methods of fiber regeneration revealed the advantages of thermal treatment: absence of costs of reagents and complex equipment; better preservation of composition; and strength of fibers. Based on the results of thermographic analysis of wastes in nitrogen and air, the destruction temperatures of their organic matrices were determined (200-460°C), and the use of calcination instead of pyrolysis was justified. The appearance and surface quality of the regenerated fibers are characterized by optical and electron microscopy. It has been established that quantitative extraction of pure carbon and glass fibers from waste crushed to 1 cm is efficient by their calcination at 700 °C for 0.5 h and 500 °C for 1 h, respectively. The principle of creating new composites with the obtained fibers based on the similarity of their composition and binding materials (matrices) has been proposed. It was shown that the introduction of 1 wt% of fibers into slag blocks and active carbon pellets considerably increases their compressive strength, but the bending strength does not change due to dispersed reinforcement. Possible improvement of mechanical properties of products requires reagent treatment of the fiber surface or the introduction of binder additives. Calculations show that the developed method of recycling composite waste can produce 2.3 tons/hour of reinforced building materials that are good for the environment and the economy, excluding expenses for landfill waste disposal and reducing the cost of the product by replacing the primary fiber for the secondary one.

1- Introduction

The world's current glass fiber production is 3–3.5 million tons per year, and the fiber is most widely used to produce composite materials [1]. Polymer composite materials (PCMs), particularly fibrous ones, are actively used in almost all industries, and recycled PCM volumes can reach 1–3% of the annual production. For some PCM waste, reinforced by expensive high-strength and heat-resistant CFs and cheap glass fibers (GFs), waste management is economically and environmentally essential (CFs may cost 15–500\$ per kg, and GFs may cost \$1.1–\$1.4 per kg [1]. The annual global production of composites is estimated to be 65,000 tons of carbon fiber reinforced plastics (CFRPs) and approximately 2 million tons of GFs. The growth of CF consumption is projected to increase by 2.5 times, up to 161,000 tons in 2025 [2].

The compositions of different components, such as glass polymer and carbon polymer, are characterized by a clear interface [3, 4]. CFRPs, thermosetting epoxy composites reinforced with CFs, are usually preferred when higher

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structural characteristics are required. CFRP is extremely durable and lightweight. It is commonly used wherever high strength/weight and rigidity/weight ratios are required, for example, CFRP and aluminum alloy [5], materials for automobiles, aerospace, marine industries, and civil engineering; wind turbines [6], glass/epoxy, carbon/epoxy, and hybrid composites [7]. The hardest requirements are stated in aerospace engineering [8]. Glass fiber reinforced composites comprise the majority of composites (98% by weight [9]). Carbon-reinforced fibers are used much more rarely because of their high performance and price.

The wide range of variations in mechanical properties determines a large area of PCM application [10]: wind turbines – 23%, aerospace and defense – 18%, sports/leisure – 17%, molding and compounds – 12%, civil engineering – 6%, automotive–5%, pressure vessels–5%, oil and gas–2%, and other–12% (2012). The composition of modern PCM can be characterized by the following data [11]: 1/3 of binder materials are thermoplastic and 2/3 – thermosetting; distribution of reinforcing fibers by composition, %: glass – 70, carbon – 12, basalt – 11, aramid – 7.

The growing interest in PCM waste recycling is reflected in publication activity: in 2019, the number of articles on carbon fiber recycling was 110, and glass fiber recycling was 45 [12]. Research on this topic is conducted in three directions: mechanical, chemical, and thermal recycling and their combinations. Coarse crushing of composites is carried out to 50–100 mm in size, while fine crushing is done to 50 μm-10 mm. New composites with recycled materials lose 50-60% of their strength, but with the reduction of the size of fragments, their strength increases. Thermal processing includes three methods: incineration, pyrolysis, and oxidation using a fluidized bed process (FBP) and is usually carried out at 450–700 °C with a yield of fibers of 66 to 77% and loss of tensile strength of up to 34% depending on the atmosphere, duration, and equipment of the process. Chemical separation of fibers from matrixes is made under exposure to reagents (acids, alkalis, zinc, and aluminum chlorides, acetone, and other solvents) under soft (up to 100°C) and rough (above 100°C and increased pressure) conditions with a decrease in mechanical properties of only 4-6% of the initial. There are also alternative approaches to the impact of electric or microwave fields on PCM, which preserve the structure and properties of the fibers.

All waste utilization methods of organic, carbon, and glass-filled plastics, focused on the recovery of the reinforcing agent (fiber), are united by the need to destroy the matrix (binder) and accompanied by the formation of various processing products. Compared to the possible methods, it shows that its recycling method is preferred for maximum retention of each filler property.

Implementation of recycling method for PCM wastes should provide less overall environmental impacts: energy spent and CO_2 emission. For example, Witik et al. (2013) report that pyrolysis for recovering CFs from composite wastes consumes $\approx$ 5%-10% of energy to produce virgin CFs [13]. Avoiding higher energy costs in traditional primary processes will reduce gas emissions because the energy comes mainly from fossil fuels. Thus, recycling leads to significant environmental enhancement [14, 15]. In addition, unutilized waste and end-of-life materials of production and consumption can serve as secondary sources for another industry, thus reducing industrial environmental pollution [16]. Recycled CF can also be produced with impressive energy savings (98%) and lower by 70% cost [17].

For example, various aircraft materials and devices include carbon and fiberglass composites. It is reported that approximately 85% of an airplane's mass (about 100tons) can be recycled as useful materials after the aircraft is dismantled [17, 18].

Within the material life cycle concept, such authors as Marsh (2009) or Tapper et al. (2020) note that the predominant methods of carbon plastic waste handling are landfill disposal, incineration, and, more recently, processing (fiber mechanical grinding or regeneration methods). However, landfill disposal and incinerating of these wastes destroy their resource potential and carry known environmental risks [19, 20].

The existing methods of composite waste utilization can be divided into three areas: physical, chemical, and thermal. Among physical methods, mechanical methods are the most common. Their main advantages are the simplicity of industrial implementation, universality (applicable for any PCM and polymers and allows for simultaneous processing of fibers and polymeric binders), and the absence of harmful emissions and fumes. On the other hand, their disadvantages include high power consumption spent, homogeneity of the size of ground materials, lower strength of the recycled reinforced plastics, and limited demand for the processed materials [21].

The review of Oliveux et al. (2015) is devoted to the consideration of fibers extracted from the waste and reused for the creation of new PCM and rational methods of their regeneration [22]. Glass fibers recovered by thermolysis or solvolysis can replace only a small part of the initial ones in the products with preservation of environmental and economic characteristics. The carbon fibers from highly loaded composites cannot be used for their intended purpose because of partial loss of properties and can be used only in less responsible materials. Furthermore, the mechanical properties of new composites differ from initial ones due to the characteristics of incorporated fibers changed during processing.

The readiness of different technologies for recycling fibrous waste to the implementation can be estimated by Technology Readiness Level (TRL) in points from 0 to 10: landfill disposal or incineration – 9, pyrolysis for CF and crushing for GF – 8, crushing for CF and pyrolysis for GF – 7, fluidized bed pyrolysis and solvolysis – 4, microwave treatment – 3 [11].

Energy demand, an important environmental indicator, depends on the composite recycling method. Its approximate values are: chemical recycling 21-91 MJ/kg; pyrolysis 24-30 MJ/kg; microwave treatment 5-10 MJ/kg; mechanical recycling 0.1-4.8 MJ/kg [23].

According to Bernatas et al. (2021) the problem of PCM waste utilization (in France, the volume of production waste is up to 15,000 t/year, operational waste – 7,000 t/year) is considered in the context of legislative incentives and logistic constraints [24]. In analyzing the array of studies, it was found that in the regeneration of CF, the priority belongs to pyrolysis, GF – mechanical grinding, waste and contaminated materials – oxidation in a fluidized bed. Furthermore, particular attention is paid to the possibilities of mechanical processing of thermoplastic-based composites, whose share in the market is gradually increasing.

