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Preliminary Studies Composite of The Experimental Near Percolation Pvdf Nickel With Particle Obtained Physics Mixture

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Abstract: *Conductive composites in which they associate the electrical properties of metallic particles with the mechanical properties and processability of conventional polymers has been intensively investigated for many years precisely because of the versatility and potential of technological applications that they can present. In this work composites were easily obtained by physically mixing the PVDF thermoplastic and the nickel particles and homogeneous films were obtained by hot pressing at a temperature of 180 ° C and 30 MPa. The electrical conductivity of PVDF / Ni of the composites studied as a function of the content of Nickel particles presented percolation threshold between 15 and 20% with a jump of 7 and 5 orders of magnitude, respectively.*

Keywords: *PVDF, Nickel, Physical Mixture Electrical Properties*

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I. Introduction

For some time the idea has been to associate the good mechanical properties of the polymers with the electrical properties of the metals with the incorporation of conductive charges. These materials are called extrinsic conductive polymers. Even with the advent of conductive polymers, the incorporation of additives through physical mixing or solution of conductors of electricity in a certain matrix as in an insulating polymer, allows to modify the conductivity of the polymer matrix, which potentiates its application and until today has great technological appeal [1,2].

The properties will depend on some factors such as: the chemical structure and concentration of each constituent; the interfacial interaction between matrix and additive; and the morphology of the mixture and even preparation conditions of the material. The final properties of these new conductive composites are influenced by the size, shape, properties and surface area of the incorporated conductor, the dispersion and distribution of the particles of the dispersed phase, and especially of the concentration and compatibility between the present phases [3,4]

The proper choice of the incorporated additive and the polymer matrix exerts influence on the properties of the composite [5-11]. The most commonly used materials are carbon black [8], nickel [9], fibers [10,11] among others, even intrinsically conductive polymers, already synthesized, are used as conductive additives in insulating polymer matrices19. such as carbon black, fibers, graphite, vitreous carbon, metallic particles or a mixture of metal oxides and among many others [12]. Factors such as the volume or mass fraction of the components of the composite must be considered, since it is from a given concentration of the incorporated material that we will have an increase of its electrical conductivity. This occurs because the increased concentration of the incorporated material creates conductive paths or conducting networks within the matrix. This critical concentration is called the percolation threshold and is explained by the percolation theory.For concentrations below the percolation threshold, the conductivity of the composite is equal to the conductivity of the polymer matrix. From a specific critical amount of the conductive particle, for example, there is a significant increase in the electrical conductivity of the composite.

However for this study of electrical conductivity the theory of mechanisms and how it occurs is necessary, however the goal of this work is not to approach the models of electrical conduction, but to estimate the moment in which it can occur. The possible paths of current in these conductive composites can be easily estimated by means of statistical modeling.

Two types of percolation can be considered initially in these models, in this one the two-dimensional material with (1) empty spaces, the separated sites are considered in equal distances, in this way there will be an equal probability of, when inserted a conductive load fill one site and a second (2) of the link established by these neighboring sites, now filled.

In both cases the conductivity will occur when a sufficient amount of particles disperse along the matrix forming a path without interruptions to current. At this moment this probability value, until then the critical probability will be the determining value for percolation to occur. In a given region, known as the percolation region, the particles of the conductive additive are increasingly close or in contact, forming a conductive three-dimensional network in the matrix, resulting in a substantial increase in the electrical conductivity of the material.

Some papers report that the percolation threshold should preferably be as small as possible to preserve the mechanical properties of the polymer material, for example, not to hinder processing and not increase the cost of the final material and even the abrasive character of the particles within the matrix.

One of the polymers used as a matrix in the preparation of conductive composites [13] is Poly (Vinylidene Fluoride) - PVDF, because it is easily processable in highly flexible films and exhibits excellent mechanical, optical, thermal properties and also because it is highly resistant to the attack of chemicals [14].

