Dielectric Spectroscopy Analysis of Composites Obtained from Thermoplastic with Feathers Flour

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***Abstract*—The article highlights the influence of poultry feather flour percentage and compatibilizers on the dielectric properties of composite materials based on low density polyethylene matrix. The behavior of composite materials was determined in terms of dielectric properties and the distribution of poultry feather flour in the basic polymer matrix, to optimize the technology and structure. The results showed that the addition of feather flour in thermoplastic matrix led to an increase in dielectric constant and dielectric loss and the presence of additives leads to better dispersion and processability of the composite material, an observation supported by X-ray analysis.**

Keywords—biocomposites; poultry feathers; coupling agents; dielectric spectroscopy; X-ray analysis

I. Introduction

Plastic materials have shaped the world by being present in our lives in an infinite range of products and applications, bringing safety, hygiene, comfort and well-being to our society. The production of polymeric materials increases from year to year, consequently, the amount of polymer waste increases, due to the wide range of applications of polymeric materials with a short lifespan. However, the challenge of today is to make the most of these material and to prevent their disposal into the environment, increasing the reuse and recycling of plastic packaging waste and contributing to the benefits of resource efficiency [1], [2].

A solution for limiting plastic waste can be to make a biodegradable polymer that retains its performance characteristics only during their use and then under certain environmental conditions or in the presence of microorganisms to decompose them. The biocomposites obtained can partially or even improve the properties of synthetic polymers.

Under these conditions, composite materials reinforced with various natural fibers have become important materials for the manufacture of industrial products. A common approach is the introduction of cellulosic fibers as a reinforcing agent in the production of composite materials, but there are other natural materials that can be used as reinforcement material in mixtures with different matrices. Fibers obtained from sheep's wool and poultry feathers can be used for reinforcement. Compared to other agricultural residues, poultry feathers are some of the most ubiquitous and inexpensive coproducts available in the world. Feathers represent up to 10% of the body weight of birds, which means that approximately 8–9 million tons of feathers are generated in the world each year. In addition to the low costs and high availability they have, the feathers of poultry have distinct and unique properties, such as low density, 0.9 g/cm^3, feathers are more lighter than other natural fibers such as cotton (1.5 g/cm^3). The feathers contain over 90% protein in the form of keratin which is useful for various applications. Despite these unique characteristics, feathers are considered waste and are dumped in landfills or used as feed for animal feed, [3].

In recent years, composite materials with improved properties have been developed using keratin and polymer matrices, especially low density polyethylene, LDPE or high density polyethylene, HDPE. The main disadvantage of the obtained composite is the weak mechanical properties due to the reduced interfacial adhesion between the components of the composite, [4].

The presence of keratin in these fibers makes these composite materials based on poultry feathers not easy to obtain. That is why compatibilizers and coupling agents are used for better processability. Keratin fibers are non-abrasive, have low density, are biodegradable, renewable, environmentally friendly, insoluble in organic solvents and have a hydrophobic behavior compared to the hydrophilic nature of the polymer matrix. These properties make this material suitable for use in structural reinforcements in polymer-based composites, [5], [6].

II. Experimental

A. Materials

Composites material were prepared using granulated recycled low density polyethylene (RLDPE) with 0.91 g/cm^3 density, 19.89 g/10min. flow index (at the 190°C temperature and with the 2.16 kg. weight), 0.23% ash content and melting point at 125-145°C.

The poultry feather flour (FF), figure 1,was use as reinforcing material agent, and it was provided by SAFIR SA Vaslui, Romania, a supplier of protein flours. The feather flour was obtained from Ross 308 chicken feathers, Fig. 1, slaughtered, through an automatic hydrolysis-sterilization-

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drying process with a humidity of max. 7%, and a fiber size of max. 3mm, Fig. 2.

Fig. 1. Poultry feather flour.