During mechanical crushing of PCM waste, their interaction with new matrices (ethylene, copolymer of polypropylene, methacrylic acid, acrylic-butadiene styrene, polystyrene foam) is insufficient [25], resulting in a reduction in ultimate tensile strength by 65%, in bending strength – by 85%. The primary mechanism of their destruction is the pull-out of fibers from the matrix. Thermal and thermochemical processing provides better preservation of properties, but in this case, also modification of fibers with organic compounds is required to improve adhesion.

A representative review of PCM waste management technologies was performed by specialists at the All-Russian Research Institute of Aviation Materials (VIAM) [26, 27]. Total Russian production of the PCM since 2020 was estimated at 118,000 tons per year. As the authors note, recycling is the primary way to solve this problem. Its undoubted advantage is creating an additional number of useful products for various industries and the absence of environmental recontamination. The paper of Protsenko et al. (2020) presents data on the processing of fiberglass reinforcing agents [28]. Thermolysis of GRP based on epoxy vinyl ester binder was carried out at 400 °C for 30 minutes. A PCM sample with such obtained reinforcing fibers demonstrates only 1.6% lower bending strength than the primary composite. According to Khurshid et al. (2020) waste carbon and polyamide fibers can be incorporated into hybrid ribbons and yarns with appropriate stretching machine settings to preserve fabric quality [29].

In Pietroluongo et al. (2020), the effects of mechanical processing on automotive waste composites based on polyamide PA66 reinforced with fiberglass have been studied. The material was pelletized and molded three times and compared with the primary material used to produce the same vehicle component. The results showed that mechanical processing causes fibers' breaking and thus reduces recycled fiber contribution to the strength of the secondary composite. However, despite the material's degradation, it retains mechanical characteristics acceptable for other automotive applications [30].

There are studies on PCM utilization using chemical methods based on depolymerization (chemical destruction) of a polymeric binder, aimed to obtain pure and non-damaged fibers by near critical and supercritical water [31], supercritical alcohol [32] or thermo-promoted chemical decomposition [33]. In this regard, the main promising methods are thermocatalysis, solvolysis, and fluidized bed oxidation. In this case, solvolysis is thermal catalysis and differs from matrix depolymerization in solvolysis; various fluids (supercritical water and alcohols) with catalysts in alkali salts have been used as a medium. However, in the case of thermo-catalysis, any other media are used. The advantages of thermocatalytic methods are reinforcing agent property retention level of 90-98% [34], high process selectivity to polymer binders, and obtaining regenerated binders [35]. On the other hand, the main disadvantages of the described waste PCM processing method are high temperature and pressure conditions (up to 27MPa), treatment with harmful reagents, and utilization of polymer matrix decomposition products.

Methanol, ethanol, 1-propanol, and acetone with alkali catalysts were successfully employed as solvent reagents to regenerate the CFs at temperatures ranging from 200 to 450°C [34]. The most widespread processing of carbon plastics using solvolysis is realized in Japan: Hitachi Chemical Co. Ltd. uses tripotassium phosphate as a catalyst and benzyl alcohol as a solvent at low pressure (up to ~2 atm) and temperature of 200°C for 10 h [35]. Some results of thermochemical recycling of epoxy-based aeronautical composites using a solvolysis process in water are described by Zhang et al. (2021) [36]. The final products were fiber and depolymerized epoxy binder. The advantages of this method are the comparative simplicity of instrumentation, energy efficiency, and the ability to use epoxy binder decomposition products in epoxy resin resynthesis.

A low-temperature catalytic recycling process was studied in [37] to recycle plastics and aircraft composites. The authors discovered that catalytic conversion transforms various binders (rubber, thermosetting and thermoplastic materials) into flammable hydrocarbons. In addition, catalytic conversion does not pollute the environment.

Because of its thermosetting nature, epoxy composite has some disadvantages: once the material has been cured, it cannot be resoldered, modified, or dissolved; therefore, recycling or repairing is very difficult. Also, the processes for producing related components are mostly limited to small average volume production due to the relatively long curing time required for the chemical formation of the polymer network.

According to Yu et al. (2016), a chemical recycling method uses composite waste based on epoxy resin and CF (CFRP) and ethylene glycol when heated [38]. It was proved that mechanical parameters of either recycled or recovered PCMs are close to those of the primary materials.

The application of critical water and supercritical acetone for recycling waste carbon and glass plastics is described by Sokoli et al. (2019) [39]. The completeness of matrix decomposition and fiber quality were a function of temperature (T = 260-300ºC), pressure (p = 60-300 bar) and the composite/solvent ratio (0.29-2.1 g/ml). Under optimal conditions, supercritical acetone provided almost complete dissolution of the resin, and the separated glass fibers had tensile strength equal to 89% of the initial one. Processing carbon plastics with water or acetone made it possible to retain the strength of separated fibers at the initial level.

The article of Shaydurova et al. (2018) shows the research results of CFRP utilization by solvolysis, with the reinforcing agent recovered from the total matrix volume [40]. The recovered agent can be further reused in PCM production. Nitric, phosphoric, and sulfuric acids, 50% V/V, and their mixtures with chromium (VI) compounds were tested as chemical solvents when samples were heated to 60–110°C for 8 hours. Treatment with chromium mixtures resulted in the gradual destruction of the binder with a mass loss of up to 52% and the recovery of visually unspoiled CFs. However, the proposed solution requires toxic chromium compounds and acids. Epoxy resin deletion from a unidirectional CF–reinforced laminate was also achieved using electrical treatment [41].

PCM thermal utilization includes three methods, dependent upon the oxygen content: 1) incineration when the oxygen content is close to or higher than the stoichiometric value; 2) gasification in a lack of oxygen; and 3) pyrolysis in the absence of oxygen. PCM incineration is the known process of waste elimination. However, this method is impractical because the only product that can be used in this process is heat. In addition, incineration leads to environmental pollution (air and water basins), so this utilization method is forbidden in many European Union countries. The gasification method is a PCM decomposition process to produce synthesis gas, which is used to produce heat and electric power. The disadvantage of incineration and gasification is the destruction of the most valuable PCM components and the high probability of emissions.

The most common utilization method for reinforced plastics is pyrolysis [42]. Pyrolysis can produce recycled CF as a high-value secondary material on an industrial scale (over 1,000 tons per year), and as reported by Tapper et al., the process has much lower energy consumption than virgin CF production [20]. Even though pyrolysis has much higher process energy costs and emissions, it is an excellent alternative to landfill and incineration because the recovered fiber can be reused, reducing the overall life-cycle energy consumption. It is only possible to regenerate fibers by pyrolysis from plastics with heat-resistant fillers, such as GFs and some grades of CFs. In the case of CFRPs, it is necessary to select pyrolysis process conditions because the properties of CFs can decrease by 30-40% if the process is not optimized.

A review article by Naqvi et al. is devoted to the pyrolytic processing of high-value composite materials [43]. The process produces coke and fibers, pyrolysis oil (the main components are benzene, toluene, ethylbenzene, p-xylene, and styrene), and gas – a mixture of H_2, CH_4, CO, and CO_2. It is noted that the pyrocarbon contaminated surface of the fibers creates difficulties for their reuse, so their oxidation under controlled conditions is conducted. The formation of carbonyl and carboxyl groups on the surface of the fibers is observed, violating their interphase binding with the secondary composite matrix. The reduction of tensile strength of pyrolyzed fibers reaches only 5-10 % against 18-50 % when firing. The prospect of creating new composites with regenerated fibers requires developing the methods of their directed molding, ordering, and others. Data presented show that a short hot sodium hydroxide solution treatment of thermally recycled fibers can increase their strength three times and restore their ability to act as an effective reinforcement in second life composite materials [44].

Yang et al. (2015) subjected mats with glass E-fibers to annealing in an oven at 500°C for 30 min [45]. To compensate for the loss of mechanical strength, they were further treated with a 1% solution of HF for 10 min to remove surface impurities (etching) and a 1% solution of ammonium persulfate to hydroxylate the surface. Although the tensile strength of the modified fibers is inferior to that of the original (~220 MPa); however, it is possible to restore it by 50-70% using chemical treatment, and the same pattern is observed for composites with their use.