This work presents a study of obtaining the percolation threshold for PVDF composites with nickel particles of samples obtained by means of physical mixing of the powders

II. Experimental Procedure

2.1 Materials

The semi-crystalline thermoplastic poly (vinylidene fluoride) -PVDF from SOLEF 1008/1001-Solvay fluoropolymers in powder form and the nickel particles (<5μm) purchased from Sigma-Aldrich were used as received. The incorporation of nickel particles into the PVDF matrix occurred through a physical incorporation through the blending of the two powders, that is, adding the metal particles to the polymer. The resulting material was pressed at a temperature of 180° C under a pressure of 30 MPa between two Kapton sheets. The average thickness of the samples obtained was approximately 140 μm.

2.2 Characterizations.

Electrical conductivity measurements were performed by applying a voltage (V) to a Keithley model 236 digital programmable voltage and current source measuring current (I). The conductivity (σ) was obtained using equation 1. With l being the thickness of the sample and A, the area of the metalized surface on both faces with a value of approximately $A=5.0x10^{-5}$ m².

$$\sigma = \frac{l}{A} \cdot \frac{i}{V} \qquad (1)$$

The tensile tests were performed on an equipment of the brand Instron-3369, performed at room temperature, with a deformation speed of 13mm/min in a 500 N load cell. Tie tests were performed according to ASTM D882 standards on samples of pure PVDF, PVDF particle incorporation, and incorporation of 5, 15 and 25% of nickel particles, respectively. Five test specimens were tested for each sample, which were kept under vacuum for moisture control for 24 hours before the tests.

The micrographs of the samples in film form were obtained in a The X-ray diffractograms were obtained in a diffractometer of the brand SHIMADZU model XRD-6000 with wavelength characteristic of copper Kα-Cu equal at 1.54Å.

III. Results

Figure 1 shows the micrographies of the PVDF/Ni composites. We can observe that for larger amounts of particles (15% and 25%) we observed a more homogeneous distribution, the formation of some agglomerates, besides some imperfections caused by the incorporation of these particles. The figure 2 shown patterns composite. Observe the characteristic peaks of the PVDF phase α in 2θ equal to 17.3°, 18.3°, 19.9°, 26.5°e We can verify characteristic peaks of the nickel particles [ICSD 4-850] in 2θ equal to 44.5°, 52.89 °, 76.43 °, 92.95 and 98,46°.

The stress-strain curves for pure PVDF films and PVDF films with incorporated nickel particles are shown in Figure 3. We can verify the influence of the particles and their abrasive character inside the matrix by decreasing the deformation with increasing quantity of particles when compared to pure PVDF. The presence of the particles caused a decrease in the tensile strength values from 55.5 MPa (pure) to 37.8 MPa (sample with 25% nickel particles). Regarding the modulus of elasticity, the presence of the particles caused an increase of 1.74GPa (pure PVDF) to 2.04GPa (25% of nickel particles).

