Chapter 7

Recycling of Polymeric Composite Materials

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Abstract

This chapter treats studies about the methods and technologies used to recycle the polymeric composite materials and develop new recipes using waste of polymer composite materials resulted from recycling. Composite materials obtained from recycling are presented, with a complete recovery of waste glass fibers. Also, the mechanical properties for new structures of polymeric composite materials, containing additional materials were presented. These were obtained from the recycling of composite waste. A morphology analysis of fracture area of composites samples was done. At present, the polymeric composite materials present a great scientific and technical interest, which justify both the development of research in this field, and the expansion of production of such materials. The author treats aspects regarding a current problem due to the large number of polymeric composite materials waste, and reduced of environmental impact. This field is representing one of the top viable research directions.

Keywords: waste, composite materials, glass fibers, polymer matrix, recycled materials

1. Introduction

In the last decades have revealed significant changes in the world in terms of the use of materials in various fields, mutations complained so special requirements of peak areas and increasingly diversified requirements related to the production of consumer goods and not least all environmental requirements.

Composite materials are considered engineering materials that can replace non-ferrous or ferrous materials. Polymer composite materials have a large applicability in a different industries such as electrical engineering, electronics, building and civil engineering, rail, road and marine, aerospace technique and aeronautical, etc. [1–8].

Composite materials consist of reinforcement material (glass fiber, carbon fiber, Kevlar, etc.) and a matrix (polyester resin, epoxy resin, and so on). Fiber glass is the most used reinforcing materials. They have many characteristics: high tensile strength, high chemical resistance, low cost. To obtain low price or to give high properties to a composite material we can include in a structure auxiliary materials, like: coupling agents, catalysts, pigments, accelerators and so on [9].

The storage of waste composite materials and the recovery of these, it's an important problem that we have nowadays. In **Figure 1**, it can be seen composite parts out of use and composite waste resulted from different production processes that occupy considerable spaces for storage. In time the accumulation of such materials can create serious problems to the manufacturing companies.

Because the interest to find solutions for recovering or recycling is very low, the accumulation in time of composite materials waste is very significant. We can obtain a material rich in glass fiber by grinding the composite materials waste. Thus, it's obtaining a very valuable

a.

b.

Figure 1. Composite materials waste stored.

reinforcement that can be embedded in other materials or can be used for obtaining reinforced composite materials. A solution for recycling such composite materials has been to grind these materials, **Figure 2** and create new composite products.

The recovery and recycling of polymeric composite materials has experienced an important concern in the last years. Researches dedicated to technologies for recycling composite materials were initiated and carried out by different authors, [10–13].

Mixtures of concrete with sand and fiberglass waste are known [14–16]. Waste composites can be used for concrete reinforcement or for a variety of construction materials. However, these materials from the known technical solutions point of view have higher density, lower mechanical properties, and the external factors like: UV radiation, moisture, sunlight, influence the degradation of these.

A chemical recycling of glass fiber reinforced epoxy resin has been proposed by Dang et al. [17]. PET reinforced with glass fiber was recycled by Giraldi et al. [18], while Bartl et al. [19] study the fibers recycling obtained from tires. Vilaseca et al. [20] treat in their research recycled Kraft fibers (recycled softwood fibers) that coming from old sacs, used as reinforcement for the preparation of polypropylene composites. Composite materials obtained from wood fibers were analyzed by Nemes et al. [21] and Augier et al. [22].

Hugo et al. [23] were investigated recycled polymers with a range of different fillers, and developed applications that use waste thermoplastic polymer.

In order to make the ornamental plates used in the field of construction, a number of manufacturing processes are known which use sand mixtures with different binders: plaster, whitewash or cement [24, 25]. The obtained material as a dry mix or mortar is poured and pressed into a mold. After reinforcement of the material, the plate is extracted from the mold, after which time is left for stabilization, and then it can be used.

Figure 2. Ground glass fiber.

For the same purpose, for the production of alternative materials it is known the manufacturing process of reinforced mortars used in construction [26, 27]. These mortars include besides sand, whitewash, cement, gypsum and various reinforcement materials, such as: hemp fiber, glass fiber, etc.

The disadvantages of these processes are the high time of plates obtaining and their reduced mechanical characteristics. Other disadvantages of the plates obtained by these processes are the high density of the material and the degradation in time under the influence of external factors: humidity, sun, UV radiation.

Reinforced materials and manufacturing procedures have a significant influence on the quality, productivity and competitiveness of composite structures. The interface between matrix-reinforced materials plays an essential part in the mechanical behavior and fabrication of composite materials.

2. Recycling of composite materials

The recycling of materials organic macromolecular surgery is more complicated than with traditional materials (glass, paper, metal), because there is an impressive variety of polymers, which in most cases are not compatible with each other, in the event of a global recycling [28–30].

For the recycling of polymeric materials there are several solutions:

- The separation of the constituents of mixtures in order to recycling of each individual component;
- The direct transformation of the mixture without prior sorting, in order to reduce the volume of waste.