According to Onwudili et al. (2016), CF and GF separated by pyrolysis at 500°C were used to produce secondary material with low-density polyethylene (LDPE) matrix (ultimate tensile and bending strengths of 11.4 and 7.5 MPa, respectively) [46]. Pre-separated fibers were oxidized under the same conditions to remove matrix residues and treated with reagents of 3-aminopropyl-trimethoxsilane and maleic anhydride grafted high-density polyethylene (HDPE) and two experimental additives. The authors found that oxidized fibers of both types provided composites with a slight

increase in strength, while their combination with modifiers increased the ultimate tensile strength by 6-124%. The higher bending strength of the materials was also due to the oxidation of the used fibers.

Conventional PCM thermal decomposition causes significant damage to CFs. Searching for better processing methods, Matsuda et al. studied the combined annealing (air oxidation) and electrical treatment to recover the resin from a multilayer CFRP molded from a unidirectional prepreg (CF that has been pre-impregnated with epoxy resin) [47]. Annealing followed by electrical treatment in a solution showed that resin separation without CF damage occurred at a temperature (up to 450°C) close to the decomposition range of epoxy resin. However, component separation caused significant damage to the fibers for un-annealed CFRP samples. There is a Russian patent on technology development for utilizing carbon and organic plastics by pyrolysis and a pilot plant construction [48]. However, its work results were not commercialized, and the project's future is unknown.

The advantages of pyrolysis include a high fiber yield at the optimized process, heat recovery from the polymer binder decomposition, equipment universality, good adhesion of epoxy binder to recycled fibers of reinforced plastic, and sample opportunities for commercial applications. The process' disadvantages include the uneven heating of the reactor's operating space and, consequently, possible incomplete binder decomposition and the need to deactivate the pyrolysis gases that contain hazardous compounds. Secondary CFs recovered by thermal regeneration [49] were used for the dispersed reinforcement of composites based on alkali-activated granulated blast furnace slag. The results obtained demonstrate the addition of secondary CFs in the amount of 0.7% vol. In addition, changes in the composite fracture pattern result in a decrease in brittleness and an increase in the composite bending strength by 35% and compression strength by 20%.

The literature review reveals a high level of research interest in regenerating carbon and glass fibers by thermal methods. Pyrolysis is considered more "gentle," but it is often accompanied by calcination of obtained fibers to release them from coke residues. A balance between the purity of the regenerated fibers and preservation of their properties is necessary, which requires thermographic analysis of new types of waste and their heat treatment mode selection. In addition, elemental analysis of calcined fibers, rarely given in the literature, will make it possible to estimate the transformation of the fiber surface during their calcination that can contribute to the chemical interaction between them and the new matrix. The reinforcement results depend primarily on the strength of this interaction, expressed in the type of failure under load studied by scanning electron microscopy. Traditionally, secondary glass fibers are combined with cheap thermoplastic binders that do not provide maximum adhesion between the components. Incorporating fibers into the matrix of similar composition and heat treatment of the obtained composites should contribute to their effective reinforcement. This hypothesis proposed by the authors forms the novelty of the present work and needs to be verified.

In addition, most of the works are devoted to regenerating relatively long fibers that retain their shape and allow the measurement of mechanical properties. However, many construction fiberglass plastics contain finely dispersed fibers (lengths about 10-100 μm). If evenly distributed in the new matrix, such fibers can provide composite isotropic mechanical properties essential in the building materials first prepared and tested in the present work. In addition, the tensile strength determined in the articles does not allow us to judge the quality of building materials with their use, for which it is necessary to measure the compression and bending strength.

Thus, the thermal regeneration of reinforcing fibers from PCM wastes represents a promising method for their recovery, and its development for non-utilized today industrial waste composites is the present work's purpose. The exploration tasks are an investigation of thermal behavior of the named composites and determination of heat treatment mode, evaluation of the mechanical properties of the regenerated CFs and GFs via tests of composite materials reinforced by them, and ecological-economic assessment of the proposed technology.

2- Experimental

2-1- Materials

This study considers three types of PCM waste, presented in Figure 1: 1) Wastes of aviation CFRP production with notations U-1,3,5 consisting of unidirectional, woven, and uniform strength carbon fabrics, respectively, and epoxy resin (production of the Russian Institution VIAM); 2) Laminate, waste from the manufacture of sanitary products, consisting of %: ER13-2400-180-13 fiberglass roving (E-type), polyester resin 37.1, micro calcite 49.4, and titanium dioxide 0.5; 3) Heat-resistant printer parts – composites of GF (25-30%) and PET plastic.

2-2- Sample Preparation

All the materials were crushed to particles of up to 1cm in size before further operations. Then, samples for thermogravimetry were transformed into powder.

Figure 1. PCM waste under study: CFRP – scraps of aviation carbon fiber reinforced plastic, office equipment – heat-resistant PET-GF composite printer parts, laminate - composite for bath reinforcement: fiberglass + micro calcite + epoxy resin

2-3- Measurements

Thermographic analysis of the studied materials was carried out on a MOM-1500 derivatograph with computer signal processing by EcoChrome software. Powdered samples of up to 500 mg were heated at 9°C/min to ~900°C under nitrogen or air. The device's mass and temperature measurement errors were 2.5 mg and 10°C, respectively. The measurements were carried out once for each sample. Calcinations of materials carried out thermal regeneration of fibers in porcelain crucibles in an electric furnace with a controlled regime (heating rate 10°C/min, final temperature 500–900°C, and holding within 0-1 h). The solid product yield was calculated as the ratio of its mass to the initial material mass. These experiments were carried out twice. The yield values are average, with a relative error of 5.7wt% (standard deviation method). The properties of the recovered fibers were defined on the equipment of the D. Mendeleev University Joint Research Centre (Russia). Micrographs of secondary fibers and reinforced pellets were taken using DigiMicroLCD optical microscope and JSM6510LV (JEOL) scanning electron microscope (SEM); their elemental composition was estimated by X-ray fluorescence method on the attachment to the microscope (3 times at different points of a single sample).

Strength parameters of virgin fibers are known from the literature: tensile strength 3.5-7.0 GPa for studied CF and 1.4-2.6 GPa for studied GF [1]. Determination of mechanical properties of recovered fibers was impossible because of their short length (15 μm to several mm), so indirect assessment was implied. Activated carbon granules used to test thermally recovered carbon fibers were prepared in the laboratory by chemical activation of a mixture of organic plastic waste and charcoal tar [50]. The 0.5-1.5% of named fibers were added to the mixture for reinforcement and then heated to 650°C. Silica-slag-based blocks were reinforced with 1-5 wt. % of thermally recovered glass fibers prepared according to [49]. Uniaxial compressive strength and bending strength (at least 6 granules of the sample with each addition) were measured by high-precision universal test machine Autograph AGS-X (SHIMADZU) running at a displacement rate of 1mm/min according to the standard Russian GOST10180-2012.The whole scheme demonstrating the methodology of the present study is shown in Figure 2.
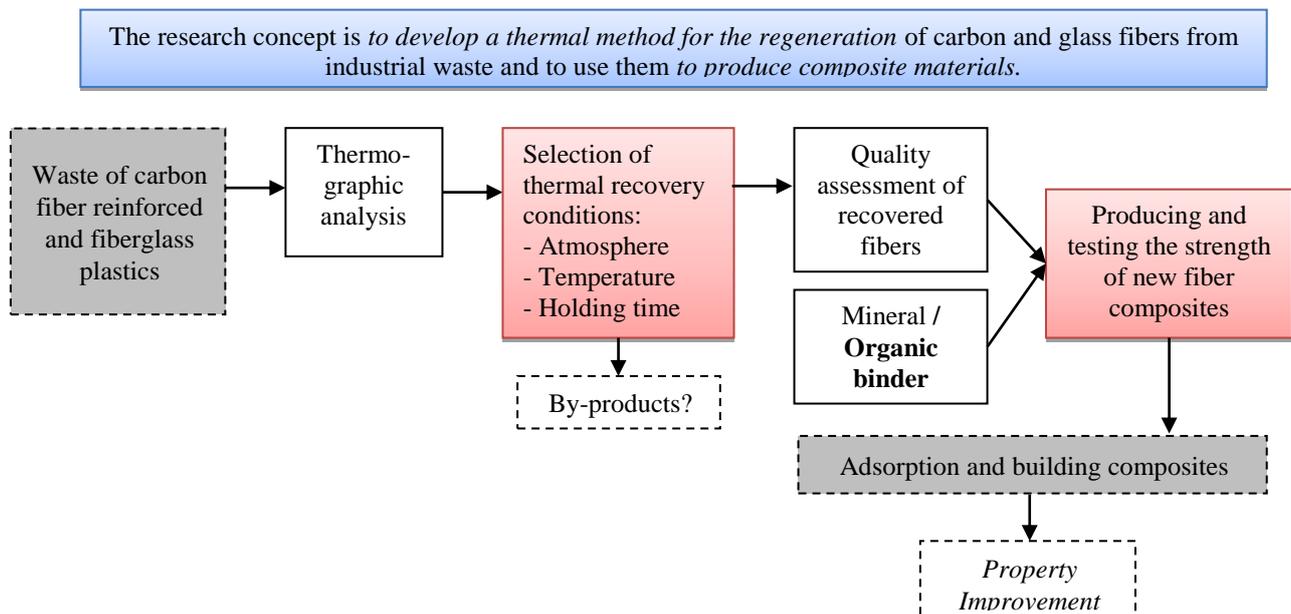