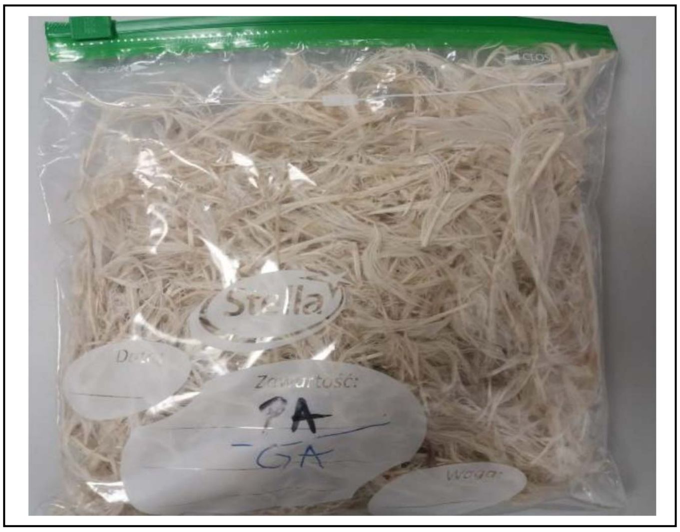

Fig. 2. Ross 308 chicken feathers.

Compatibility agents (C) were used in different percentages to increase the interface adhesion between the composite components. The following materials were included in the compatibility agent: Licowax PED 521 GR (Clariant) was used for excellent anti-sticking properties, Vistamax (ExxonMobil Chemical) was introduced for better fiber dispersion and for increasing matrix-fiber adhesion, Hostavin N30 (Clariant) was used for excellent thermal and color stability, CaCO3 was used as a filling agent and to prevent flour agglomeration during composite processing and virgin LLDPE to improve components dispersion.

Several recipes have been prepared, Table I. For each recipe 25 kg of various agents, in different percentages were mixed. The mixture was prepared in two stages into laboratory mixer with 30 kg capacity. First, additives and feather flour was mixed for 10 min. and then over the obtained mixture was added RLDPE and mixed again for 10 min. The final mixture was extruded to obtain granules on a two-stage extruder (the second stage is for thermal protecting of feathers flour).

B. Testing methods

Dielectric spectroscopy method studies the molecular response of materials when are stimulated in frequency. The test material is placed between two parallel electrodes, with 20 mm diameter, to form a capacitor. A sinusoidal signal of 1Vac value with a frequency between 10^{-2} -10^{6} Hz is applied on the material. The real permittivity (ε') and dielectric loss (Tg δ) is usually measured to determine various physical and chemical properties, [7].

X-ray analysis was performed with 1174 Skyscan tomograph to determine the dispersion of the components in the polymer matrix. This advanced technique provides 2D / 3D image insights into samples of any shape or size, of materials with higher X-ray absorption. 2D images are obtained using a 1.3 megapixel X-ray camera that allows to scan the entire volume of the sample in minutes (pixel size of 6-30 µm). In order to characterize the internal structure of materials, it is necessary to be virtual cut in 2D images, and then with a special reconstruction software the 2D images overlap to obtain the three-dimensional shape of the analyzed material.

TABLE I. RECIPES OF COMPOSITE MATERIALS WITH RLDPE/FF/C

No. of sample	**Recipes**										
	Materials	***Unit***	***I-RLDPE***	***II-RLDPE/10FF***	***II-RLDPE/20FF***	***II-RLDPE/30FF***	***II-RLDPE/50FF***	***III-RLDPE/10FF/32C***	***III-RLDPE/20FF/32C***	***III-RLDPE/30FF/38C***	***III-RLDPE/50FF/38C***
1	RLDPE	%	100	90	80	70	50	58	48	32	12
2	LLDPE	%	x	x	x	x	x	10	10	12	12
3	LICOWAX PED 521 GR	%	x	x	x	x	x	2.25	2.25	2.7	2.7
4	VISTAMAX	%	x	x	x	x	x	2.25	2.25	2.7	2.7
5	HOSTAVIN N30	%	x	x	x	x	x	0.5	0.5	0.6	0.6
6	CaCO3	%	x	x	x	x	x	17	17	20	20
7	FF	%	x	10	20	30	50	10	20	30	50

III. Results and Discussion

Several series of composite samples, based on recycled low density polyethylene (RLDPE) as the matrix with 0, 10, 20, 30, 50% of poultry feather flour (FF) as filler and additives were manufactured. Including poultry feather flour and additives in the polymeric matrix leads to changes in dielectric response of material [8][9].

