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Advanced Polypropylene and Composites with Polypropylene with Applications in Modern Medicine

Doina Elena Gavrilă, Victor Stoian, Alina Caramitu and Sorina Mitrea

Abstract

Synthetic polypropylene (PP) is used extensively in many fields of medicine. On the one hand, it is utilized in the manufacture of medical equipment: syringes, storage, transport, electric cables, etc. On the other hand, synthetic, nonabsorbable isotactic PP (iPP) is often used to perform meshes for hernia and pelvic organ repair operations, as well as in urinary incontinence. Products that release in time from meshes are depending on the conditions in which they are utilized, can produce undesirable reactions for the human body. For this reason, nonabsorbable synthetic PP was replaced in surgical sutures and meshes with bio polypropylene (bio PP). The chapter analyzes the specific characteristics of these polymers as well as their degradation due to the influence of different factors: humidity, perspiration, temperature, and presence of bacteria. Obtaining new composite materials with PP as matrix and metal powders as fillers is considered as a possibility of their use in vital problems such as cancer detection and treatment. These allow the emergence of new strategies in the design of biosensors that use nanocomposite materials with different fillers and polymeric films. The chapter analyzes the characteristics of new composite materials with PP matrix and metallic powders of iron (Fe).

Keywords: polypropylene, bio polypropylene, degradation, surgical sutures, polypropylene composite with iron powders

1. Introduction

Polypropylene (PP) is a nonpolar, partially crystalline thermoplastic polymer from the polyolefin group, in some aspects similar to polyethylene (PE) concerning the electrical properties, but it has improved mechanical properties and thermal resistance. In these latter days, PP is the most used thermoplastic, exceeding PE [1]. The properties of PP depend on molecular weight and molecular weight distribution, crystallinity, and tacticity [2]. It is a low-cost polymer with excellent properties like flame resistance, transparency, high heat distortion temperature, dimensional stability, and recyclability, making it ideal for a wide range of applications. It is a lightweight polymer with a density of 0.90 g/cm^3 that makes it suitable for many industrial applications and has chemical resistance, long life for electrical

and mechanical applications, and thermal and optical properties. It is environment-friendly and nontoxic and has replaced polyvinylchloride (PVC) because it does not release any toxic gases when burnt [3]. PP can be modified by using micro- and nano-sized fillers or reinforcing agents to get desired characteristics required by the application area. The tacticity of PP can be controlled: the branching of linear PP can be done, resulting in higher molecular weight product with higher tensile strength, higher modulus of rigidity, and higher heat resistance. PP does not absorb water [4].

2. Biopolymers and biopolypropylene

Biopolymers are polymers produced by living organism and contain monomeric units that are covalently bonded to form larger structures. A classification according to monomeric units and the structure of the biopolymer divide these materials into three classes:

- Polynucleotides (DNA and RNA) which are long polymers composed of 13 or more monomeric units covalently bonded in a chain. DNA (deoxyribonucleic acid) and RNA (ribonucleic acid) are examples of polynucleotides with distinct biological function. DNA consists of two chains of polynucleotides, each in the form of a helical spiral.
- Polypeptides, which are short polymers of amino acids.
- Polysaccharides, which are often linear-bonded polymeric carbohydrate structures.

Other examples of biopolymers are rubber, melanin, lignin, cellulose, etc.

An important difference between polymers and biopolymers is their structure. All polymers are made of repetitive units called monomers. Although biopolymers have a well-defined structure, this is not a defining characteristic [5–7].

Structural biology is the domain for the study of structural properties of biopolymers. The exact chemical composition and the sequence in which the units are arranged are called the primary structure, in the case of proteins. Many biopolymers fold into a characteristic compact shape, as well as secondary and tertiary structure. These structures determine the biological functions and depend in a complicated way on their primary structures. Most synthetic polymers have much simpler and more random structures. Molecular mass distribution is missing in biopolymers: all types of biopolymers contain similar sequences and numbers of monomeric units. The phenomenon is called monodispersity, in contrast to the polydispersity from synthetic polymers. Biopolymers have a polydispersity index of 1.

Synthetic biopolymers are human-made copies of biopolymers obtained by abiotic chemical routes. Abiotic components or abiotic factors are nonliving chemical and physical parts of the environment that affect living organism and the functioning of ecosystems. In biology, abiotic factors can include water, light, radiation, temperature, humidity, atmosphere, acidity, and soil. The macroscopic climate often influences each of the above [8, 9].

As humanity becomes more aware of climate change and pollution with plastics, the idea of using biopolymers is becoming more and more important. At this time, alternative methods of producing biopolymer materials are however

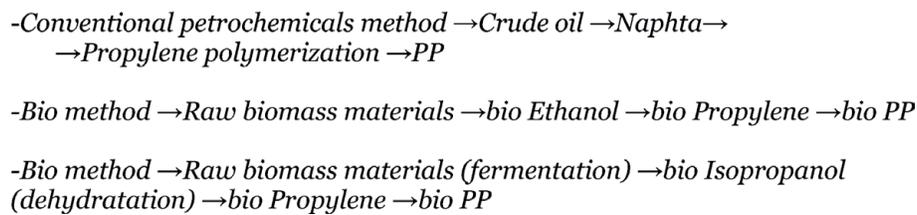


Figure 1.
Scheme of methods for obtaining PP and bio PP.

reduced. It is necessary to accelerate the transition from petrochemical to bio-based plastic materials and to obtain a great diversity of them, including polyolefins as PP and PE [10]. Recently, in the bio-based polymer industry, the world's first parallel production of bio-based PP and bio-based low-density PE at the commercial scale has been announced. However, due to the technical difficulties, production of bio PP from biomass has not been established at the industrial level. The new production method is proposed which sees various biomasses, mainly nonedible plants fermented to produce isopropanol (IPA), which is then dehydrated to obtain propylene. This one could prove to be a more cost-effective way to manufacture bio PP. **Figure 1** shows schematic methods of obtaining PP and bio PP.