Figure 2. Flowchart to explain the study methodology

3- Results and Discussion

3-1- Thermal Decomposition of Composites

Selection of rational conditions for thermal regeneration of fibers requires studying the process of thermal decomposition (pyrolysis or oxidation in nitrogen and air atmospheres, respectively) of waste containing them. Figure 3 presents a thermographic analysis of the decomposition of the studied waste materials under nitrogen and air when heated up to 900°C.
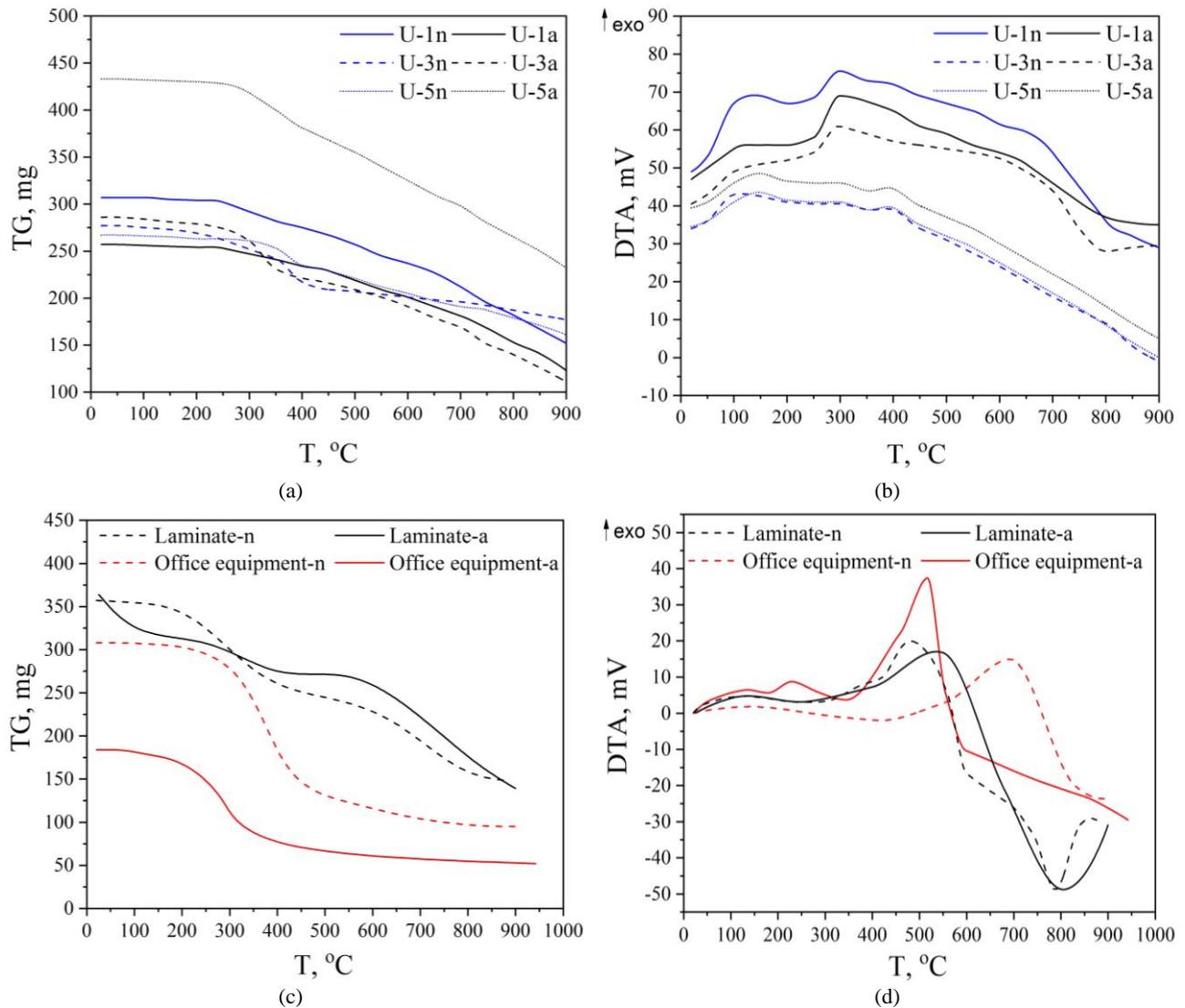

Figure 3. Results of thermographic analyses of waste PCMs: a, c–mass change curves (T-G), b, d–temperature derivative curves (DTA) of PCM samples (a – air, n - nitrogen)

The following is the overall picture of the studied CFRPs thermal decomposition: when heated to ~300°C, their degree of destruction does not exceed 9 wt%, and the direct loss of mass occurs in the range of ~300°C–410°C when a remarkable slope of TG curves is observed. Depending on the resin type, the processes occur between 450 and 700°C depending on the resin type, while carbon fibers do not show mass loss up to 600°C [22]. The same TGA conditions for CFRP [34] showed only one weight loss peak around 400–475°C and an average solid yield of 39.3 wt.%. However, binder decomposition is continued at higher temperatures (Figure 3-a) up to 900 °C without clear peaks. These differences appear to be due to the destruction of the residual epoxy binder of the carbon plastics.

There is no noticeable medium effect on the material decomposition up to ~500°C, except for sample U-3. Upon further heating, air oxygen becomes an oxidizing agent and accelerates the loss of mass of the samples, and the oxidation also affects the carbon fibers themselves. The yields of pyrolysis residues of samples in nitrogen at 900°C are relatively high: 18%–52%, and oxidation in the air gives 0.3%–34%. The heat treatment products look like individual black fibers without any binder residue.

The DTA curves of sample U-1 show an extended exothermic effect of polyester resin decomposition without any significant differences in both atmospheres. The conversion of U-3 in nitrogen over the entire temperature range is exothermic, with weakly expressed peaks at ~100°C and ~400°C (Figure 3-b). In the air, it also has an anexo-effect with a clear peak at ~310°C, which terminates at ~800°C. Weak exothermic effects were found in nitrogen (~150°C and 390°C) despite usually fixed endo-effects for breaking chemical bonds, and a more pronunciation done in the air accompanied the mass loss peaks of U-5 associated with the binder oxidation at ~300–360°C(Figure 3-b).

The mass loss curves of laminate (Figure 3-c) have two decomposition regions: ~200–420°C (bonding resin decomposition) and ~550–820°C, explained by fiberglass or micro calcite transformation. They are close in both atmospheres, but oxidation promotes the first stage. At the same time, the office equipment has one region associated with the PET matrix decomposition at 300–460°C in nitrogen and180–340°C in the air, which is in good accordance with theoretical PET destruction. The solid product yields of heat treatment at 900°C were 42% for laminate (including11% of fiberglass and 31% of $CaCO_3$), and they were 27.5%–30.5% for office equipment (molten glass fibers). Interestingly, the first stage of laminate decomposition in both atmospheres is accompanied by heat emission, which is accompanied by heat absorption (Figure 3-d). The decomposition of waste office equipment shows high exothermic effects with peaks at 500°C in air and 670°C in nitrogen. These results could be compared with thermal degradation of epoxy-vinyl-ester binder [28]: intensive combustion at 340-445°C and complete mass loss at 523.5°C also accompanied by heat emission.