From the view point of recycling, waste can be classified as:

- Manufacturing waste (10% of total waste) mainly formed of a single material. Because they are not contaminated (or less purified) with other materials, recycling is easier. Typically, these wastes are reintroduced into production lines.
- Waste easily separable. They consist of 1–2 or more polymers (mix macroscopic scale) otherwise contaminated materials (fillers). These materials are, at least theoretically separable.
- Microscopic mixtures or intimately connected (soldered, interpenetration). This is the case most difficult to treat because the separation of constituents is difficult or even impossible, requiring complicated operations. In this category fits very well with organic matrix composites. The most representative example is the waste from the automotive industry. In this case, the blend will find materials (resins) thermoplastic, polymer mix, fibers, fillers and multilayer composite materials.
- Materials of the recycling, currently applies in particular to the first two categories mentioned.

The recycling after separation of mixtures is a much more interesting because you have to drive theoretically product with performance very close to the base polymeric materials. In practice, the properties of recycled materials approaching initial properties of the base material, unless methods are very effective waste sorting and waste have undergone significant degradation during operation.

Sorting of waste is done according to the basic polymeric material. The sequence of operations mainly comprises the following steps:

Shredding. At this stage, the materials must be recovered shrinks size to be easily transported and handled.

Separation of metals is well-established methods, obviating the mixture all existing metal fragments (e.g. electromagnetic methods).

- Shredding and/or spraying. This stage is complementary to the first mentioned. At this stage takes place and the washing waste. Choppers additional step is required for further processing easier.
- Washing and drying is intended to remove all impurities. In general washing is done with water and detergent, but depending on the nature of impurities at this stage can put complex problems. Flushing is required followed by spin drying to remove water.
- Separation using air or hydro cyclones and disposal are conventional methods for separating materials based on the difference in density. The process stream is brought into contact with a stream of air (water) in a cyclone. With these separation methods do not obtain high degrees of purity. Moreover, this mineral phase (mineral fillers) often change the apparent density of the polymer, making it difficult to separate.

By materializing the proposed project creates prerequisites for achieving scientific and technological results, competitive at European level in order to increase the visibility of Romanian research, especially to subsequently transfer the results in socio-economic practice.

The resolving of proposed assignment will lead to the development of science-based knowledge of the manufacturing processes of parts of polymeric composite materials reinforced with biodegradable waste. The aim is to achieve a topic fundamental research, advanced to develop methods and technologies for recycling polymer composite materials and develop new recipes using biodegradable waste. It thus aims to improve the quality, productivity and competitiveness of industrial products. This is possible by using a multidisciplinary approach to research that brings together knowledge of chemistry, mathematics, physics, rheology and technological engineering.

3. Proposed new composite material and manufacturing method for ornamental synthetic plates

Both composite materials and waste composite materials resulting from production processes occupy important storage areas with an impact on the environment.

It shows the utilization of the glass fiber waste obtained by grinding the waste resulting from the manufacturing technological process of composite materials or removing them from their use and incorporating them in a product with applications in the field of industrial constructions, offering superior mechanical characteristics to the existing similar products.

3.1. The matrix

The mold, for obtaining the ornamental synthetic plates, is made of two separate modules, one of silicone rubber and one of fiber reinforced composite material. In **Figure 3** are presented the steps of mold forming.

Achieving the active part of the silicone rubber mold eliminates the need for additional separation planes, and the mold stiffness is ensured by reinforcing it with a fiber reinforced composite material. The use of matrix mold from the silicone elastomer eliminates additional separation planes reducing the cost of the mold.

The manufacturing process of the matrix, involves the following phases:

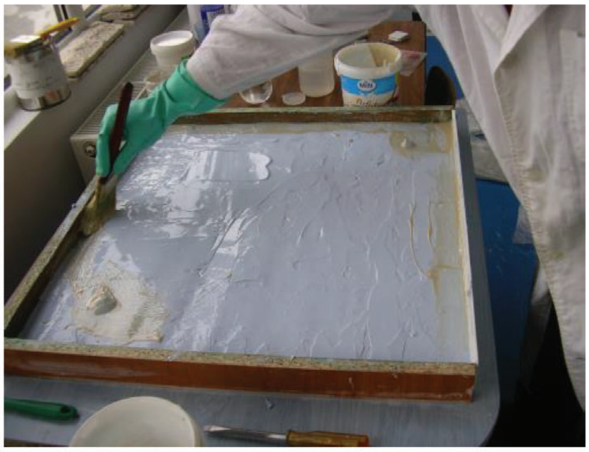
- arranging the stones, **Figure 3(a)**,
- filling the joints with gypsum,
- forming the outer frame,
- applying the demulation layer, PVA (Polyvinyl alcohol),
- preparation and application of the silicone rubber, **Figure 3(b)**,
- realizing the composite structural element for the silicone mold, fiber glass/polyester matrix, **Figure 3(c)**,
- demulation of the mold from fiber reinforced composite material, **Figure 3(d)**,
- demulation of the mold from silicone rubber, **Figure 3(e)**,
- trimming edges, **Figure 3(f)**.

