Synthesis, Characterization and Stabilization Analysis of Ferrofluid Based on Amazon Vegetable *Copaifera O. L. Oil*

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Abstract

The Nanoscience and Nanobiotechnology develops several researches using nanostructures with magnetic properties. These nanostructures can have different physical properties depending on their composition and therefore can be used in various applications such as: drug markers and carriers, microelectronics, magnetic separations and environmental applications. Furthermore, the use of natural components in the synthesis of these nanostructures may come to complement these nanomaterials due to their underexplored properties and their renewable origin. This research will aim to carry out a pre-study to analyze the stability of magnetic fluids based on Fe_3O_4 *nanoparticles functionalized with vegetable oil from the Copaifera SPP plant, known as Copaiba. Therefore, the* Fe_3O_4 *nanoparticles will be synthesized by the coprecipitation method by hydrolysis in an alkaline solution. Ferrofluid will be prepared by dispersing magnetic nanoparticles, at different concentrations, in the "in natura" oil. For the samples characterization measurements were taken of: UV-vis, FTIR, NIR spectrophotometry, XRD and Magnetic Susceptibility, the temporal analysis was recorded periodically, observing the possible stability of the samples. This work reports that the ferrofluid showed good stability and the magnetic properties associated with the nanoparticles the properties of the Amazon oil used.*

Keywords: magnetite; copaiba oil; magnetic fluid;

1. Introduction

The Amazon has a multitude of natural resources of great importance that can be used in Nanotechnology Applications, as is the case of oils of vegetable origin, which have peculiar properties and can be found/produced in abundance and its renewable origin. Among these oils, we can highlight the oil extracted from *Copaifera Officialis L*. popularly known as Copaiba, which is rich in unsaturated fatty acids (Oleic, Palmitoleic, Linoleic and Palmitic), being widely used in cosmetic and pharmaceutical industries. Copaiba oil is a transparent liquid whose color varies from yellow to brown. The correct designation for copaiba oil is oil-resin as it consists of resinous acids and volatile compounds, this liquid resin is rich in sesquiterpenes and diterpenes and different concentrations. Its most volatile fraction is composed of sesquiterpenes, while the heavier, resinous fraction is composed of diterpenes. The essential oil fraction is basically composed of sesquiterpenes and can make up 80% of oil-resin, depending on the species of copaiba. The resin fraction is made up of diterpene bicyclides and tetracylic acids derived from the labdane, kauramo and clerodane groups. This type of material has a great potential for possible biomedical applications, in addition to specific applications to investigate physical, chemical or physicochemical phenomena [1-4].

Fe_3O_4 nanoparticles are sources of several studies due to their magnetic nature and biocompatibility in different media. Its applications reach several fields of nanotechnology, mainly in Magnetic Resonance Imaging (RMI), Magnetohyperthermia (MHT), Controlled Drug Release, Magnetic Separation, Microelectronics and Environmental Applications. Ferrofluids, on the other hand, or magnetic fluids, are colloidal dispersions formed by nanoparticles with magnetic characteristics dispersed in a carrier liquid that can be organic or inorganic. One of the great challenges in producing magnetic fluids is to obtain an appropriate stability time, and thus make the magnetic nanoparticles remain in suspension, in the form of isolated entities, thus preventing agglomeration and therefore precipitation, however possible time. Therefore, it is necessary to study new types of carrier fluids to facilitate the stabilization of the compound [5-9].

The objective of this work was the molar magnetic characterization of a magnetic fluid synthesized from magnetic nanoparticles of magnetite (SPIONs), coated with a stabilizing molecular layer, for surface treatment, and dispersing in a carrier liquid, in this case, the oil plant from copaiba.

2. Experimental procedure

2.1 Materials

The analytical grade reagents were commercially available and used without further purification protocols. Iron (III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), iron (II) chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$) and Oleic Acid ($C_{18}H_{34}O_2$) were purchased from Aldrich. Sodium hydroxide (NaOH). The *Copaifera Officialis L*. oil was then extracted in the Amazon Rainforest, through artisanal processes.