Biopolymers remain less than one percent of plastics manufactured worldwide and remain until very recently 2–4 times more expensive than petrochemical products. Moreover, most biopolymers do not yet save more carbon emissions than are required to manufacture them. As most biopolymers are produced from plants, sugars, starches, or oils, it was estimated that replacing 250 million tones of the plastic manufactured each year with bio plastics would require 100 million hectares of land (7% of the arable land of the Earth).

3. Analysis of degradation of bio polypropylene for surgical sutures and for meshes used in medical repair operations

3.1 Generalities on suture materials

Surgery sutures have improved in recent years, both in terms of surgical technique and materials used, which explains successes in transplant, vascular, and digestive surgeries, etc. Initially, sutures were made from natural biological materials, modern materials being synthetic, mostly polymers. Suture materials are absorbable (resorbable) or nonabsorbable (non-resorbable) and monofilamentous or multifilamentous (braided sutures), their diameter being a very important factor. Generally, fast-healing tissues and internal organs are treated with resorbable materials, while slow-healing tissues and tissues with high mechanical exposure, such as the skin and tendons, are treated with non-resorbable materials. Synthetic suture materials are absorbed by hydrolysis due to water whose molecules attack the ester linkages and break up the polymer chains. The degradation for biological suture materials as the same for synthetic suture materials begins with the breaking down of polymer chains into smaller fragments. The fragments are then phagocytized by the enzymatic action of special types of mononuclear and multinuclear white blood cells [11, 12]. The idea of using sutures as a medicament transport system is a topic of interest for modern surgery, reducing the risk of infection and inflammation. Bio PP is mainly absorbed by hydrolysis due to water, the amount of water being essential for the rate of hydrolysis reaction.

3.2 Materials

Bio PP from BioSintex is a monofilament sterile suture made of isotactic PP (iPP). The studied bio PP materials were blue and had the thickness of.

1 EP (European Pharmacopoeia) and 3.5 EP, respectively. The degradation of bio PP mesh designed for surgical treatment of urinary incontinence (white color, with monofilament diameter 0.10 mm, typical size pore 1.7×0.3 mm, thickness 0.50 mm) was studied.

3.3 DSC analyses.

Measurements have been made with the Calorimeter 200F3, Maia, Netzsch-Geratebau GmbH with a measuring cell: gas flow control device (N_2); sealing press; heating rate $10^\circ\text{C}/\text{min}$, sample mass approximately 10 mg, sample pan aluminum. Temperature range was -30 to 200°C . One scan was used in DSC analysis. The crystallinity determination was performed using analysis software Netzsch TA—Proteus.

3.4 Preparation of solutions: methods

Perspiration consists of water, minerals, lactate, and urea. On average, the mineral composition is sodium (0.9 g/l), potassium (0.2 g/l), calcium (0.015 g/l), and magnesium (0.0013 g/l). We prepared a sodium solution in water with a concentration of 0.9 g/l. A water solution was prepared in which beechwood was introduced. This is because experimental measurements have shown the existence of bacteria in beechwood. In these experiences there are two categories of degradation media: water and salt or beechwood solutions in water. In both cases we made measurements by introducing samples 1, 2, 3, 4, ..., 40 days, ..., 60 days in the two categories of environments. In the water important changes appeared 42 days, while in the solutions with salt, changes appeared after 4 days.

3.5 Results

For bio PP blue, thickness 1 EP, which has not been degraded, the initial crystallinity of the material is 55.91%, the melting point is $T_m = 168.7^\circ\text{C}$, and the corresponding value is 1.247 mW/mg. DSC curve for blue bio PP, thickness 1 EP, kept for 6 days continuously in water shows that there are no fundamental changes in this case: the melting temperature, T_m , remains the same, and there is a small decrease from 1.247 mW/mg to 1.154 mW/mg. There is a very low increase in crystallinity: from 56.40 to 56.87%. What does this little increase in crystallinity show? It is known that the penetration of the water takes place initially in the amorphous area, it destroys by the phenomenon of hydrolysis and then enter in the crystalline area. For bio PP, after 6 days, the water entered the amorphous zone and destroyed a part of it. This shows that suture materials studied retain practically their mechanical characteristics.

Measurements made on white meshes from bio PP and DSC characteristics show changes identical to those that occur in iPP. The crystallinity of the meshes was 52.24%, melting temperature was 170.5°C , and has the value of 1.044 mW/mg. The bio PP meshes originally introduced for the repair of the hernia are currently used in several anatomical sites. From millions of meshes implanted annually around the world, more than 10% are removed due to complications. The fundamental question of degradation of PP in vivo has not been elucidated [13, 14]. We consider

that in vivo there are a number of factors such as acid solutions, salted solutions, and solutions with bacteria and enzymes that produce a strong destruction of PP. As will be seen from the analyses, the salt solutions destroy bio PP in a short time, affect the crystalline system, and consequently cause the destruction of the material. Substances that are removed can affect the body and cause complications. According to the data from the literature the degradation products are acids and alcohols from PP degradation due to the action of the water [11].