3.2. The material

The process of ornamental synthetic plates manufacturing consists in a mixing, in a recipient, of calcium carbonate with the polyester matrix 5 minutes, casting these materials into a modular mold made from silicone and reinforced by a fiber-reinforced composite material and maintaining at room temperature for 20 minutes until the matrix gel point has been reached, mixing in another recipient for 10 minutes of waste glass fiber ground with a polyester matrix and 0–0.3 mm sand, and molding it in the die over the initial molded material, holding molds in the die approx. 2 hours at 60°C, resulting an ornamental plate that is released from the mold after composite material polymerization. The material together with the unpolymerised matrix is deposited in a modular mold.

The sand was used as a low-cost reinforcement material in the form of particles with transparent aspect. The morphological analysis of the sand is shown in **Figure 4**.

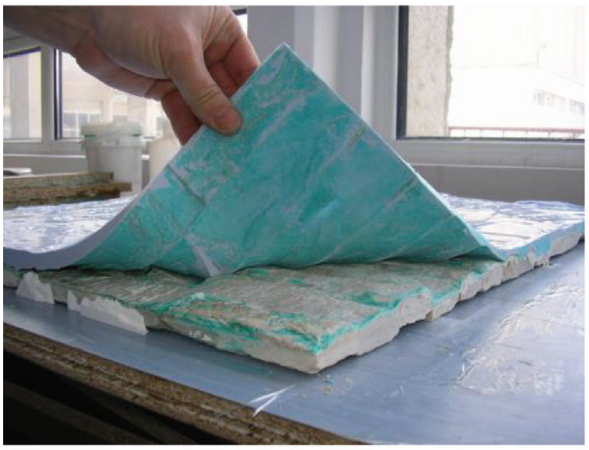
a.



b.

c.

d.



e.

f.

Figure 3. Steps of mold forming.

The manufacturing process of synthetic decorative plates involves the following phases:

a. preparing the mold and applying the first layer release agent,

b. preparing the first mixture consisting of 60% polyester matrix and 40% Calcium Carbonate $CaCO_3$, mixed approx. 5 minutes in a recipient,

c. casting the first mixture so that it will cover more than 1–3 mm the height of the mold asperities and the maintenance until reaching the gel point, at room temperature,

d. preparing the consolidation mixture consisting of 30% sand of the 0–0.3 mm sort range, 30% grinded glass fiber waste and 40% polyester matrix and mixing it for 20 minutes,

e. casting the consolidation mixture over the first mixture until the mold filling and leveling the upper part,

f. transferring the mold with the composite mixture in a polymerization heat and maintaining in the mold at a temperature of 60°C, about 2 hours, until the composite material polymerization,

g. mold release and obtaining the ornamental synthetic plates.

The composite material consists in obtaining a synthetic material composed of two component mixtures, **Figure 5**:

- the first mixture, which forms the surface layer that copy the mold and render the appearance of the synthetic plate, **Figure 6**, consist of 60% polyester matrix and 40% Calcium Carbonate $CaCO_3$, mixed approx. 5 minutes, casted and maintained until the gel point was reached;
- the second mixture, of consolidation, consist of 40% polyester matrix, 30% sand of the 0–0.3 mm sort range, 30% grinded glass fiber waste, casted over the first mixture and maintaining in the mold until the polymerization, resulting a synthetic composite material reinforced with glass fiber having superior mechanical properties to similar materials used in construction.

The percentages mentioned above represent the percentage of the total volume of the constituent materials.

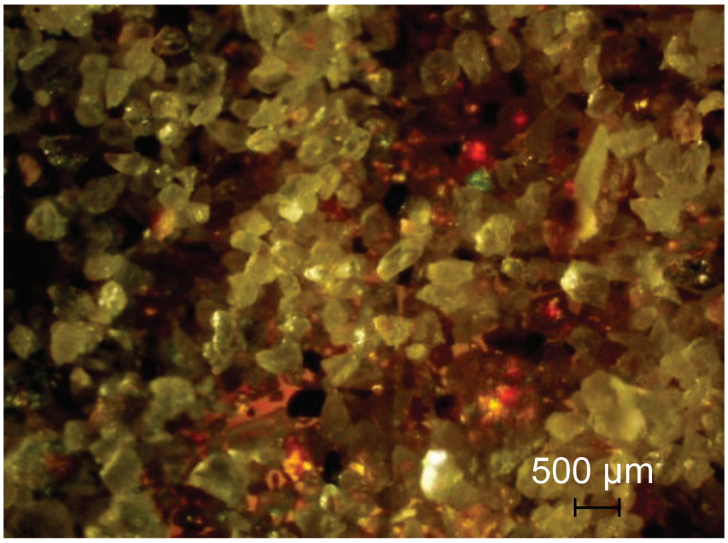


Figure 4. Sand grains.