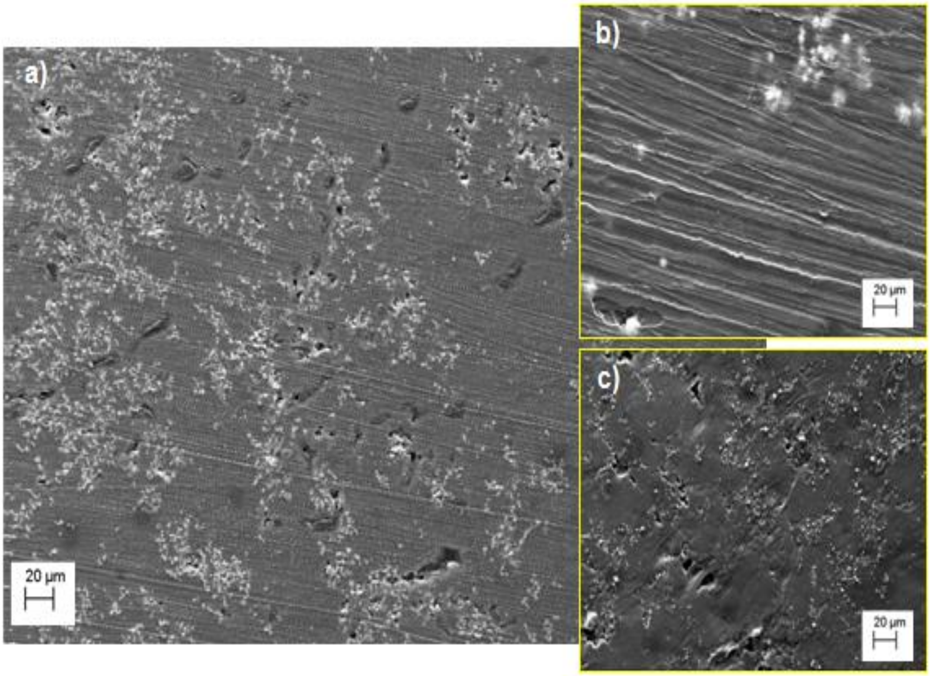

Figure 1 SEM micrographs of PVDF film with 25% nickel particles (500x) and highlighted (5KX).

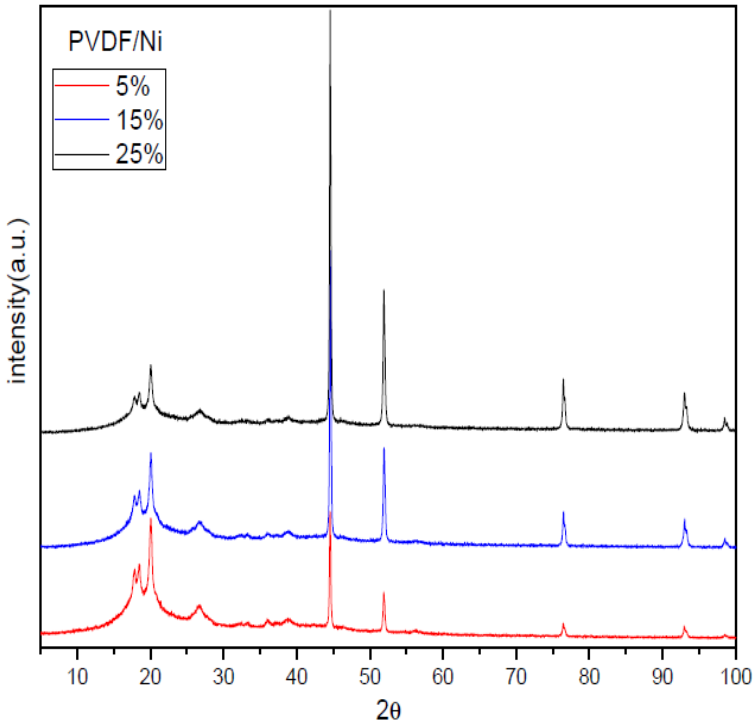

Figure 2 PVDF/Ni x-ray Diffractograms.

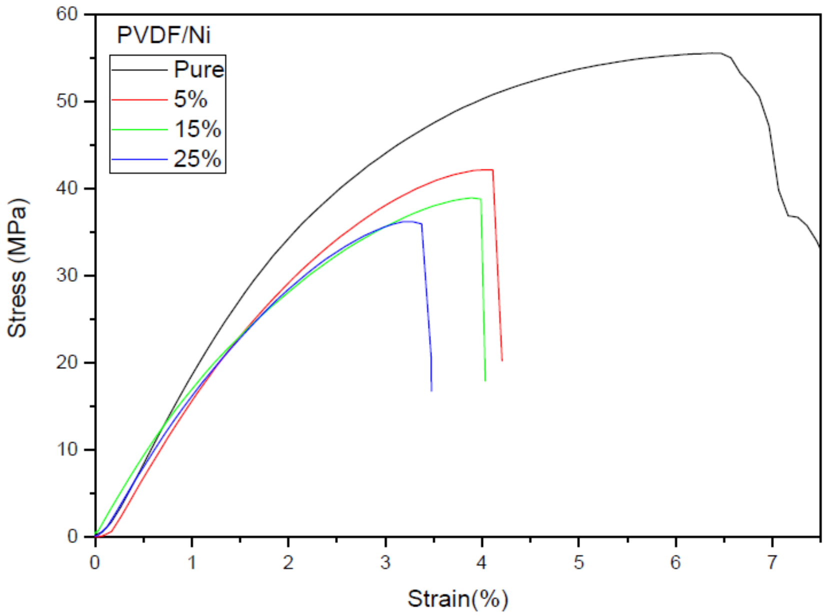

Figure 3 Tensile tests of pure PVDF and PVDF/Ni films with 5, 15 and 25% nickel particles incorporated.