From the graph of the dielectric constant of materials RLDPE/FF it is observed that an amount of addition of feathers of 10% leads to an insignificant increase of the value of the dielectric constant, compared to the reference polymeric matrix. This increase is more pronounced at low frequencies due to the interfacial polarization phenomenon. As the amount of feather flour increases, the value of the dielectric constant is 3.28 for the material with 20% FF, and for the material with 30% FF the dielectric characteristic is similar to that of the material with 20% FF, but at low frequencies the interfacial polarization it is higher. The dielectric characteristic of the material with 50% FF registers the highest values compared to the base matrix and to the other composite materials RLDPE/FF, reaching a value of 4.7. The characteristic shapes can be explained by the fact that processing and homogenizing additives was not used for RLDPE/FF composite. The graph of dielectric losses show that the material is not homogeneous and the losses increase as the amount of feather flour added is higher, Fig. 4.

The dielectric constant characteristics for RLDPE / FF / C composite materials are more uniform compared to those of RLDPE / FF composites, but they have higher values, probably due to the presence of the filling agent $CaCO_3$. Also, it is observed that for the composite material RLDPE/FF/C the compatibilizing agents have an important role in the processability and homogenization of the material. The interfacial polarization phenomenon is more pronounced due to the fact that the filler has a higher molecular mobility than the matrix, reaching for the composite material RLDPE/50FF/38FF the value of 5.55, Fig. 5. From the graph of dielectric losses it is observed that the Tg δ values for RLDPE/50FF/38C composites is higher compared to RLDPE/50FF, Fig. 6.

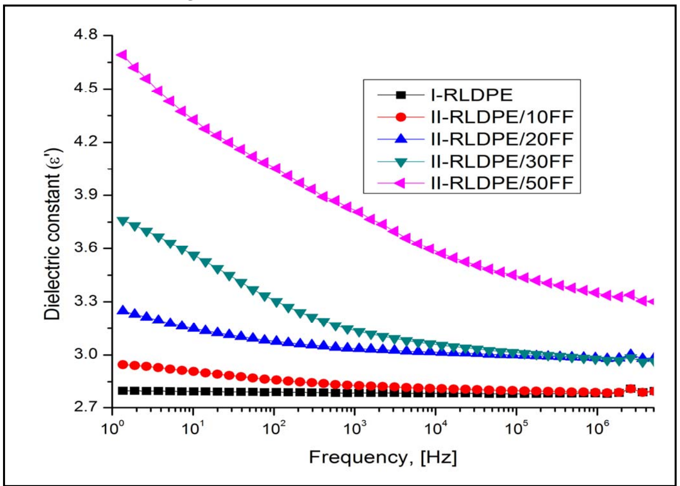

Fig. 3. Variation of dielectric constant, ε' vs. frequency for composites RLDPE/FF