Figure 5. The synthetic material consisting of two component mixtures.

The obtained material is a compact material with resistance at external agents, the process being easy to achieve. The composite material provides superior mechanical characteristics to traditional materials and can be used in other applications in the construction field such as reinforcing structures.

The following advantages are obtained:

- composite material waste utilization, thus solving the significant problem of glass fiber waste;
- enlarge the range of materials used in construction;
- making a composite material having superior mechanical characteristics and low density with respect to traditional materials;

- getting some plates with good look, imitating the natural stone, which can be colored in large quantity in the production process and can be easily mounted on facades and buildings;
- the technological simplicity of the process does not require substantial investment;
- the use of matrix mold from the silicone elastomer eliminates additional separation planes reducing the cost of the mold;
- increasing the mechanical characteristics when using these materials at low temperatures.

3.3. Mechanical tests

For mechanical tests, from the obtained material was done cubic specimens with 50 × 50 × 50 mm dimensions, according to EN 12320-3 standard.

The obtaining process of the composite plates that include in the structure glass fiber waste was hand lay-up. The mechanical properties of composite plates were determined to perform the experimental test at compressive load.

Figure 6. The ornamental synthetic plate.

No.	Force [KN]	Average force [KN]	Average compressive breaking strength [MPa]	Density [Kg/m³]
1.	185.8			
2.	191.2			
3.	193.2	189.96	78.27	1380
4.	187.3			
5.	192.3			

Table 1. Compressive tests results.

Table 1 shows data following the compressive stress of cubic specimens, the constituent composite material remains bonded through filaments of reinforcement material.

The composite plates provide higher mechanical properties, lower costs and reduce waste materials in the environment.

The experimental data shows that the new materials have good mechanical properties and they can be successfully used in the dimensioning and verification process of composite structures resistance.

3.4. Microscopy study

The microstructure of fracture samples from waste glass fibers/sand/polyester matrix composites was analyzed using a metallographic microscope type Optika XDS-3 MET [31, 32].

The sand grains contain in the structure over 90% silica (SiO_2). The glass fibers are made from silica sand, which melts at 1720°C.

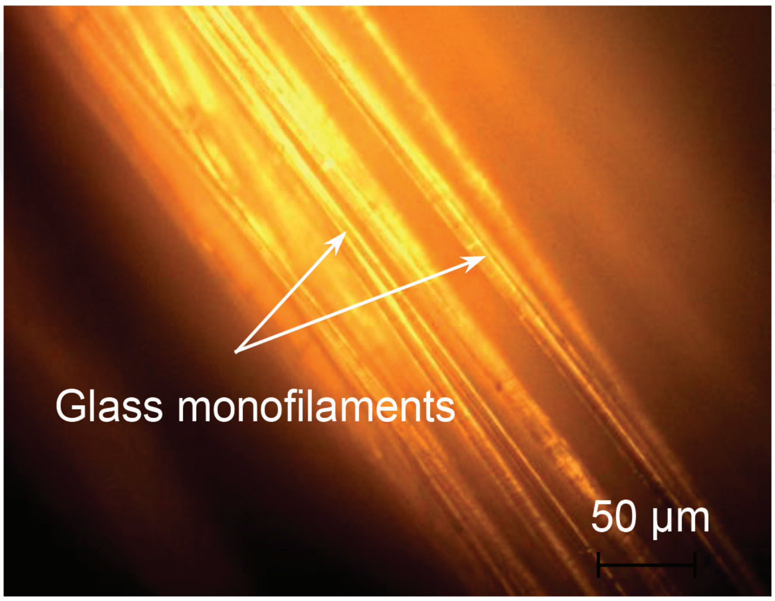


Figure 7. Non-impregnated glass fiber monofilaments.

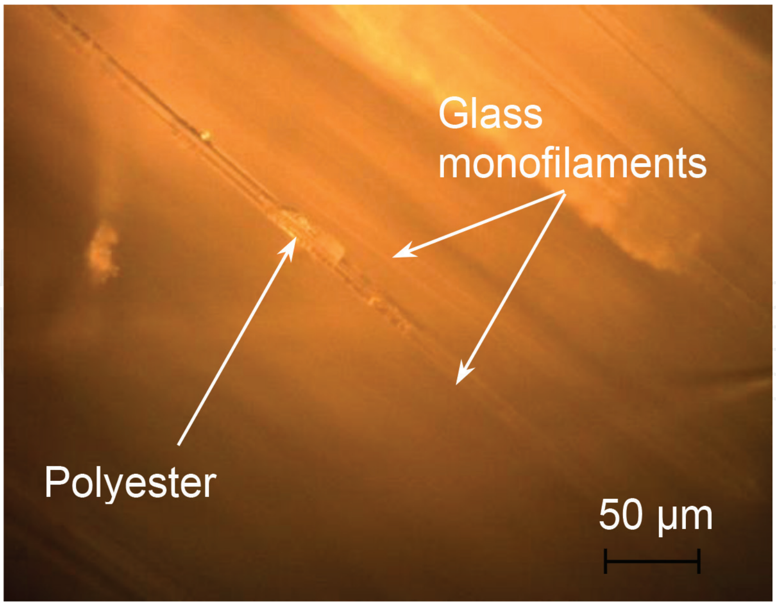


Figure 8. Impregnated glass fiber monofilaments with resin.