This result indicates that the nickel particles are acting in the matrix as a reinforcement by limiting the movement of the polymer chains and increasing the tensile strength and the decrease of their deformation, which results in impoverishment of the mechanical properties.

Figure 4a illustrates the behavior of the conductivity achieved for the individual nickel particles and for the PVDF/Ni films are shown in Figure 4b. It is found that at a nickel concentration below 15% the conductivity value increases slowly, which suggests that there are few interconnections between the particles. For higher concentrations the conductivity jump is caused by the geometric percolation, in other words by geometric contact of the nickel particles [3,15,16]. Above this concentration, between 15 and 20%, the conductivity presents a percolation threshold that results in an increase of seven (7) orders magnitude reaching a saturation value exclusively of the nickel particles (10^{-4} S/cm) (Fig. 4a).

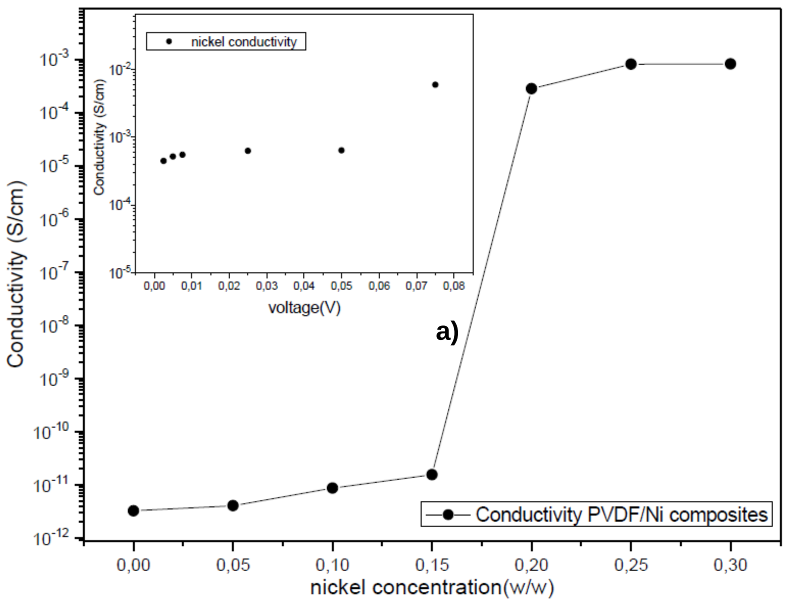

Figure 4 (a) Electrical conductivity of nickel particles and (b) Electric conductivity of the films as a function of the percentage of nickel particles incorporated in the pure PVDF.

b)

Although the conductivity of extrinsic composites has already been intensively investigated physical mixing can be shown an easier route when it is intended to forge the properties of the material. even with low mechanical properties, conductivity permitting values close to the semiconductors, allows the composites to be made according to the desired conductivity and the definition of their applications, such as electronic components.

IV. Conclusion

The work developed allowed to obtain films of the PVDF composite with incorporated nickel particles from the physical mixture. The results of SEM showed a morphology that the composite of PVDF / Ni with 5% of particles was observed a random distribution of the particles. For 15 and 25% of nickel particles a more homogeneous distribution was observed along the surface of the material, verifying the formation of a lattice, which indicates the construction of interconnected conducting paths for the composite with 25% of particles.
The results of tensile tests as expected evidenced that the presence of nickel particles leads to large losses in the mechanical properties of the composites with the reduction of the breaking strength and the deformation itself. Regarding the electrical conductivity obtained experimentally, it is verified that the PVDF/Ni composite presented a percolation threshold between 15-20% with an increase of 7 and 5 orders of magnitude, respectively, reaching values of the order of 10^{-4} S/cm of the added charge only.

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