The important fact that the GRP mass loss curves and residue yields are close in nitrogen and air (Figure 3c) makes it possible to calcine GRP without losing GFs. At the same time, temperatures of matrix destruction in the air are lower than those in nitrogen, as oxidation promotes the process [51]. Therefore, annealing of waste PCM laminate [47] at temperatures up to 450°C followed by electrical treatment was also recommended for careful utilization. Based on thermography, the recommended temperature of the powdered CFRP and GRP samples required to destroy their organic matrixes is 410–460°C.

3-2- Pyrolysis or Calcination?

Both heat treatment methods aim to destroy organic matrix and release reinforcing fibers, whose mechanical properties usually decrease sharply at temperatures above 500°C. Depending on the atmosphere and duration [12], PCM waste pyrolysis at 400-600°C preserves the tensile strength of fibers from 50 to 95% of the initial one. However, the surface of fibers is often covered with residual pyrocarbon and requires additional calcination in air at the same temperature [25, 45]. However, the surface of the fibers is often covered with pyrocarbon residues and requires additional calcination in air at the same temperature [25, 45]. According to Matsuda et al., oxidation in air at 450°C provides separation of epoxy resin and undamaged CFs [47]. As shown above, this process during calcination proceeds at a lower temperature ("soft" conditions); therefore, it is accompanied by less loss of fiber strength, requires less energy (see exo-effects in Figures 3-b and 3-d), and does not form toxic volatile products. For these reasons, calcination was chosen as a method of fiber regeneration from the studied PCM.

As for the mechanical properties of heat-treated fibers, they retain 90-95% of the initial ones at optimized conditions of pyrolysis and oxidation [25]. Based on the literature data and preliminary experiments (Figure 4), it is shown that annealing in the air, in contrast to pyrolysis in an inert atmosphere, provides the separation of fibers with a clean surface. Therefore, it is necessary for their further successful incorporation into matrices of composite materials.

Figure 4. Micrograph (×1000) of fully calcined CFs

3-3- Characteristics of Recovered Fibers

Temperature and time of calcination are the most important factors determining oxidation processes; hence, the quantity and quality of the fibers separated from the waste. The thermography data (Figure 3 a and c) shows that the minimum temperature for matrix destruction of carbon plastics is 410°C, glass plastics – 340/420°C. However, multiple sources usually recommend a heat treatment temperature of waste PCM of 500-700°C [42-46].

In practice, the speed and degree of decomposition of composite materials also depend on the size of their fragments. Since the recyclable waste is 0.5-1 cm in size, the process requires a higher temperature or time than the TGA data. It was found experimentally that at temperatures less than 500°C, oxidation of binders was slow (waste fragments retained a solid structure after 1 hour of heating). In laboratory conditions, the crushed wastes were calcined in air at a heating rate of 10°C per min with varying temperatures and holding times (Tables 1 and 2). The appearance of carbon and glass fibers regenerated by calcinations is shown in Figure 5.

Table 1. CFRP calcination product yields

Sample	U-1			U-3			U-5		
Temperature, ℃ (processing time 0.5h)	500	700	900	500	700	900	500	700	900
Yield, % of sample mass	75. 2	58. 3	1. 2	61. 6	49. 0	16. 2	82. 7	70. 0	34. 1

Table 2. GRP calcination product yields

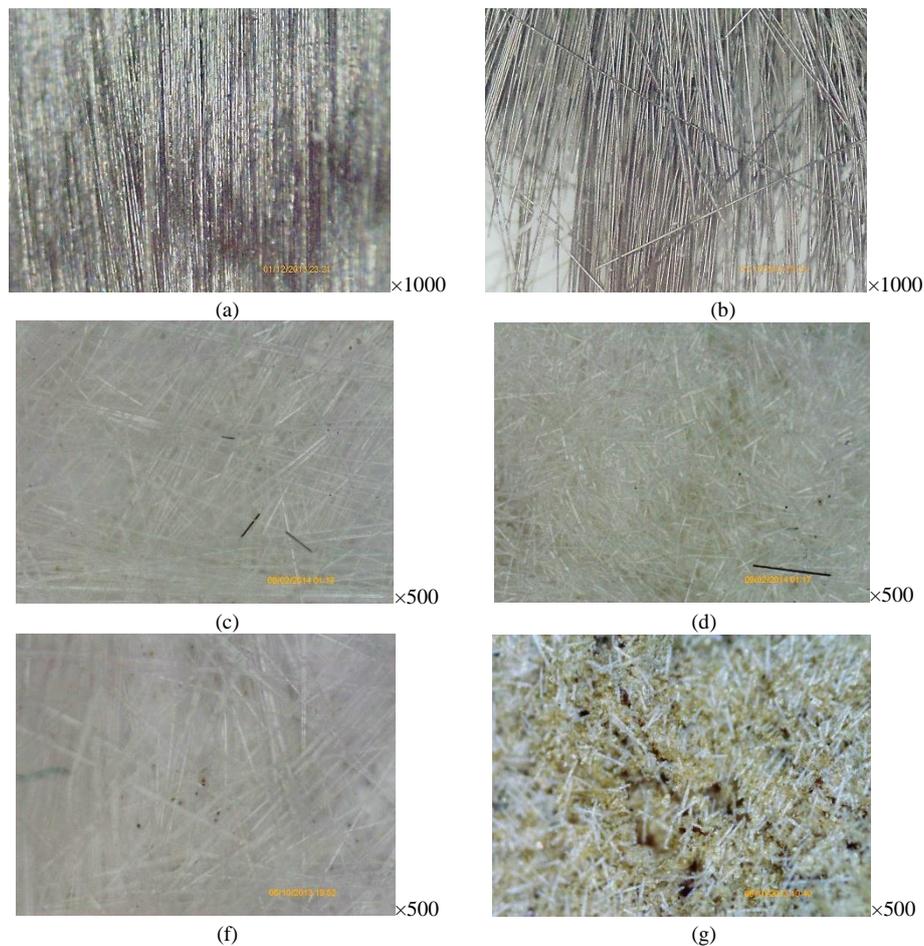| Sample	Laminate				Office equipment			
Temperature, ℃	500	500	700	700	500	500	700	700
Processing time, h	0	1	0	0. 5	0	1	0	0. 5
Yield, % of sample mass	71. 7	66. 8	65. 9	66. 5	61. 2	32. 0	30. 5	21. 8

Figure 5. Micrographs of thermally recovered CFs from U-3 (a–500°C, b–700°C) and glass fibers (d–laminate, 500°C; e – office equipment, 500°C; f–laminate, 700°C; g – office equipment, 700°C)

It is established that CFRPs samples need a temperature of 700°C and a minimal oxidation time of 30 min to obtain separate and not damaged fibers (Figures 5-a and 5-b). Higher temperature and retention prolonged for 1 h lead to a sharp decrease in the target product yield (Table 1). The appearance (Figures 5-d and 4-e) of fibers from GRPs already at 500°C is similar to their initial form, so this temperature is proper to destruct the organic matrix. The influence of holding time is also important: for printer composite parts, it decreases the product yield at both temperatures (Table 2). However, a 1-hour holding is required to release the fibers as their yield of 32 wt% is close to the initial content. That is following Oliveux et al. (2015): in oxidant conditions, epoxy resins are more easily degraded than in inert conditions, and at temperatures in the range of 500–600°Cit is possible to remove resin residues completely [22].

In the product obtained from carbon polymer at 500°C, CFs are still bound by polyester resin residues, while at 700°C, they are already fully separated. Carbon fibers are rather resistant to temperature; for example, treatment at 550°C, firstly in nitrogen and then in the air, preserved the tensile strength of secondary CFs at a level of 95% [51]. The results of the recovery of GFs at 500°C are satisfactory; individual transparent glass filaments are seen in the photos. Treatment at 700°C does not visually influence GFs from laminate but causes a partial transformation from printer parts into ash (Figure 5-g).