2.2 Methods

The magnetic fluid was prepared using magnetic nanoparticles and copaiba oil "*in natura*". The nanoparticles were obtained through the coprecipitation method by alkaline hydrolysis of Fe^{2+} and Fe^{3+} ions in NaOH solution [7,8,10]. First, 27,1g of $FeCl_3$ are mixed together with 10g of $FeCl_2$ and 90 ml of deionized water. Soon after, the sample was taken to a magnetic stirrer at 800 rpm maintaining a temperature of 60 °C for about 15 minutes. Next, a 25% NaOH was added to the mixture. When NaOH is added, the initial solution breaks the bonds of the compounds causing the color change to black, indicating a precipitate formed by magnetite nanoparticles, the sample is still left on the shaker until reaching 74°C for better formation of the magnetite crystal lattices. Upon reaching the temperature, the solution was removed from the mechanical stirrer and inserted close to an external magnetic field to decant the particles that are suspended in the supernatant. The precipitates are then washed with deionized water for several days to remove impurities. The last step in obtaining nanoparticles consisted of the drying process at room temperature for 7 days and calcination at a temperature of 60°C, forming a solid that was macerated and sieved.

To facilitate the formation of an easily adsorbed molecular layer on the surface of the nanoparticles, they underwent a chemical treatment process, where there was dispersion in oleic acid solution and sonication for 10 min. Finally, SPIONs are dispersed in different concentrations in vegetable oil and after several homogenization processes, they are ready for characterization.

Magnetic Nanoparticles were characterized using the techniques of Magnetic Susceptibility and X-Ray Diffraction. On the other hand, the synthesized copaiba vegetable oil magnetic fluid was characterized by the techniques of Near Infrared Spectroscopy, FTIR and UV-Vis. For UV-vis and near infrared measurements, samples were diluted 5% in grain alcohol. The stability of the magnetic fluid was analyzed through photographs, acquired every hour for a period of 12 hours and then every 12 hours for 10 days, this process was repeated twice more to ensure reliability in the results.

3. Results and Discussions

To obtain the mean diameter of the magnetic nanoparticles, it is considered that the crystallinity of the sample is maintained, that is, that the interplanar spacing between the sets of interplanar planes is constant [9]. The diffraction pattern indicates that SPIONs have a cubic spinel structure indicating that it is a characteristic structure of cubic magnetite ferrite. Through the X-ray diffraction technique, it is possible to determine the mean diameter of the magnetic nanoparticles, with the aid of the Debye-Scherrer equation, obtaining a value of 21 nm ±0.3 [10, 11].

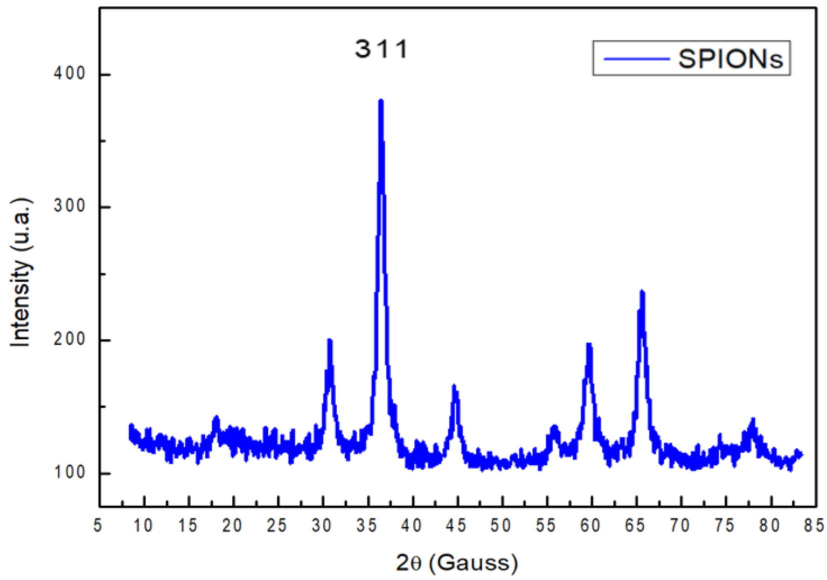

Figure 1. XRD of SPIONs.

The SPIONs data obtained through the Molar Susceptibility technique using the Gouy Method coupled to a 1.5 Tesla Magnetometer, presented an Average Molar Susceptibility of 2630.10^{-5}, permeability of 1,0263 and Magnetization of 1,0732, which may indicate the superparamagnetic characteristic of the sample [12].