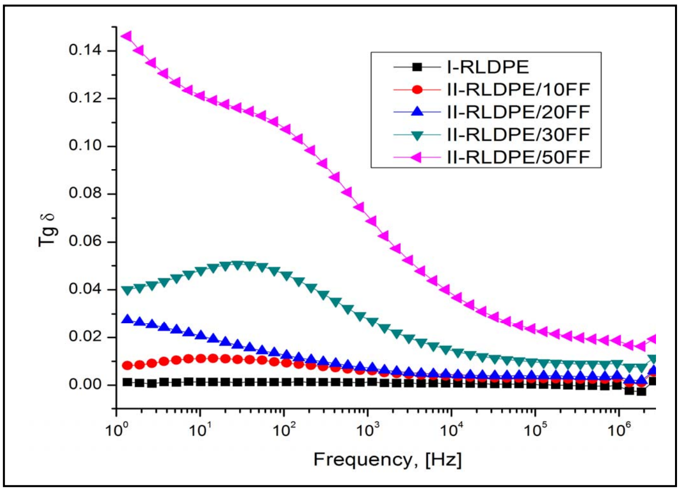

Fig. 4. Variation of dielectric loss, tg δ vs. frequency for composites RLDPE/FF

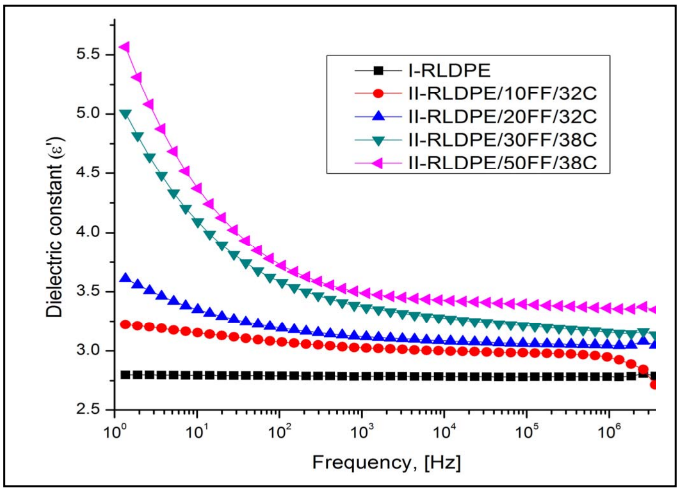

Fig. 5. Variation of dielectric constant, ε' vs. frequency for composites RLDPE/FF/C

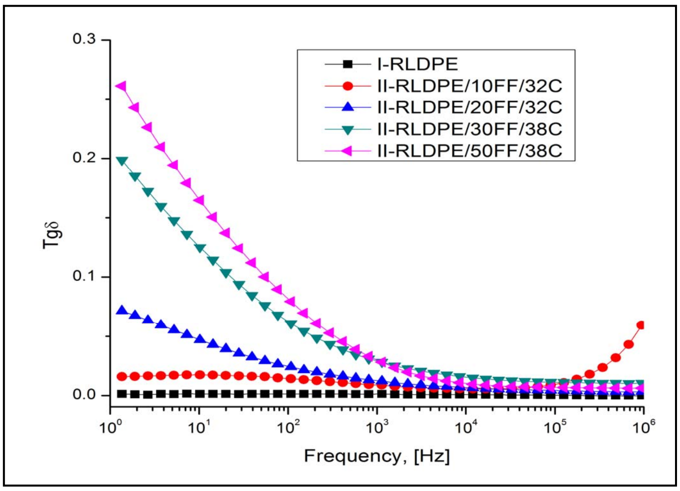

Fig. 6. Variation of dielectric loss, tg δ vs. frequency for composites RLDPE/FF/C

The characteristics of a composite material are defined not only by the basic matrix, but also by the materials used as filler and compatibilizing agents. Depending on the production process and technological parameters, different complex structures can be obtained. Skyscan 1174 with X-rays allows to obtain images from inside the composite material. Fig. 7 shows the 2D image, the binary image and the reconstructed 3D image from the 2D form of the basic polymer matrix RLDPE. In this image it is observed that the matrix does not contain filling elements and can be observed surface imperfections.

Fig. 8 and 9 present images for composite RLDPE/30FF and RLDPE/50FF, respectively, and it can be observed that the FF dispersion inside the polymeric matrix is not uniform, agglomerating during the compositing process. Also, quantitative differences can be observed between this composite materials. In Fig. 10 and Fig. 11 it can be observed images for composite RLDPE/30FF/C and RLDPE/50FF/C respectively and it can be observed that the FF dispersion inside the polymer matrix is uniform.