Figure 2 shows DSC curve for bio PP blue, thickness 3.5 EP, which has not been degraded, and the DSC curve for the same material that was kept in water for 42 days. There is a very slight increase in crystallinity in this case by degradation: from 63.08% to 63.84%. There is a very low melting temperature decrease from 165.8°C to 165.7°C but also a very small increase from 1.051 mW/mg to 1.15 mW/mg. What is most important in this case is the appearance of a second additional maximum melting at 154.8°C, indicating a second important type of crystalline lamellas that appeared due to the material degradation. The lower melting temperature indicates that these lamellas melt more easily.

To study the influence of perspiration on bio PP sutures, samples have been kept in sodium solution in water for 4 days. In this case there are more important changes in the melting temperature and in the decrease of crystallinity (**Figure 3**).

The discontinuities that occur are due to the melting of various crystalline lamellae at different temperatures, indicating a degradation of crystalline domains. As shown in **Figure 3**, there are several Flory components, each of these components having a single active crystallization center. The T_m values as well as crystallinity decrease. The melting point decrease may be related to the existence of chain branches. Below T_m , the amorphous regions alternate with regions which are lamellar crystals. The amorphous regions contribute elasticity, and the crystalline regions contribute strength and rigidity.

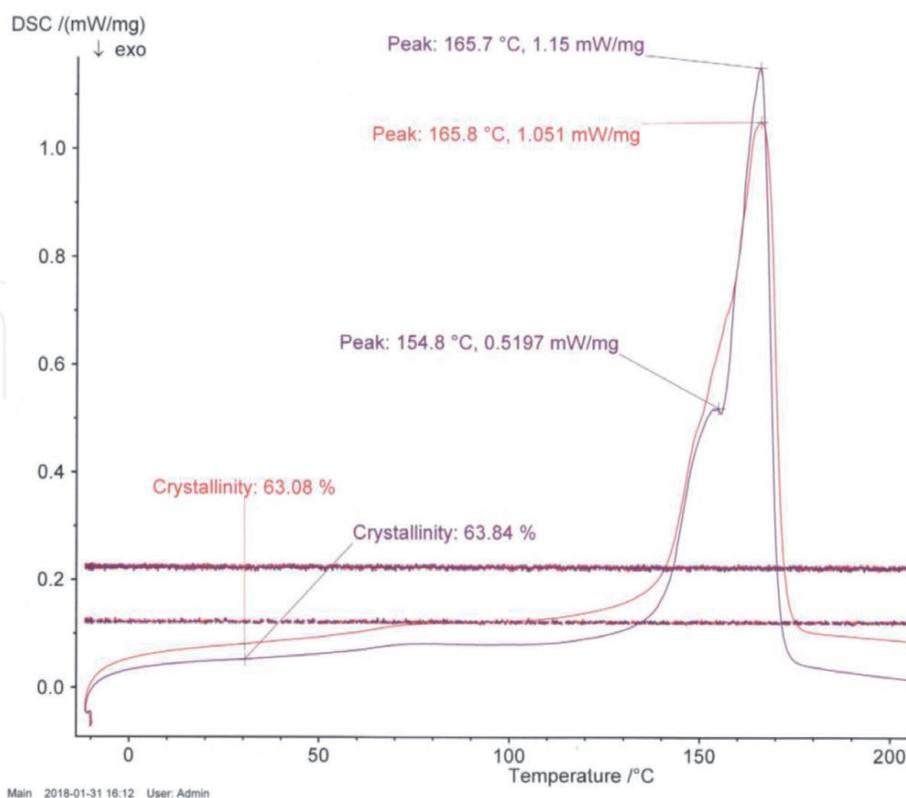


Figure 2.
DSC dependencies for non-degraded blue bio PP, 3,5 EP, (red color) and blue bio PP, 3,5 EP, kept 42 days continuously in water.

For DSC curves of bio PP, 3.5 EP, which was kept for a longer time in water, 60 days, melting point discontinuities and an increase in crystallinity are observed (Figure 4).

Outside the main peak at 168.6°C, a very close maximum is observed at 164.7°C, the value 1.325 mW/mg being higher than those obtained for non-degraded material. An increase in crystallinity was obtained: from 63.08% to 66.40%. As was observed in analyzes, bio PP fibers show very good water resistance being considered as high-performance materials in this field. This confers a high thermal resistance keeping the elasticity and mechanical resistance characteristics at

