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Morphological and thermal evaluation of blends of polyethylene wax and paraffin

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ABSTRACT

The thermal behavior and the morphology of blends of polyethylene wax and paraffin were investigated to evaluate the feasibility of using these materials to obtain a new temperature-indicating device to use in order to indicate failures in electrical connections due to overheating. The samples were evaluated with differential scanning calorimetry (DSC), X-ray diffraction (XRD) and dynamic mechanical thermal analysis (DMTA). The results showed that the crystallinity decreases as the concentration of polyethylene wax increases. In the compositions tested, the components were not miscible in the crystalline phase, and these compositions exhibited solid/liquid transitions at temperatures between those of the individual components.

1. Introduction

In power transmission and distribution networks there are numerous connections of wires, cables and electrical equipment terminals. If one of these connections fails, the supply of electrical power can be compromised in certain circumstances.

Predictive maintenance of these electrical connections is usually performed by means of temperature monitoring. An increase in the electrical resistance of a connection results in the dissipation of energy by the Joule effect, causing a change in the temperature.

As part of their maintenance programs, power companies often monitor connections with thermal imaging. A survey of the maintenance history of a power company was performed to identify the temperature range that typically indicates overheating and failure in a connection. It was found that most failures occur between 60 °C and 100 °C; few failures were recorded at temperatures less than 60 °C or greater than 100 °C.

Currently, thermal imaging is used to monitor connections. However, there are several intrinsic uncertainties to this method [1,2], and these uncertainties can often result in an incorrect diagnosis or prevent detection of the defect. Commercial thermochromic materials were tested but they fail when used outdoor.

A new temperature-indicating device capable of detecting failures in electrical connections due to overheating was developed. This device relies on melted wax, which when mixed with a color pigment indicates that the connections are overheating. The waxes selected have melting temperatures close to the low and high temperatures indicating connections failures. Furthermore, they have a low cost. For the sensor to be able to detect various temperatures, different wax blends were used. A prototype of the sensor was developed, and an application for a patent has been filed with the INPI, number BR 10 2013 021700 0, "Thermosensitive sensor for monitoring overheating of electrical connections" ("Sensor termosensível para monitoramento de sobreaquecimento em conexões elétricas").

Waxes encompass a wide range of substances that may be of organic, natural (animal, vegetable or mineral) or synthetic origin [3]. The wax group includes paraffin and polyethylene wax.

Paraffins are saturated hydrocarbons obtained from petroleum fractions and usually consist of a mixture of different alkanes with between 18 and 55 carbon atoms in a chain. Paraffin wax is marketed in several grades [4] that differ in their melting points, generally between 30 °C and 90 °C. The greater the number of carbon atoms in the chain, the greater the molecular mass and consequently the higher the melting temperature will be [5–7].

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Polyethylene, both the wax and the polymer, is formed from ethylene. The difference between the polymer and the wax is in the number of repeat units in the ethylene monomer, which directly affects the molecular mass. Polyethylene wax has a molecular mass between 200 and 1000 g/mol, whereas the molecular mass of the polymer is between 10,000 and 6000,000 g/mol [8].

This study examines the morphology and the thermal behavior of various compositions of polyethylene wax and paraffin to evaluate the feasibility of using these materials as temperature indicators for electrical connections in power distribution networks.

2. Experimental procedure

2.1. Materials

The two types of wax used in the tests were polyethylene and paraffin. The polyethylene wax, (WAX 2000, AMC do Brasil), has a viscosity of 80 SUS (approximately 15.6 cSt) at 120 °C. The macrocrystalline paraffin wax (PARAFINA BR 140/145 °F—1% and 2% oil, Petrobrás) has a viscosity of 6.01 cSt at 90 °C.

The two types of wax were studied individually and in blends containing 20, 30, 40, 50, 60, 70 and 80% polyethylene wax in the paraffin by mass. The components were mixed using a magnetic stirrer with heating (Quimis, model Q 261).

2.2. Differential scanning calorimetry (DSC)

The thermal behavior of the mixtures was studied with a Differential Scanning Calorimeter (NETZSCH-Gerätebau GmbH, model DSC 204 F1). Samples (5–10 weighted) were first heated in an inert atmosphere (nitrogen) from 20 °C to 150 °C at a rate of 10 °C/min. The temperature was held at 150 °C for 3 min, and then the samples were cooled to 0 °C at 10 °C/min. Next, the samples were reheated to 150 °C. The thermal behavior was evaluated in the second heating run to eliminate the effect of thermal history.