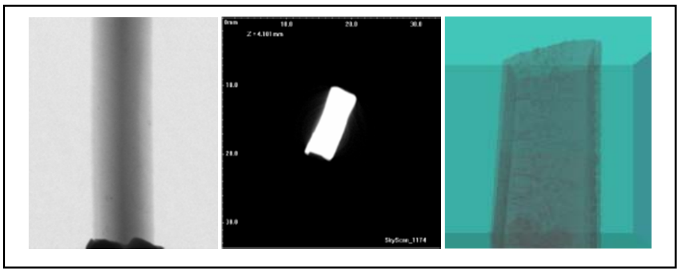

Fig. 7. Xray image for RLDPE

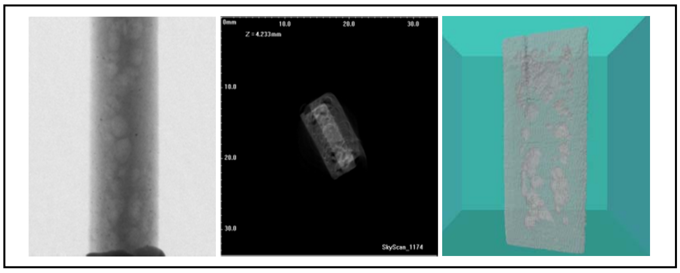

Fig. 8. Xray image for composite RLDPE/30%FF

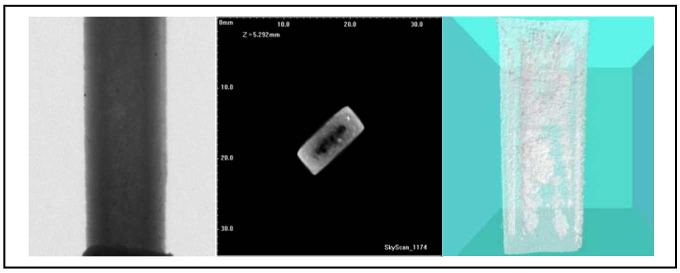

Fig. 9. Xray image for composite RLDPE/50%FF

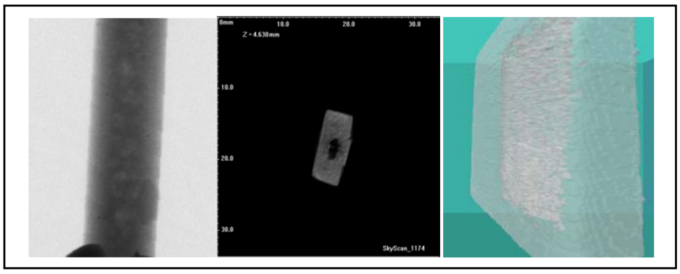

Fig. 10. Xray image for composite RLDPE/30%FF/32C

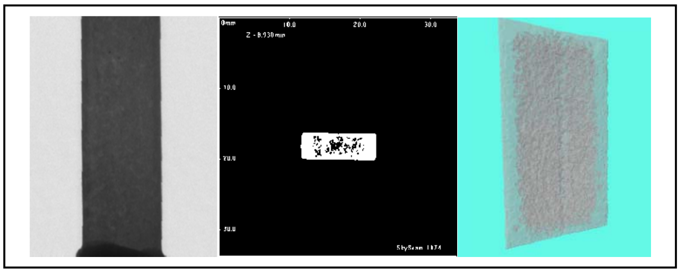

Fig. 11. Xray image for composite RLDPE/50%FF/38C

IV. Conclusions

Introducing natural polymer causes significant changes on dielectric properties. Increasing the percentage of FF leads to an increase in the values of the dielectric constant and the loss factor. The interfacial polarization phenomenon is more pronounced at low frequencies for composites materials RLDPE/50FF/38C. By introducing processing additives, the agglomeration of the flour during the composite processing is prevented and the structure of the material becomes more homogeneous, which is observed by X-ray analysis and probably the mechanical properties are improved. Future research will focus on the characterization of mechanical properties and the identification of potential applications of these materials.

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