SEM method was also applied to evaluate the structure and size of recovered GFs and CFs (Figures 5 and 6). Glass ones are smooth, nonporous with 12–13 μm diameter, over 100 μm length for office equipment, and several mm length for laminate. It should be noted that they contain some residues on the surface when heated to 500°C, which could serve as points of adhesion at the introduction of fibers into mineral matrices. On the contrary, the evident difference for the carbon fibers is observed: only 700°C treatment provides their separation.
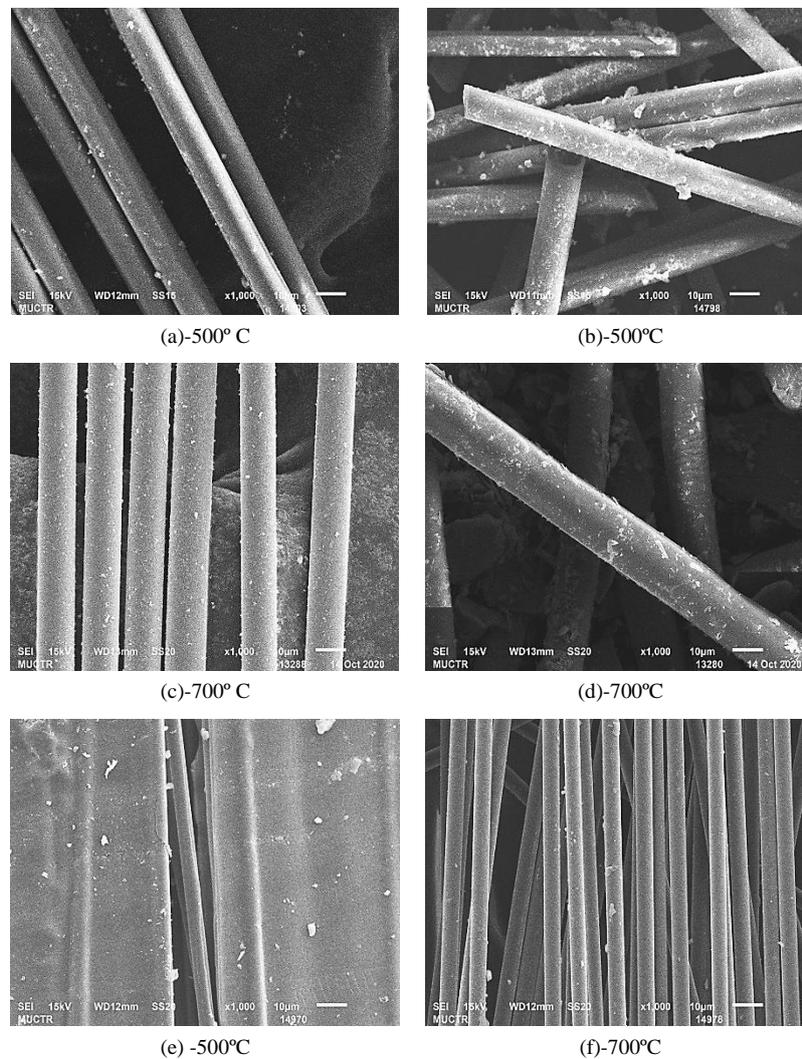

(a)-500° C   (b)-500°C

(c)-700° C   (d)-700°C

(e) -500°C   (f)-700°C

Figure 6. SEM photos (×1000) of thermally recovered glass fibers (a, c – laminate; b, d–office equipment) and carbon fibers (e, f – sample U-3)

The found temperature intervals of composite waste treatment are also recommended in the literature, ranging from 450 to 700°C [12]. The specific temperature of the process is determined by the surface cleanliness and preservation of the mechanical properties of fibers.

A possible change in the surface composition of the regenerated fibers during oxidation is essential for their interaction with new matrices [43]. Data of the X-ray fluorescence analysis of the surface of the recovered fibers are shown in Table 3. They show the elemental composition of the material surface, excluding hydrogen.

Table 3. X-ray fluorescence analysis of thermally recovered fibers

Element	Content, wt%				
	U-1	U-3	U-5	Laminate 500/700°C	Office equipment 500/700°C
C	86. 6	81. 9	83. 5	5. 3/34. 2	11. 7/13. 8
O	12. 5	18. 1	14. 8	56. 7/39. 0	45. 0/48. 4
Si	-	-	-	21. 7/14. 3	16. 6/15. 0
Ca	-	-	-	9. 7/8. 11	10. 3/6. 19
Al	-	-	-	5. 8/3. 68	9. 7/3. 97
Mg	-	-	-	0. 51/0. 31	-/-
Others	0. 9	-	1. 7	0. 28/0. 40	0. 27/12. 6

According to Table 3, thermally recovered CFs contain carbon (>81.9%) predominantly, whereas GFs contain oxygen compounds of several mineral elements. It is known that initial CFs should contain only carbon and E-type GFs consist of Si, Al, Ca, Mg, Na, and K. So, the thermally treated materials are enriched with oxygen, which points to their partial oxidation (in the form of carbonil- and carboxy- groups [46]) and usually decreases mechanical indicators. The recovered glass fibers also include residual carbon derived from $CaCO_3$ (laminate) or PET matrix (equipment parts). The shift of heating temperature from 500 to 700°C results in an increase in carbon content in laminate and other ash elements in equipment waste.

3-4- Reinforcement with the Recovered Fibers

According to the Carbon Composites e.V. association, the market for composite materials demonstrates 6% growth per year, and carbon plastics – 13-17% [10]. Lightweight, high-strength structures are especially relevant in the automotive industry, and the increasing trends of resource- and energy-saving make the creation of composite materials with cheaper secondary fibers promising.

Mechanical properties should be the main quality indicators at thermal regeneration of the reinforcing fibers when their introduction into building materials is planned. According to the literature data, glass fibers recovered by fluidized-bed pyrolysis at 450 and 550°C [52] showed a tensile strength reduced by 50% and 80%, respectively. Carbon fibers are less sensitive to temperature: above 600 ºC, the tensile strength of some pyrolysis-recovered CFs was reduced by over 30%, and the others were heat-resistant [25]. However, the parameters of the described ground fibers could not be measured due to their short length. Secondary composites using recovered fibers are usually created based on thermoplastic materials, such as LDPE [46]. The strength values of the fibers themselves [11] are 2-3 orders higher than their matrixes [27], so the bonding of their components plays a decisive role in strengthening composites.

Based on this hypothesis, the authors propose to combine fibers and matrices according to the principle of their composition similarity. In particular, it is rational to introduce carbon fibers into carbonized materials and glass fibers into mineral materials. This work proposes using secondary fibers: introducing CFs into active coal pellets and GFs – into silicate bricks. They have relatively low compressive strength: active coal – 100-1000 kg/cm2 (10-100 MPa) [53], silicate bricks – 52 MPa, which can be increased by dispersed reinforcement [49]. Tensile strength, usually determined for composites with secondary fibers, does not matter for these materials (not rated). On the contrary, compression and bending loads are the most important and controlled by the national standards, their values being absent in the literature.

Similarly, GFs recovered from waste material by thermolysis at 500°C, according to Mukhin & Klushin (2012), can serve for the production of glass-ceramic building material [54]. Like the laminate studied in this article, they are mixed with calcium carbonate, which does not prevent their further processing. The same technique revealed the reinforcing effect of the high-dispersed glass fibers obtained from printer parts at 500ºC (Table 4). The bulk density and porosity of the tested materials are also given there. The obtained structure is also characterized by visual microphotography (Figure 7).