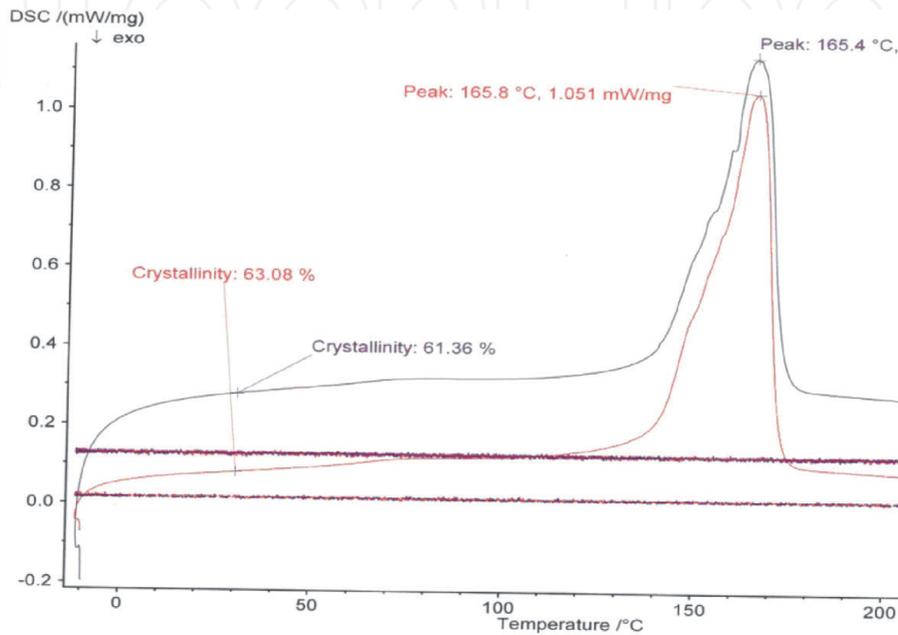


Figure 3. DSC curve for blue bio PP, thickness 3,5 EP, (red color) and DSC curve for blue bio PP, 3,5 EP, kept continuously 4 days in a sodium solution in water (mauve color).

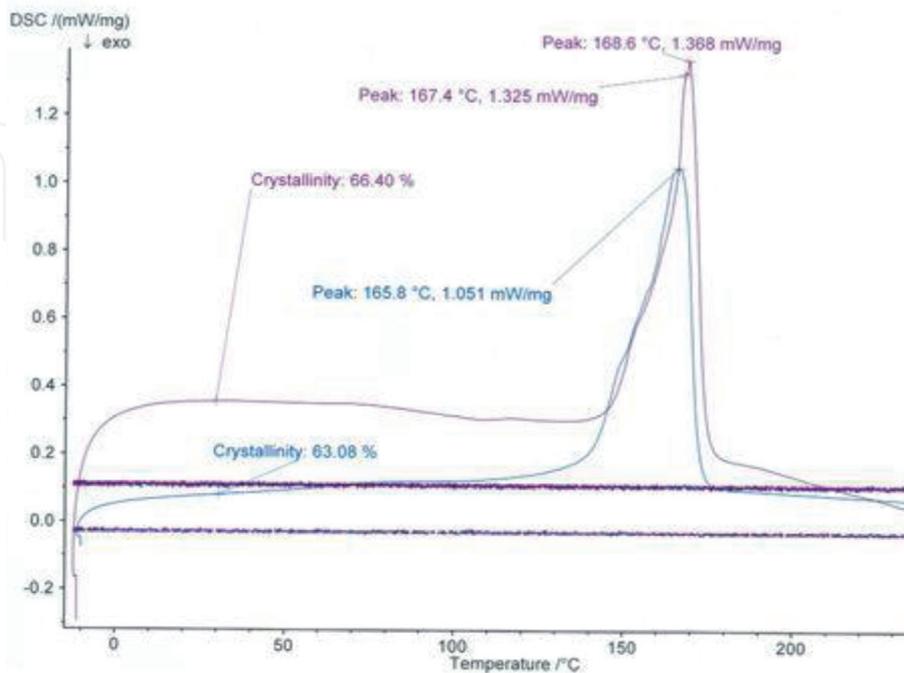


Figure 4. Comparison between DSC curves for blue bio PP, 3,5 EP undegraded, (blue curve) and the blue bio PP, 3,5 EP, 60 days in water (mauve curve).

temperature up to 90°C and good resistance to acids and alkalis. The polymer is depolymerized by the conversion of the polymer chains into a monomer or mixture of monomer, oligomers, dimers, etc. Regarding the samples that were introduced into the water with beechwood, reduced degradation was obtained compared to those introduced only in water.

At this time, sutures with PP and PE composite filaments allow the transport of drugs for different treatments.

4. Effect of powders of iron on the properties of isotactic polypropylene

4.1 Composites for biosensors

Early diagnosis of cancer is crucial for successful treatment of this disease. Highly sensitive methods are needed for measuring cancer diagnosis markers present at very low levels during early stages of the disease [15]. Major progress in the field of nanotechnology for the creation of small integrated and reliable nano-transducers in combination with biological detection elements has revolutionized the field of biosensors in the last decade. Such biosensor systems allow obtaining biochemical and biophysical signals associated with a particular disease at the level of a single molecule or cell. At present, cancer can be diagnosed mainly by monitoring certain antigens in the blood stream or other fluids of the human body or by histological examination. Modification of electrodes with nano-materials is a major interest for science and nanotechnology because they can be used in different fields in this regard. However, the immobilization strategies of biomolecules are a challenge because their specific properties must be maintained after immobilization in order to obtain reliable sensors [16]. The sensitivity of the sensors can be increased by using nano-materials with polymeric matrix and metallic fillers. Innovative biosensor strategies would allow cancer testing to be performed more rapidly, inexpensively, and reliably in a decentralized setting.

The composite materials with polymeric matrix and metallic fillers, in particular nanoparticles, present the modification of their general characteristics. The development and continuous improvement of the technology for producing and molding the above materials is accompanied by research on their mechanical, electrical, and thermal properties [17–19]. These characteristics as well as changes in moisture absorption, glass transition temperatures, flame retardant, solubility etc. must be given special attention. Interesting and intense studies refer to these materials. It is therefore possible to replace the traditional materials with new composite lightweight materials, and their use can be practicable in many fields.

A novel pan-milling technique was developed to prepare ultrafine PP-Fe composite powders, in which the average grain size of the Fe particles attain a nanoscale level [20]. The authors of the article [21] compose a detailed review to provide a description of the recent advances in the preparation of polymer nanocomposites via mechanical milling.