Figure 2 of the spectrum of the magnetic fluid shows the characteristic bands of the composition of the vegetable oil of copaiba, highlighting the diterpene fraction which is located in the absorption bands between 1800 and 1600 cm^{-1} identifying the groups of carboxylic acids or esters in 1736 cm^{-1}. Highlighting the sesquiterpene fraction in the absorption region between 3000 and 2800 cm^{-1}, identifying the axial deformations of the C-H bond of aliphatic hydrocarbons at 2930 and 2859 cm^{-1}. At 1240 cm^{-1} the axial deformation for the C-O bond is observed. We can observe the angular deformation of CH_3 in the absorption band at 1377 cm^{-1} indicates that CH_3 is linked to the carboxylic group [8, 13].

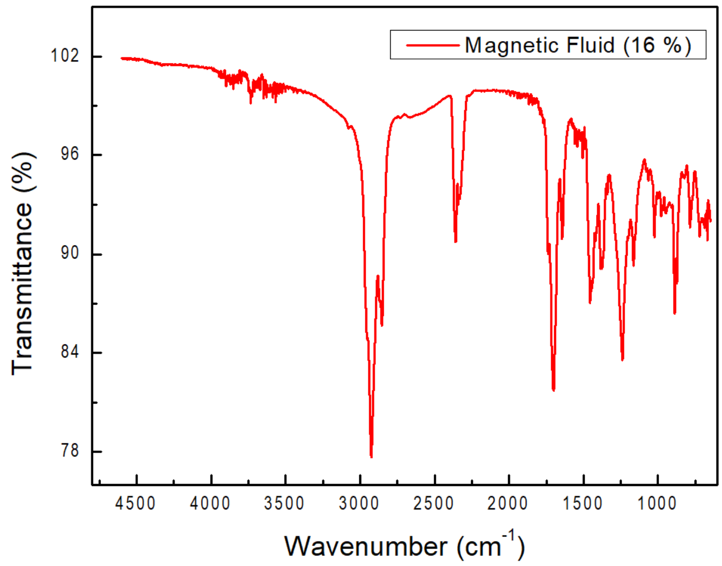

Figure 2. FTIR of Magnetic Fluid.