Table 4. Strength of slag-based building blocks reinforced with obtained GFs (* 6 samples; ** 4 samples)

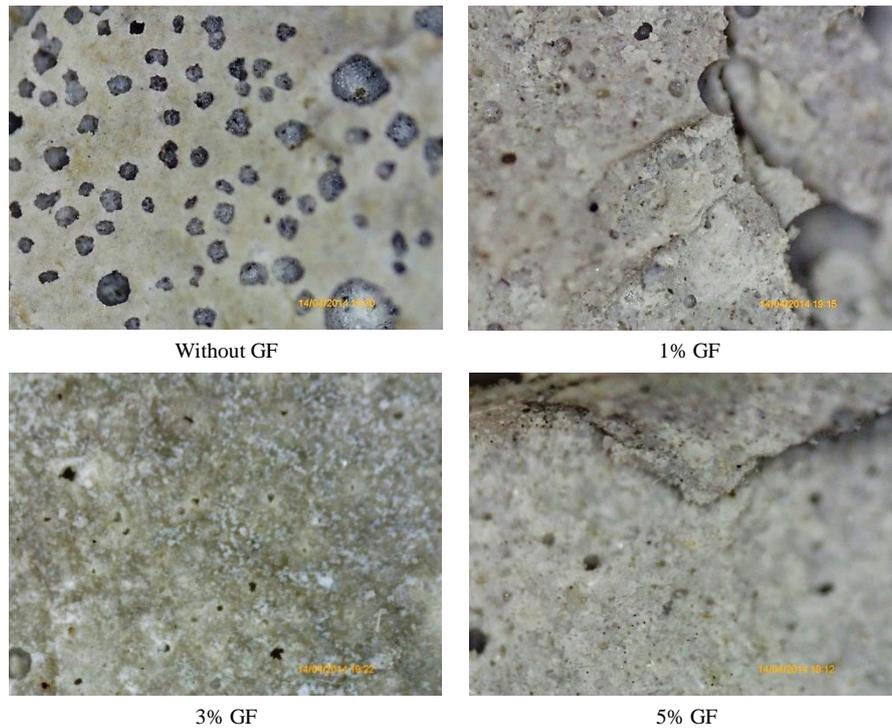| Characteristics of samples	Sample No.			
	1	2	3	4
Dose of GF additive to the raw composition, %	0	1	3	5
Average compression strength, MPa	37.4	51.2	36.2	39.5
Average bending strength, MPa	17.3	17.2	13.1	12.6
Bulk density, g/cm³	2.11	2.15	2.14	2.13
Porosity (by water), %	10.8	7.2	6.7	9.9

Figure 7. Visual photographs (×500) of slag-based building blocks reinforced with obtained GFs

In the reinforced silicate products, optical microscopy (Figure 7) recorded a homogeneous structure with a uniform distribution of short GFs.

It is established that theadditionof1wt% of the fibers significantly increases the compression resistance of the composite material, not affecting its bending strength, while 3 and 5wt% cause contrary changes. The marked positive effect could be caused by slightly higher density and lower porosity of the reinforced material (Table 4). The ultra-short glass fibers fill holes of the composite structure (Figure 7) and thus percept deformation loads. However, their addition of over 1 % decreases the bending resistance of the building blocks. This result correlates with Oshima et al. (2020) [41] and is explained by another breaking mechanism: releasing fibers from the matrix under great load [25]. The decrease in bending strength and improvement of this indicator requires more detailed research.

Glass fibers recovered from the laminate waste were also added (1%) to the slag mixture but caused a decrease in both compression and bending strength. To understand the low strength of reinforced materials, it is necessary to consider their possible fracture mechanisms [7]: tension-loaded specimens show the fiber-matrix interface microcracks, matrix microcracks, and fiber breakage resulting in fiber pull-out, fiber breakage, and delamination. In this case, it is possible to loosen the composite by non-directional fibers and weaken the silicate matrix bonds. However, these assumptions require further research.

As for secondary CFs, their application for reinforcing construction materials by our colleagues is described in [49]. The addition of only 0.7% by volume to slag-based blocks increased their compression strength by 20% and bending strength by 35%. However, this solution is expensive because of CF's high cost and significant spending on industrial production of building blocks.

In order to confirm the ability to use the CFs obtained by calcinations of waste CFRP at 700°C as a reinforcing component, they were added to the raw composition for the laboratory-scale activated carbon production. It has a low compression strength without the additive. To increase this index, 0.5–1.5wt% fiber was added to the raw paste for its preparation (Figure 8, Table 5).

Table 5. Strength of organoplastic-based activated carbon pellets reinforced with obtained CFs

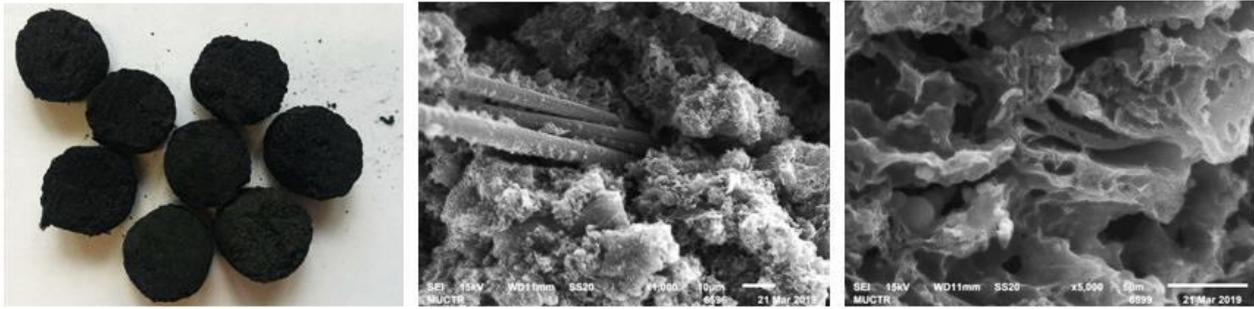| Characteristics of activated carbon s	Sample No.			
	1	2	3	4
Dose of CF additive to the raw composition, %	0	0. 5	1	1. 5
Average compression strength, MPa	0. 38	0. 82	1. 28	0. 31

Figure 8. Photographs of activated carbon pellets with the addition of 1% recovered CF at magnification×1, ×1000 and ×5000

The maximum compression strength (three times higher than that of the sample without additives (which is an outstanding result) is provided by reinforcement with 1wt% of secondary CFs, while further addition causes the loosening and weakening of the structure. This positive change is achieved by the fibers' strength and chemical contact with the carbonaceous matrix of the granules (Figure 8). A similar effect was highlighted by Oliveux et al. (2015) but at a much higher addition: 20–22wt% of recovered CFs added to polycarbonate increased tensile strength and bending strength by ca 2 and 2.5 times, respectively [22].

Despite the progress of the compressive strength, the bending strength could not be increased, which indicates insufficient interaction of the fibers with the matrix. A possible solution to this problem is to treat the regenerated fibers with inorganic [44, 45] or organic [46] modifiers, which requires more detailed studies.