Mechanical properties of metal-PP polymer matrix composites were investigated experimentally [22]. High-density polyethylene (HDPE), PP, and polystyrene (PS) were used as polymer matrix, and Fe powder in 5, 10, and 15 vol% was used as metal. The modulus of elasticity, yield and strength, elongation, Izod notched impact strength, Shore D hardness, and fracture surfaces of the composites were determined. Addition of 5 vol% reduced Izod impact strength and increased the modulus of elasticity. Thermal stability of polymer nanocomposites prepared by melt mixing of PP with organo-modified clay and maleic anhydride-grafted PP has been studied [23].

Electrical properties of PP-Fe-modified and PP-Fe-nonmodified powders were studied [18]. The phenomenon of percolation and the increase of the electrical conductivity were obtained by two methods of preparation: ball milling and extrusion route. The extrusion resulted in a good dispersion of Fe particles in the PP matrix.

4.2 Materials

The polymer used as matrix was isotactic polypropylene (Tipplen). Conductive reinforcements used were two types of iron powders from Nanografi LTD. Histogram granulometric distribution of iron powder 50 nm showed the existence of a continuous distribution of particles with a maximum around 140 nm and a sharp decline of the number of particles after this value [24]. From SEM analysis for Fe powders, it was found that the mean particle size was 82.2 nm (calculated from eight measurements) for the first powder of 50 nm and the mean size (calculated from 6 measurements) of the particles of the second powder of 800 nm was 2695 nm [24, 25].

4.3 Sample preparation

PP composite samples with iron nano-powders were prepared by extrusion. Samples were obtained in two stages. In the first step, the components (PP and iron powder) were blended for 1 h in a Turbula T2F cylindrical mixer with 1.3 l mixing bowl and with clamping device from rubber, having a rotation speed of 40 rpm. By extrusion (Brabender Ketse laboratory extruder) the composite granules were obtained. In the second step, the composite granules were injected (Dr Boy A35 injection machine); thus the samples disk shaped are obtained with a diameter of 30 mm and a thickness of 2.5 mm. Polymeric composite materials with increasing content of powders (3, 5, and 8%) were prepared.

The working parameters on the extruder were extruder screw speed (45 rpm) and power funnel screw speed (700 rpm). The temperatures on the extruder heating areas in five zones were 170, 175, 180, 190, and 200°C.

4.4 Experimental

4.4.1 Mechanical tests: microindentation hardness and Shore hardness testing

Indentation hardness tests are used in mechanical engineering to determine the hardness of a material to deformation. In microindentation hardness the testing of materials is made with low applied loads, typically of 2N (roughly 200 gf). The Vickers (HV) hardness and Young's (E_{IT}) module were determined by instrument microinjection tests according to ASTM E2546-15 and the Oliver-Pharr calculation method. Mechanical tests were performed at ambient temperature, relative air humidity $35 \pm 5\%$, with Micro-Combi equipment.

4.4.2 Thermal conductivity tests

Thermal diffusion measured between 25°C and 95°C was determined using a "flash" method (ASTM E-1461: 2007), with a LFA 447 NanoFlash (Netzsch). As a radiation source, a xenon performance lamp was used, and the irradiation time on the front face of the sample was 0.18 ms. Samples were analyzed three times at each temperature. The increase in temperature on the other surface of the sample was measured with an InSb-type infrared (IR) detector. The sample analyzed had

cylindrical shape $\varnothing = 12.7$ mm and height $h = 1.95$ mm, in pressed state, and was analyzed at 25°C. Samples were grafted into suspension. Working conditions are as follows: the data acquisition software used was Nanoflash, temperature/°C was 25.0, analysis type was single layer, the analysis software used was Proteus LFA, and the analysis method used was Cowan model.

4.4.3 Electrical conductivity tests

It is known that the thermal conductivity and the electrical conductivity are in a close correlation. The dielectric tests are performed by the Solatron IL MAv-0.6 269 ... + 400°C impedance analyzer; temperature rise rate (heating/cooling) was 0.01–30°C/min; thermal stability was max ± 0.01 °C; frequency domain was 10 μ Hz ... 20 MHz; thermal stabilization was max. 8 min; the data acquisition software used was Smart. Tests were conducted in air at 3 V. The samples had a diameter of 30 mm and an average thickness of 4.3 mm. The electrical conductivity in alternating electrical current was determined.

4.4.4 DSC analysis

The conditions of measurements are described in Section 3.3.

4.4.5 SEM analysis

Electron beam scanning microscope with field emission source and ion-focused beam was used. The images were made at 1 or 2 kV acceleration voltage with a very large lens approach. The detector used was that of the Everhart-Thornley secondary electron with the Faraday cup, resulting in micrographs that highlight the morphology and topography of the analyzed surfaces.

4.5 Results and discussions

4.5.1 Analyzing the situation of reinforcement of the polymeric materials

Analyzing the situation of reinforcement of the polymeric materials with different filler materials from **Table 1**, it could be concluded that the use of dispersing fillers would lead to a decrease in the composite resistance compared to the non-filler polymer. This observation is susceptible to interpretations, because **Table 1** shows in all cases an increase in the modulus of elasticity E_{IT} , the energy of the reversible elastic deformation $W_{elastic}$, and the yield of the elastic zone η_{IT} (%), which indicates an improvement in the elastic characteristics. The addition of rigid filler particles produces an increase in the modulus of elasticity which is proportional to the volume percentage of the filler. The worsening of mechanical properties can be attributed to the occurrence and development of cracks in the vicinity of the filler particles and the formation of agglomerates of filler material. These agglomerates are initiated by the voids resulting from the partial detachment of the matrix.