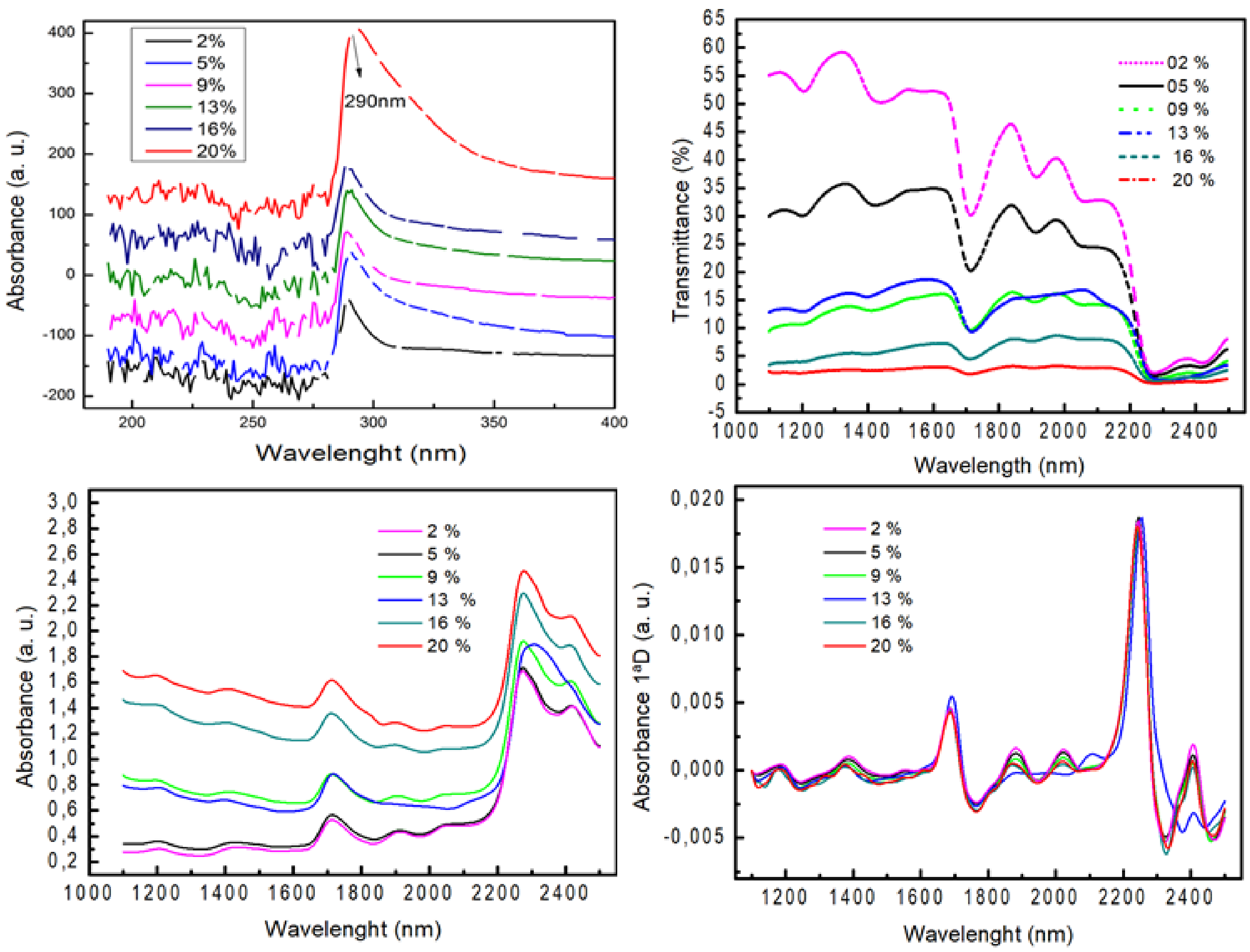

Figure 3: a) Uv-vis spectrum of magnetic nanofluid b) NIR (transmittance) c) NIR (Absorbance) d) NIR of samples (Absorbance 1ª Derivate).

Figure 3 shows the spectra of the magnetic fluid in different proportions, where the nanoparticles/natural oil ratio, in percentages ranging from 2 to 20%. Figure 3a shows the absorbance data obtained by the UV-Vis spectroscopy technique, where it is possible to observe an interaction peak at 290 nm, this peak is associated with the interactions between the SPÌONs and the carboxylic groups present in the natural oil. In Figure 3b, we have the transmittance spectrum made in the infrared region (1100 to 2500 nm), the samples showed peaks characteristic of essential oils, at 1700 nm we have a peak associated with vibrations characteristic of high concentrations of CH_2. In Figure 3c we have the absorption measurements in the infrared spectrum with its first derivative shown in Figure 3d, the data indicate interaction peaks at 1211 nm associated with CH_2 bonds, at 1390 nm associated with CH_3 bonds, between 1664 nm and 1725 nm that carbon bonds are associated in long chains and at 2144 nm and 2200 nm they are associated with CH=CH double bonds [8, 13]. The data indicate that the structure of the natural oil is preserved in the magnetic fluid.

The data related to the temporal analysis of the stability test may indicate that, all with good stability dissipated in the first 12 hours, after this time the 5% sample starts to destabilize, possibly the agglomeration of the nanoparticles occurs, making them susceptible to gravitational effects [14]. As the 2%, 9%, 13%, 16% and 20% dissipation of total stability time greater than 5 days, having its progressive decay over the days. It was also possible to observe that those with 13% and 16% remained stable during the 10 days of experimentation.

4. Conclusion

This study reports the successful preparation, characterization with temporal analysis of an organic magnetic fluid using superparamagnetic nanoparticles coated in essential oil of Amazonian origin. Molar Susceptibility and XRD data show that SPIONs have an average diameter of 21 nm and are highly susceptible to magnetic fields. The analysis by spectrophotometry in UV-Vis and NIR indicate that the magnetic fluid has the properties associated with natural oil. the samples showed good stability, the data indicate that the concentrations of 13% and 16% are the most suitable for the synthesis of magnetic fluids based on the vegetable oil of *Copaifera Officialis L.*

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6. References

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