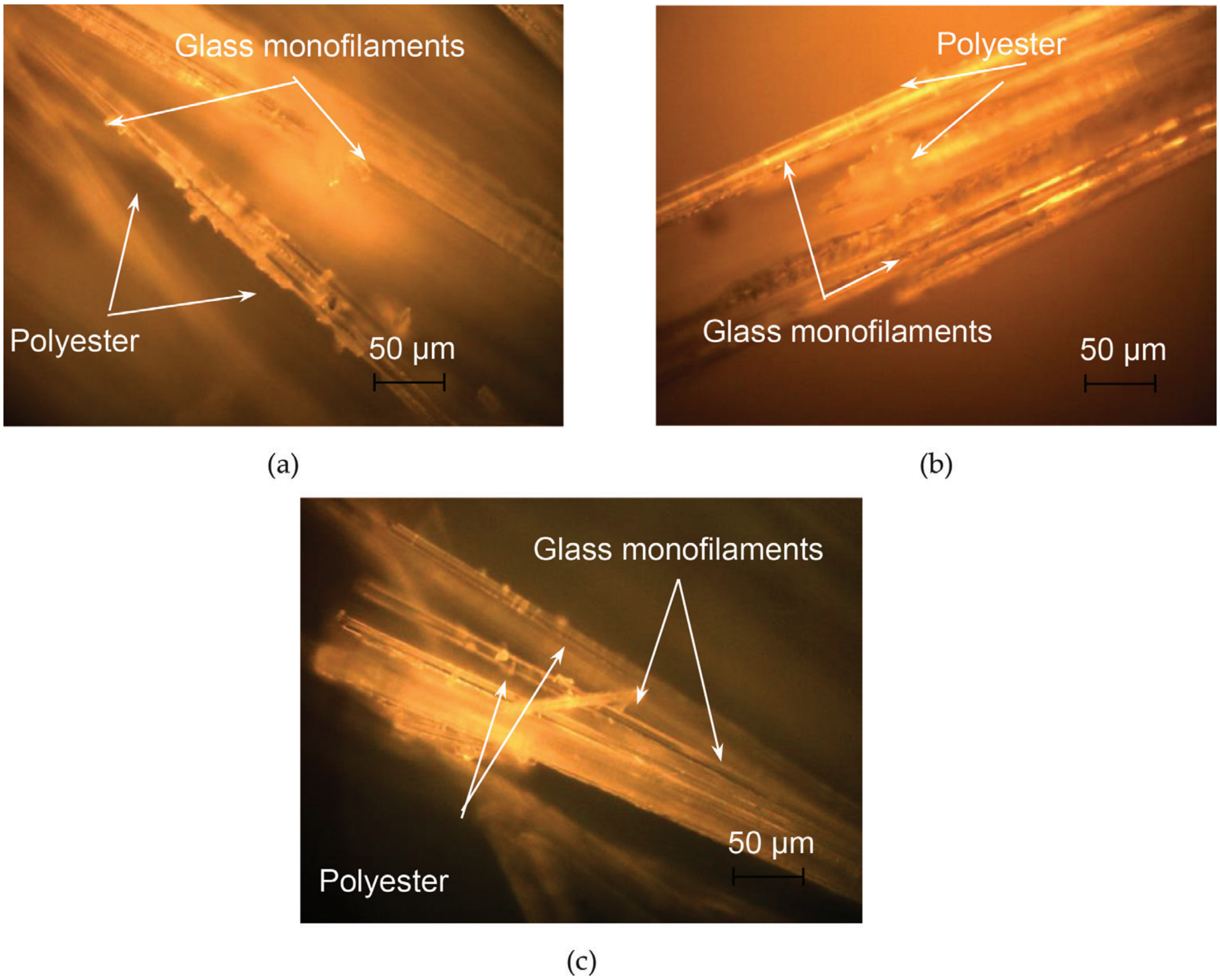


Figure 9. Waste glass fiber monofilaments impregnated with resin.

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The monofilaments of non-impregnated glass fiber have a smooth and glossy surface, specific to the glass. These were analyzed using the optical microscopy, **Figure 7**. To have a good adhesion at the interface between matrix and fibers, the surface of glass fibers is treated with silane. In **Figure 8** it's show the impregnated glass fiber monofilaments with resin.

Figure 9 shows the adhesion between matrix and glass fiber monofilaments. **Figure 10** illustrates that sand grains and glass fiber monofilaments are well impregnated with resin according to the morphological analysis of the fracture area. It can be observed a good compatibility between resin, filaments and sand, and a good impregnation of the matrix.