Shore durometer scale D was used to measure the hardness and to characterize the penetration resistance of a penetrator in the composite materials studied. The obtained results are presented in **Table 2**.

All the materials studied have a higher Shore hardness than pure iPP. It is noted that the higher Shore hardness values are obtained for the composite iPP-Fe 82.8 nm.

	H_{IT} (MPa)	HV	E_{IT} (GPa)	h_{max} (μm)	$W_{elastic}$ (μJ)	W_{total} (μJ)	η_{IT} (%)
iPP	114.7 ± 17.1	10.8 ± 1.6	1.4 ± 0.16	23.7 ± 0.5	2.4 ± 0.9	8.3 ± 0.1	28.6 ± 1.1
iPP with 8% Fe 2695 nm	104.3 ± 14.1	9.8 ± 1.3	1.4 ± 0.07	23.9 ± 0.9	3.2 ± 0.1	8.7 ± 0.1	37.0 ± 0.1
iPP with 3% Fe 82.2 nm	110.7 ± 5.6	10.4 ± 0.5	1.4 ± 0.01	23.5 ± 0.1	3.1 ± 0.4	8.8 ± 0.01	34.9 ± 5.2
iPP with 8% Fe 82.2 nm	115.8 ± 9.6	10.9 ± 0.9	1.5 ± 0.07	22.9 ± 0.2	3.0 ± 0.4	8.8 ± 0.1	34.4 ± 4.9

Table 1.

H_{IT} is indented hardness HV is Vickers hardness E_{IT} is elastic modulus h_{max} is the maximum depth of penetration $W_{elastic}$ and W_{total} are the energies of the reversible elastic deformation and the total deformation, respectively and η_{IT} (%) is the yield of the elastic zone.

iPP	Powders (%)	iPP with Fe 82.8 nm	iPP with Fe 2695 nm
55.1	3	67.2	56.1
	5	70.0	62.5
	8	72.8	65.2

Table 2.
 Shore hardness scale D for the composite materials studied.

λ , W/(mK) iPP	Powders (%)	λ , W/(mK) iPP with Fe 82.8 nm	λ , W/(mK) iPP with Fe 2695 nm
0.127	3	0.127	0.14
	5	0.129	0.145
	8	0.134	0.14

Table 3.
 Dependence of the thermal conductivity (λ) on the concentration of metallic powders.

4.5.2 Thermal conductivity

Thermal conductivity, λ , characterizes the ability of a material to transmit heat when subjected to a temperature difference (**Table 3**). The thermal resistance is mainly caused by phonon scattering processes, including phonon-phonon scattering, boundary scattering, and defect or impurity scattering [26, 27]. In composite materials, phonon scattering is mainly due to the existence of an interfacial thermal barrier, which results from the acoustic mismatch or the damage of surface layer between the filler and polymer matrix. In order to lower the thermal resistance or enhance the thermal conductivity of the composite, measures should be taken to reduce the interfacial thermal barrier, which is closely related to the filler dispersion and the filler-matrix interaction. Many theoretical studies have studied the thermal conductivity dependence of composite polymers on the amount of metal filler or on the volume fraction of the filler, emphasizing the increase in thermal conductivity with the increase in the volume of the filler [28]. Experimental studies in the inorganic filler/polymer composites have shown that the thermal conductivity increases with the filler content, but very high filling load is used to obtain high thermal conductivity. Large amounts of filler affect and worsen the mechanical properties. Filler size and shape are important factors that influence thermal conductivity and other properties. At the same particle size, smaller particle size leads to a lower interparticle distance and more chances for the formation of the thermal conductive pathway. A significant increase in the thermal conductivity of iPP filled with combined filler, with total constant filler content of 7.5%, was highlighted [29].

The thermal conductivity values of the composites are very close. The lowest thermal conductivity is that of pure iPP and for iPP with 3% concentration of powders with size of 82.8 nm. But other observations can be made: it is observed that the largest increase in values of thermal conductivity is obtained for 8% concentration of Fe powders with size of 82.8 nm. It is very important that this composite has the best thermal endurance [25]. The thermal conductivity values show approximately the same increase for 3, 5, and 8% concentration for Fe powders with size of 2695 nm.

4.5.3 The electrical conductivity

The electrical conductivity in alternating electrical current was determined (**Table 4**). It is known that the thermal conductivity and the electrical conductivity are in a close correlation.

iPP	Powder (%)	iPP and Fe 82.2 nm	iPP and Fe 2695 nm
σ ($\Omega^{-1} \text{ m}^{-1}$)		σ ($\Omega^{-1} \text{ m}^{-1}$)	σ ($\Omega^{-1} \text{ m}^{-1}$)
1.2×10^{-4}	3	1.49×10^{-3}	1.46×10^{-3}
	5	1.53×10^{-3}	1.49×10^{-3}
	8	1.56×10^{-3}	1.53×10^{-3}

Table 4.

Dependence of the electrical conductivity σ ($\Omega^{-1} \text{ m}^{-1}$) of iPP composites with 3, 5, and 8% concentration of Fe powders for $\sim 2 \times 10^4$ Hz.