3-5- Eco-Economic Assessment of Glass Fiber Regeneration and Reinforcing Reuse

This section presents the eco-economic assessment of the possible production of building blocks based on silicate slag (silicate bricks or slag-based building blocks), reinforced by 1% of thermally regenerated fiberglass, as characterized above. The raw material is silica sand, blast-furnace slag, liquid glass (sodium silicate solution), and recycled glass fibers. The production block diagram is shown in Figure 9.
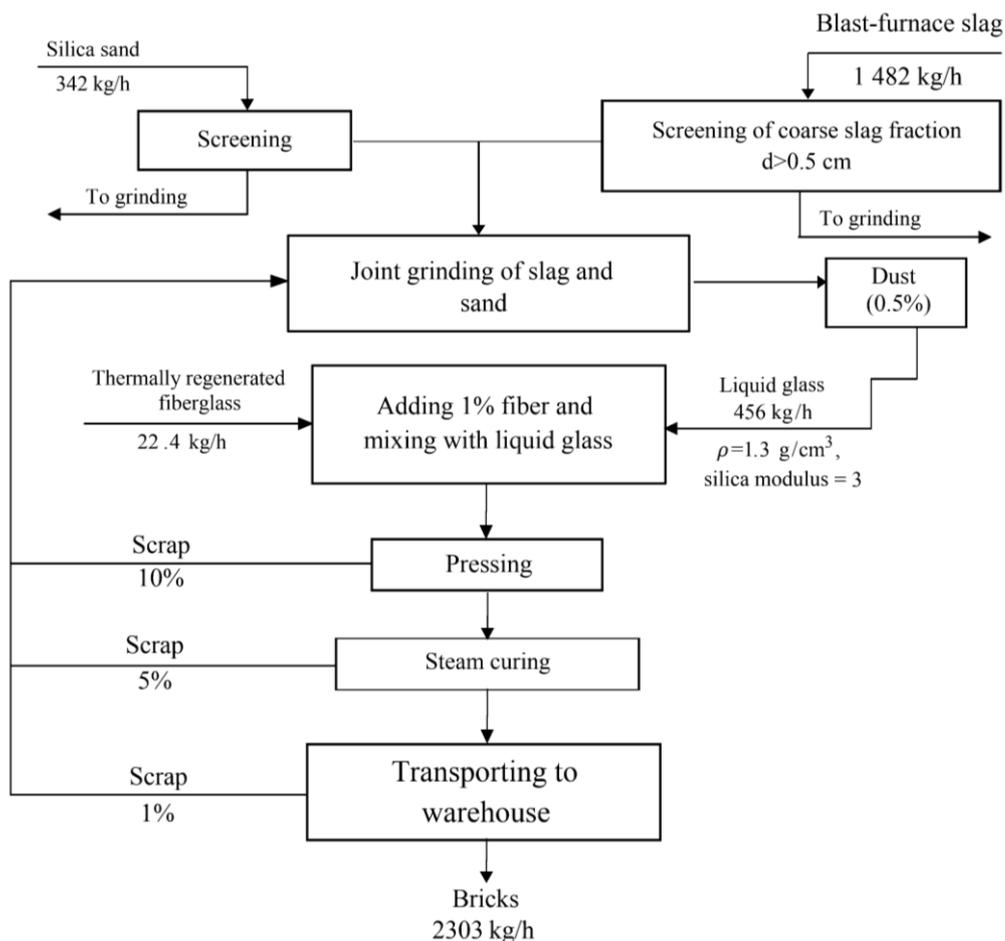

Figure 9. Block flow diagram of fiberglass reinforced bricks production

The methodology of eco-economic calculations is taken from the textbook [55]. Calculation of the production cost of building materials is presented in Table 6.

Table 6. Calculation of the production cost of 1000 pcs of lime-sand bricks

No.	Cost items	Price, (Rub/t)**	Raw materials consumption, (kg/h)	Annual consumption of raw materials, (tons/year)	Annual costs, Thousand (Rub/year)	Production costs of 1000 pcs, (Rub.)
			Materials			
1	Quartz sand	150	342	2028	357	**102**
2	Granulated blast furnace slag	300	1 482	8785	3090	**883**
3	Liquid glass	27	456	2703	86	**25**
4	Secondary fiberglass	0*	22.8	135	0	**0**
	Total materials					**1009**
5	Energy for technological purposes	-	-	-	1035	**296**
6	Wages of direct labor	-	-	-	928	**265**
7	Insurance payments	-	-	-	279	**80**
8	Maintenance costs	-	-	-	3675	**1050**
	Total shop cost of production					**1691**
9	Works general expenses	-	-	-	1417	**406**
10	Other production expenses	-	-	-	334	**96**
	Total works general expenses of production					**502**
	SUM-TOTAL					**3202**

* Fiberglass waste was donated by the issuer;   ** 1 Rub. = 0.016 Eur.

Brick production without fiberglass as a reinforcing component in the amount of 135 tons/year is considered the base variant, in which the fee for utilization of 450 tons/year of fiberglass waste is 108,000 rubles/year. The production of building materials reinforced with fiberglass in 1% of the mass of raw materials (135 tons/year respectively) is considered the proposed variant.

The annual eco-economic effect is equal:

$$E_y = C_{base}^P - C^P \tag{1}$$

where C_{base}^P is current annual costs of the base variant, Rub/year, and C^P is current annual costs of the proposed variant, Rub/year. For both variants:

$$C^P = C_i + E_n \times CC_i + P_{sw} \tag{2}$$

where C_i is production cost, Rub/year; E_n is norm coefficient of capital efficiency (0.15 Rub/(year-Rub)); CC_i is capital costs for the plant, Rub; and P_{sw} is payments for solid waste disposal, Rub.

The annual present value costs of the base variant include the cost of producing 3.5 million bricks and the removal of waste fiberglass:

$$C_{base}^P = 3500 \cdot 3202 + 0.15 \cdot 22322000 + 108000 = 14663300 \text{ Rub/year} \tag{3}$$

The costs of the proposed variant differ in the absence of payments for waste removal:

$$C^P = 3500 \cdot 3202 + 0.15 \cdot 22322000 = 1455300 \text{ Rub/year} \tag{4}$$

The annual commercial eco-economic effect is equal:

$$E_y^C = C_{base}^{PC} - C^{PC} = 10800 \ Rub/year > 0 \tag{5}$$

Consequently, the proposed production of building products with reinforcement obtained from waste fiberglass at the given parameters is environmentally and economically efficient. The potential profit from the increased compressive strength of reinforced bricks could increase the calculated effect, but this needs to be estimated apart.

4- Conclusion

Carbon and glass fiber regeneration from waste composites is of economic and environmental interest. Thermal regeneration (pyrolysis or calcination, or their combination) provides a means of recovering heat-resistant fibers without mechanical damage or using chemicals. This paper investigates the known waste carbon plastics and non-utilized industrial fiberglass plastics, including parts of office equipment with highly dispersed fibers and laminating coatings with the organo-mineral matrix. Thermogravimetric analysis showed that the polymer nets of the studied CFRP and GRP wastes are mostly destroyed at ~200–430°C, accompanied mainly by exothermic effects. The air atmosphere promotes matrix oxidation and fiber release at a lower temperature than nitrogen. The advantages of composite waste annealing despite pyrolysis are shown based on literature analysis and pre-tests. The optimal heat treatment conditions of the waste ground to 1 cm differ from the TGA results; it is rational to calcine the waste in the air at 700 °C for 0.5 h for CFRP and 500 °C for 1 h for GRP. This thermal treatment provides the quantitative recovery of fibers. However, their mechanical properties cannot be measured because of their small length. Micrographs of the recovered fibers reveal their nearly clear surface free of binder residues and the absence of damage, but the ultimate analysis points to elevated oxygen content, indicating surface oxidation.

Based on a comparison of known parameters of the fibers and binders, a novel principle for making composite materials is proposed: a similar composition of regenerated fibers and matrix should provide their interaction and reinforcing effect. The obtained CFs up to 5 mm long were successfully used to reinforce activated carbon granules, increasing their compression strength three times. We proposed using separated short (5-20 μm) GFs to reinforce building slag blocks needed to improve their compression strength; the addition of highly dispersed glass fibers of 1% resulted in its growth by 37% due to higher density and lower porosity. However, we have not increased the reinforced composites' bending strength, which seems to be caused by weak bonding with their matrix. This problem could be solved by treating regenerated fibers with alkaline, acid, oxidative, or organic modifiers. The surface composition of thermally recovered fibers and their interactions with new matrixes also needs further exploration.

Thus, both thermally recovered fibers may be reused as reinforcing components in composite materials of corresponding (carbon or mineral) composition. Calculation of the cost and eco-economic effect of producing silicate bricks with regenerated fiberglass reveals the benefit of this waste recycling by eliminating payments for its disposal. In order to understand the impact of thermal regeneration conditions on the fibers' quality, it is necessary to find a way to measure their tensile strength.

5- Declarations

5-1- Author Contributions

Conceptualization, A.V.N.; methodology, A.V.N., and N.N.K.; software, L.K.V.; validation, I.V.P.; formal analysis, I.V.P., and L.K.V.; investigation, I.V.P., and L.K.V.; resources, N.N.K.; data curation, I.V.P., and L.K.V.; writing—original draft preparation, A.V.N.; writing—review and editing, A.V.N.; visualization, L.K.V.; supervision, A.V.N.; project administration, N.N.K.; funding acquisition, A.V.N. All authors have read and agreed to the published version of the manuscript.

5-2- Data Availability Statement

The data presented in this study are available in article.

5-3- Funding

5-4- Acknowledgements

5-5- Institutional Review Board Statement

Not applicable.

5-6- Informed Consent Statement

Not applicable.

5-7- *Conflicts of Interest*

The authors declare that there is no conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancies have been completely observed by the authors.

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