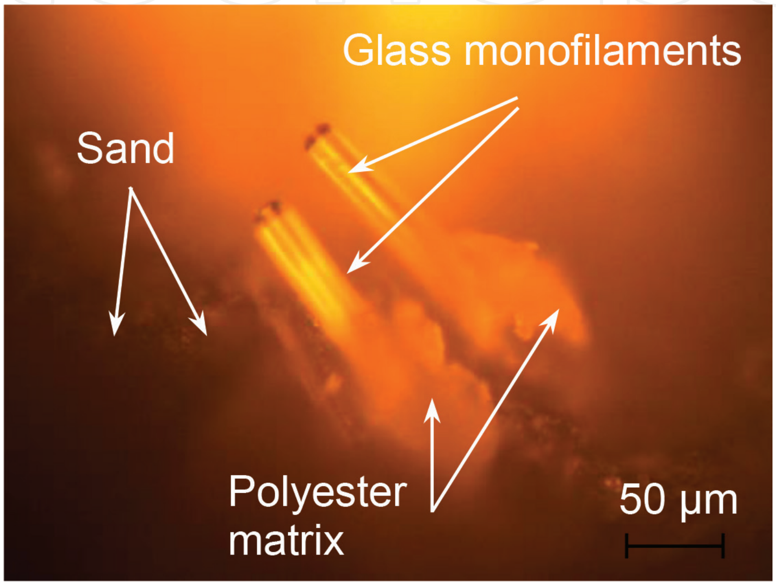

Figure 10. The fracture zone of waste fiber glass/sand/polyester resin plate.

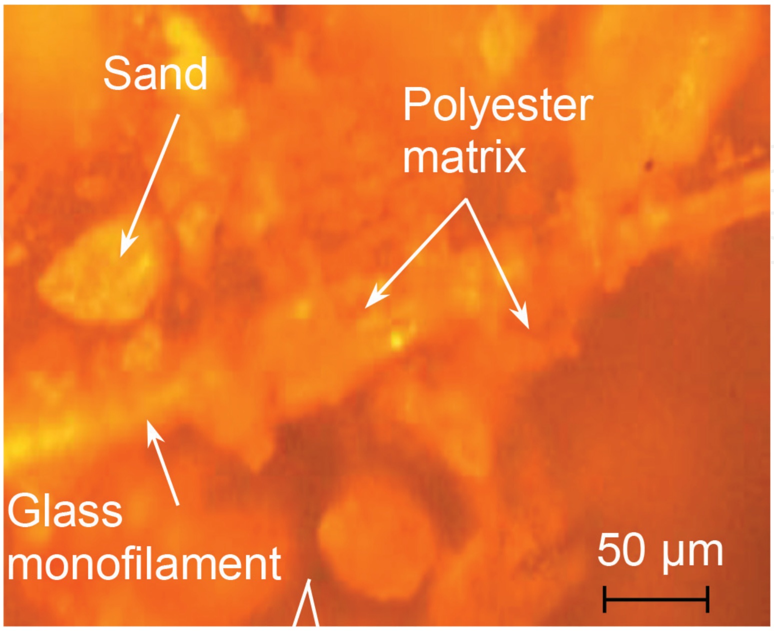

Figure 11. Sand and glass monofilaments in polyester resin.

According to the **Figure 11**, it can be observe the achieved connection between polyester matrix, glass fibers and sand, because of the particles of sand and polyester resin that were well glued on the glass monofilaments. Thus, a composite material with low density and high mechanical properties has obtained. These types of materials allow one reuse of glass fiber waste. Using these types of materials at low temperatures increases their mechanical characteristics.

4. Energy dispersive x-ray analysis

With the help of the energy dispersive x-ray spectroscopy (EDX) was performed the elemental analysis of the polyester resin and waste fiber glass. The weight fraction ratio is composed on the total weight of the chemical substances analyzed. The predominance of silicon and aluminum can be observed in the **Figure 12**, after the elemental EDX analysis was done of the waste glass fiber [32]. Also, small amounts of carbon, oxygen, sodium, magnesium and calcium are detected. The obtained data are expressed in two ways, both atomic percent (At.%) and weight percent (Wt.%). The atomic and the weight percentages of the fiber glass elements are: C with 30.96At.%, 17.67Wt.%; O with 24.12At.%, 18.34Wt.%; Na with 0.34At.%, 0.37Wt.%; Mg with 0.38At.%, 0.43Wt.%; Al with 14.43At.%, 18.50Wt.%; Si with 21.12At.%, 28.19Wt% and