Concentration (%)	Powder size (nm)	Crystallinity (%)	T _m (°C)
iPP	—	53.46	170.4
3	Fe 2695	50.01	170.5
5	Fe 2695	49.06	170.7
8	Fe 2695	41.51	171.9
3	Fe 82.2	48.10	170.4
5	Fe 82.2	49.14	169.9
8	Fe 82.2	36.03	171.00

Table 5.

Variation of the crystallinity and melting temperatures T_m with the content and sizes of powders.

As observed from the electrical conductivity measurements, the conductivity for the frequency $\sim 2 \times 10^4$ Hz shows an increase with an order of magnitude, the highest values being at 8% concentration of Fe powders. For the investigated samples, the electrical conductivity increases (both in pure and in iPP with Fe powders) at increasing frequency. Increases of the loss factors were obtained with the increase of the metallic powder content, while the variations of the electrical permittivity were very small [30].

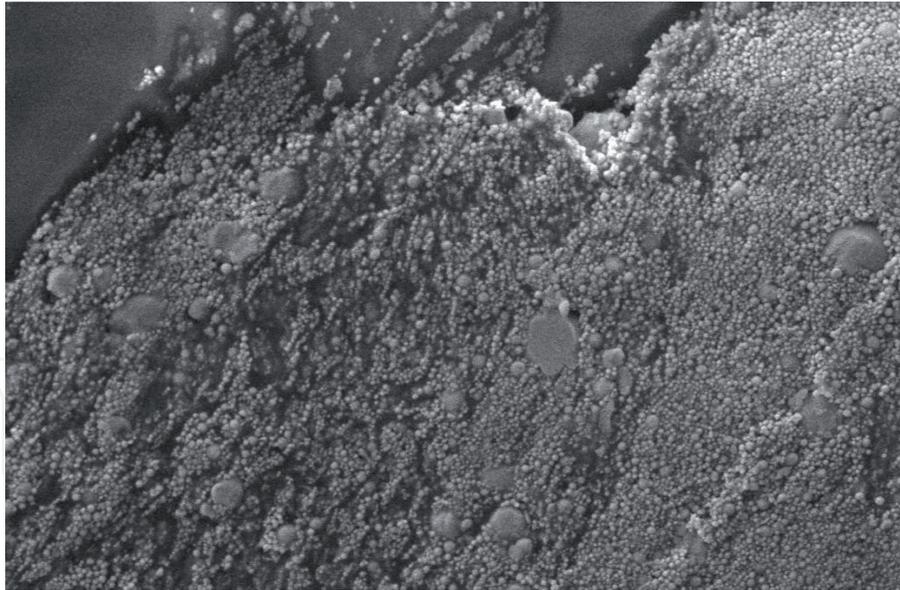
4.5.4 Incorporation of metallic powders into polymers

Incorporation of metallic powders into polymers is expected to impact the crystallization degree and the melting characteristics. DSC analyses were conducted to investigate the behavior of the studied materials. The melting temperatures and the crystallinity were determined from DSC thermograms (**Table 5**). The decrease of crystallinity with the amount of powders, if they are of the same type and are identical in size, was observed. This shows that the particles introduced into the iPP penetrate not only in the amorphous but also in the crystalline domains. The highest decrease was in all cases at 8% concentration of powder.

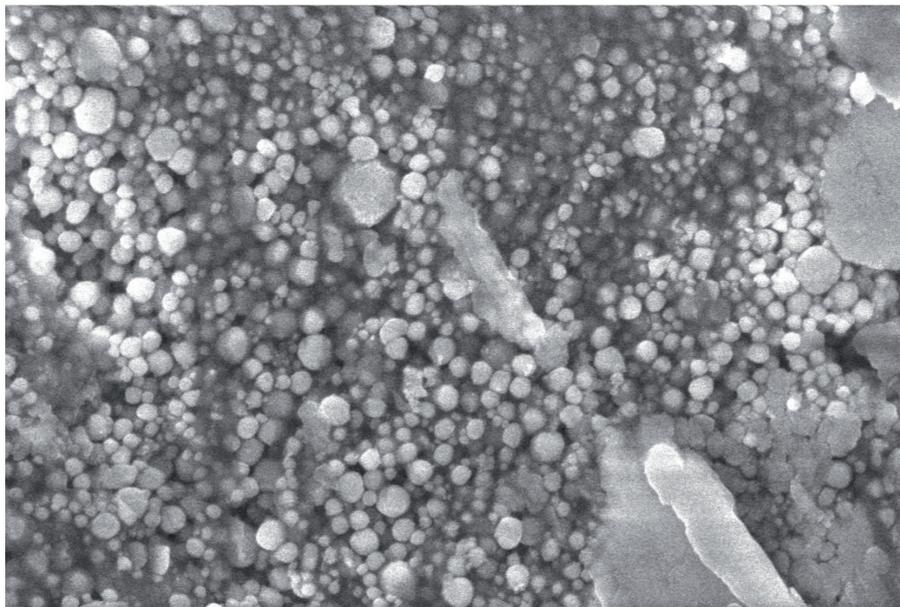
Crystallinity clearly depends on the size of the particles introduced: the larger the particles, the less they affect the crystallinity of the iPP. The decreases are highest in the case of small particles because they enter more easily in the crystalline domains. Interesting changes occur in melting temperatures. Generally melting temperature increases with the increase in powder content and at 8% concentration, temperature has the highest value.