2.3. X-ray diffraction (XRD)

The morphologies of the samples were studied using an X-ray diffraction analyzer (Shimadzu, model MAXima_X XRD7000) with a copper target and a $K\alpha$ radiation source at a wavelength of 1.5402 Å. The diffractograms were obtained with diffraction angles (2θ) ranging from 10° to 100° in increments of 0.02°/min and an integration time of 0.6 s. The voltage was 40 kV, and the current was 20 mA. The specimens had a diameter of 20 mm and a thickness of 2 mm.

2.4. Dynamic mechanical thermal analysis (DMTA)

The softening temperatures of the specimens were examined using Dynamic Mechanical Thermal Analysis (NETZSCH-Gerätebau GmbH, model DMA 242) in penetration mode. The samples were heated from 25 °C to 110 °C at a rate of 3 °C/min in a nitrogen atmosphere. The applied static force was 0.5 N, and the dynamic force had a maximum value of 0.4 N and a frequency of 50 Hz.

3. Results and discussion

The results obtained from the DSC thermal analysis are shown in Fig. 1. The heat flow for the pure paraffin exhibited two well-defined endothermic peaks, which are labeled a′ and b′. Peak a′ occurs at approximately 45 °C, and peak b′ occurs at approximately 65 °C. According to previous studies [5,6,9,10], the first peak (a′) can be attributed to a solid–solid transition of the crystalline structure and the second peak (b′) corresponds to the melting of the material. The polyethylene wax has three defined endothermic peaks

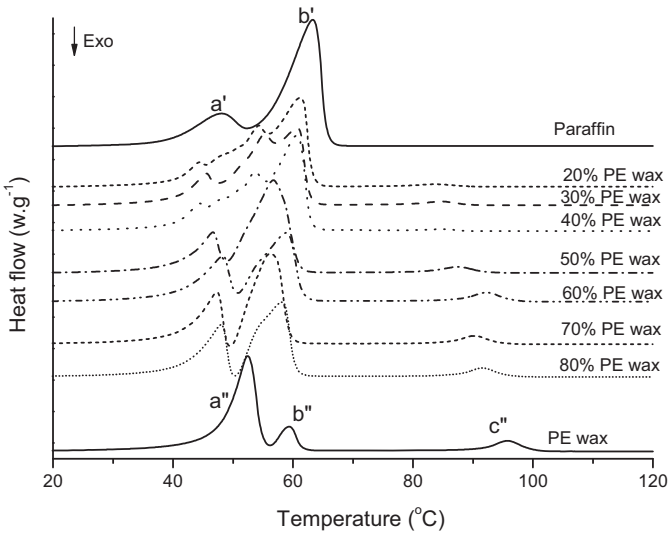

Fig. 1. DSC heating curves of pure polyethylene wax (PE wax), pure paraffin and their compositions.

(a″, b″ and c″), at significantly different temperatures, which can be an indication of phase separation in the crystalline structure. Peak a″ is the highest one, at around 50 °C and can be attributed to a solid–solid transition that occurred to the paraffin because no melting was observed at this temperature. Peak b″ can be attributed to the melting of the wax fraction with the lowest molecular weight. According to previous studies [11], peak c″ can be related to the melting of the small fraction of linear low-density polyethylene in the sample.

Regarding the intermediate blends, the temperature at which the third peak (c″) occurs decreases proportionally with the percentage of polyethylene wax in the blends, indicating formation of smaller crystallites due to the miscibility of the components in the molten state [5].

In the samples containing 20%, 30% and 40% polyethylene wax, the two waxes are partially miscible in the solid state. The amorphous region is miscible because of the miscibility of the components in the molten state, but is probably immiscible in the crystalline phase because it is possible to distinguish between the contributions of crystalline melting near 45 °C and 65 °C associated with the paraffin and the peak near 50 °C associated with the polyethylene wax. For the samples containing more than 50% polyethylene wax, this distinction was not clear, probably due to thermodynamically favorable interactions between the waxes in these concentrations [12], forcing the crystallites to become similar in size for both waxes.

The areas under the endothermic curves were evaluated over two temperature ranges, from 30 °C to 75 °C and above 80 °C (Fig. 2). In Fig. 2(a) it can be observed that the lower temperature range area decreases as the percentage of polyethylene wax in the blends increases, due to the area of this material which is lower than the area of paraffin. The area for the higher temperature range shown in Fig. 2(b) had the opposite behavior because the relative quantity of polyethylene wax, the material that is responsible for the second endothermic peak at higher temperatures, was greater.

The spectrum obtained by X-ray diffraction (Fig. 3) for the paraffin exhibited two intense and well-defined separate peaks, whereas the spectrum for the polyethylene wax exhibited a single and broad peak. As the concentration of polyethylene wax in the blends increased, the diffraction peaks became broader and less intense, and the crystallinity degree decreased (Fig. 4), because of increasing amounts of the lower-crystallinity polyethylene wax.