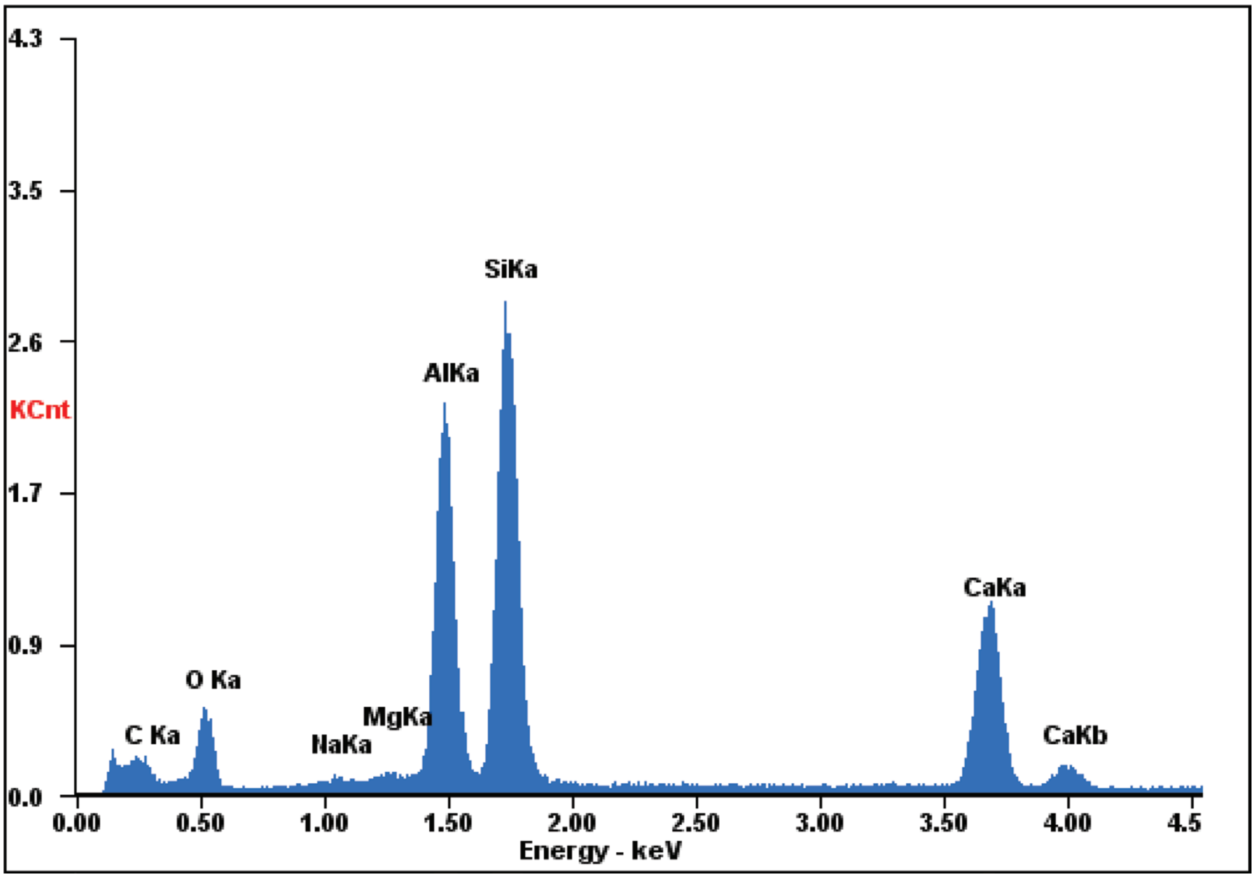

Figure 12. EDX analysis of the chemical constituents from the glass fiber.

Element	At.%	Wt.%
Si	21.12	28.19
Al	14.43	18.50
Ca	08.66	16.50
O	24.12	18.34
C	30.96	17.67
Mg	00.38	00.43
Na	00.34	00.37

Table 2. EDX analysis of the glass fiber.

Ca with 8.66At.%, 16.50Wt.%. In **Table 2** are presented the elements on the surface of a waste glass fiber.

The EDX analysis of the matrix polymer is presented in **Figure 13**, [32]. The predominance of carbon, silicon and oxygen is obviously in this case study. Also, small amount of sodium, aluminum and calcium are detected. The atomic and the weight percentages of the polyester matrix