4.5.5 SEM analysis

Figure 5(a and b) shows the morphological analyses for iPP with 8% concentration of powders with size of 82.2 nm, and **Figure 6(a and b)** shows the



a



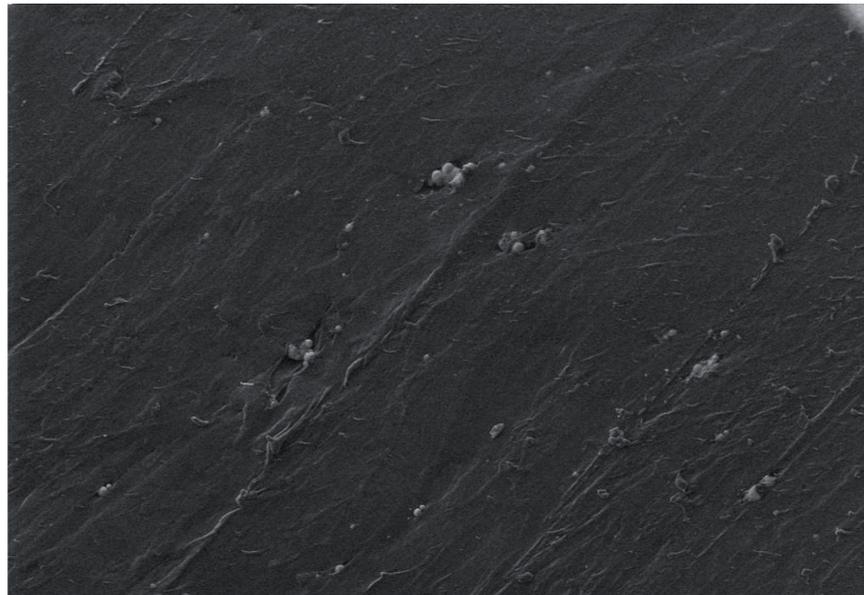
b

Figure 5. SEM analysis of iPP with 8% concentration of Fe powders with size of 82.2 nm: 20,000 \times (a) and 50,000 \times (b).

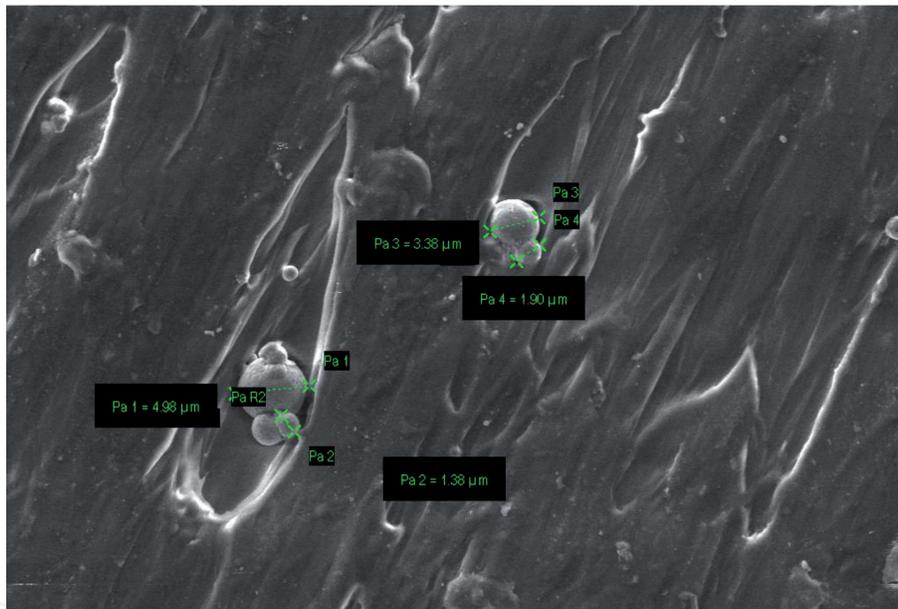
morphological analysis for iPP with 8% concentration of powders with size of 2695 nm. A relatively uniform distribution of Fe particles with size of 82.2 nm is observed. They even entered the crystalline areas.

This confirms that extrusion preparation allows good dispersion of Fe particles in the PP matrix, a fact confirmed by other authors [18]. In this case the existence of some agglomerations of particles is observed. For the most part, these agglomerates are initiated by the voids resulting from partial detachment of the matrix.

Examination of **Figure 6** shows that the larger particles with size of 2695 nm penetrate first into the amorphous zone and then into the crystalline zone. This fact is in accordance with the crystallinity values obtained for these nanopolymers. In the figure agglomeration of particles also appears, especially in the amorphous areas or in areas with defects.



a



b

Figure 6.
SEM analysis of iPP with 8% concentration of Fe powders with size of 2695 nm: 1000× (a) and 5000× (b).

5. Conclusions

Bio PP materials for sutures show very good water resistance and present more important phenomenon of degradation in the presence of water for a longer period of time, over 40 days. Bio PP shows much more severe destruction of the crystalline domains in sodium solution in water. Bio PP meshes show the same degradation phenomena as those of bio PP sutures. For the samples that were introduced into water with beechwood, reduced degradation was obtained, variations comparable to those for samples introduced only in water.

An improvement in the elastic properties of the composite materials (iPP-Fe powder) was observed. In all cases studied, the increase in Shore hardness was

achieved. The phenomenon of percolation has not been obtained. Thermal conductivity has little variations. It is noted that the highest Shore hardness values are obtained for composite iPP-Fe 82.8 nm, for 8% Fe concentration. This material also has the best thermal endurance. For applications in the field of biosensor construction, particles with dimensions of the order of nanometers are required.

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