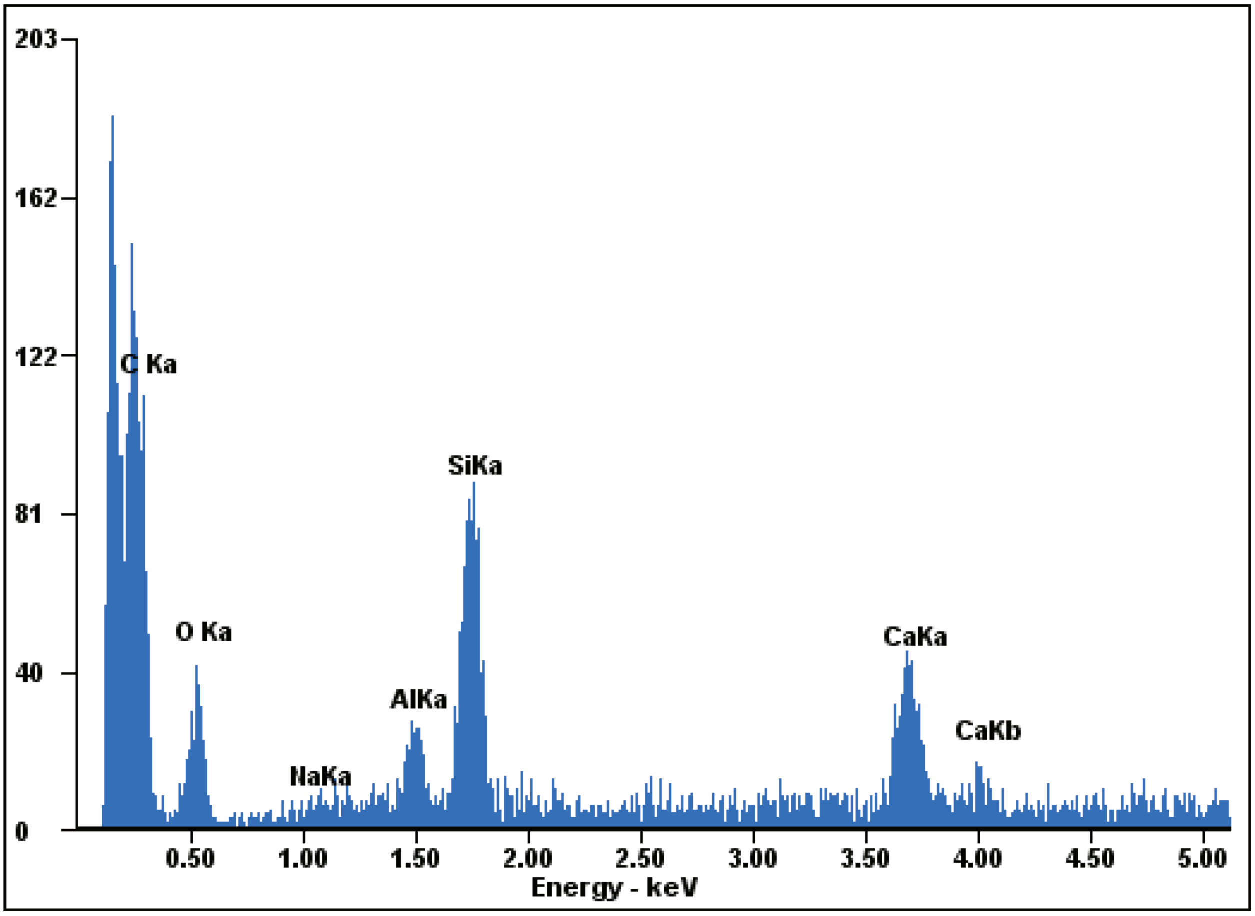

Figure 13. EDX analysis of the matrix polymer.

Element	At.%	Wt.%
C	74.80	62.18
Si	04.78	9.30
O	15.97	17.69
Ca	02.87	7.98
Al	01.28	2.39
Na	00.29	0.47

Table 3. EDX analysis of the matrix.

elements are: C with 74.80At.%, 62.18Wt.%; O with 15.97At.%, 17.69Wt.%; Na with 0.29At.%, 0.47Wt.%; Al with 1.28At.%, 2.39Wt.%; Si with 4.78At.%, 9.30Wt.% and Ca with 2.87At.%, 7.98Wt.%.

In the table above, **Table 3**, are presented the elemental quantitative analyses that give us the polyester matrix elements on the surface.

5. Conclusions

The storage of waste composite materials and the recovery of these, it's an important problem that we have nowadays. Composite parts out of use and composite waste resulted from different production processes occupy considerable spaces for storage. The manufacturing companies can be affected by serious problems, because of the accumulation in time of these types of materials.

A solution for recycling these composite materials has been to grind these materials and create new composite products.

A new composite material obtained from recovered materials, with a complete recovery of glass fibers waste is described in this study.

The composite material for obtaining the ornamental synthetic plates consist of a mixture that forms the surface layer, that copy the mold and render the appearance of the synthetic plate, consist of 60% polyester matrix and 40% Calcium Carbonate $CaCO_3$ and a consolidation mixture consist of 40% polyester matrix, 30% sand of the 0–0.3 mm sort range, 30% grinded glass fiber waste, casted over the first mixture and maintaining in the mold until the polymerization.

The experimental properties obtained indicate a very good mechanical behavior of the new composite materials. The compressive tests indicate a high value, superior of traditional materials, like concrete.

The fracture area of the samples from glass fibers waste/polyester resin/sand composites was microscopically analyzed. A good compatibility between resin, filaments and sand, and a good impregnation of the matrix was obtained.

The new composite material contained grinded glass fiber waste, polyester matrix and sand all mixed together. After polymerization of the resin we obtain a composite material with superior mechanical properties. This material can be used in different applications, like: strengthening composite parts (ornamental garden stones, ornamental composites plates, garden furniture, additive materials and so on), polyester reinforced concrete.

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