ISSN: 2230-9926

Available online at http://www.journalijdr.com

IJDR

International Journal of Development Research

Vol. 12, Issue, 11, pp. 60150-60153, November, 2022

https://doi.org/10.37118/ijdr.25736.11.2022

RESEARCH ARTICLE OPEN ACCESS

DEVELOPMENT OF MAGNETO-POLYMER NANOEMULSIONS BASED ON THE AMAZON OIL OF *CARAPA GUIANENSIS AUBL*

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ARTICLE INFO

Article History:

Received 11th September, 2022
Received in revised form
25th September, 2022
Accepted 08th October, 2022
Published online 30th November, 2022

KeyWords:

Nanoemulsions; Amazon Oil; Policondensation method; Magnetic Nanoparticles.

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ABSTRACT

The present study aimed to synthesize and characterize a nanostructured complex based on nano emulsions from polymers extracted from Carapa *Guianensis Aubl. essential* oil. and doped with Fe_3O_4 nanoparticles. The nanoparticles were synthesized by the hydrolysis co-precipitation process in alkaline medium, the polymers by the polycondensation method and the nano emulsions by the ultrasonic homogenization technique. To characterize the material, the following techniques were used: Spectroscopic (UV-Vis and NIR), Electronic (MET and SEM) and Magnetic (SM) Micrographs.The data obtained in the NIR and UV-Vis spectra showed the molecular groupings characteristic of fatty acids present in *C. guianensis* oil. The synthesized polymer presented molecular interactions associated with bonds of unsaturated fatty acids, in addition to the specific vibrations of the Amazonian oil. Both characteristics can be found in nano emulsions, with the addition of unique electronic interactions bands. The data obtained by MET, SEM and SM indicated the good formation of Fe_3O_4 nanoparticles and nano emulsions, both with spherical shape, with superparamagnetic characteristics and mean diameter of 10.23 nm and 340 nm respectively. The data indicate that the development of this new nanostructured complex was a success.

Citation: Silva, L.G.F., Pacheco, H. P., Martins, Q. S., Santos, J. G. and Silveira, L. B. 2022. "Development of magneto-polymer nanoemulsions based on the amazon oil of carapa guianensis Aubl*", International Journal of Development Research, 12, (11),* 60150-60153.

INTRODUCTION

Carapa guianensis Aubl. popularly known as Andiroba, is part of the *Meliacea* family. The plant is found throughout the Amazon region, mainly in the vicinity of river beds and wetlands (PEREIRA; TONINI, 2012). *The* vegetable essential oil of *C. guianensis* is extracted in an artisanal way, using the pressing method, with decanting and is rich in unsaturated fatty acids (Palmitic, Oleic, Linoleic and linolenic), some vitamins (A and B2), some minerals (Ru, Cu, Au) and due to their anti-inflammatory, antibacterial, antioxidant properties and their renewable origin, are already present in various applications in the cosmetics, health and food industry (SILVA *et al*., 2020; SARQUIS *et al*., 2020; ROMA *et al*., 2013; VENDRAMINI *et al*., 2012). The polymerization process of Amazonian essential oils produce polymers, particularly of *diterpenic* and lauric acid compounds*,* with the possibility of adsorption of these molecules on the nanoparticles surface by complexation of the carboxylic groups tothe superficial Fe^{3+}ions using a stabilizing layer and properties close to their raw material (SILVA, *et al*., 2020; RODRIGUEZ *et al*., 2013; PEREIRA and TONINI, 2012; RIGAMONTE-AZEVEDO *et al*., 2004).

Because they are highly responsive to external magnetic fields, Fe_3O_4 nanoparticles have a great focus on Nanoscience, Nanotechnology and Nanobiotechnology (ZHAO *et al*., 2021; SALMANIAN *et al*., 2021). Besides possessing this characteristic known as superparamagnetism, this type of nanostructure has properties such as: high coercivity, low Curie temperature, stability, low toxicity, biocompatibility and biodegradability in organic media (WU; HE; JIANG, 2008). More recent studies indicate that by being incorporated into other structures, these nanoparticles can add new properties in addition, the use of embedded magnetic nanoparticles can be used in applications such as contrast agents, drug delivery, magneto hyperthermia and photodynamic treatments (PELLOSI *et al*., 2018; ZHANG *et al*., 2015; BROJABASI *et al*., 2014). In conjunction with the nanoparticles, the Nanoemulsions have been gaining a lot of prominence. This type of nanostructure has already been used for about 40 years as a source of calories, essential fatty acids and, more recently, as drug release systems (SHARMA *et al*., 2020; MASON *et al*., 2006). Studies also indicate that nano emulsions can be produced on a large scale, using common industrial operations, that is, they can be easily introduced in commercial applications, ranging from disinfectants, drug delivery, food quality systems and cosmetics (NASEEMA *et al*., 2021; MCCLEMENTS and JAFARI, 2018; SUTRADHAR and AMIN 2013; LEDET *et al*., 2013). In this work data resulting from the synthesis and characterization of hybrid nano emulsions based on Amazonian vegetable oil from *Carapa guianensis Aubl* will be presented. and doped with Fe_3O_4 nanoparticles.

MATERIAIS E MÉTODOS

Ferric Chloride ($FeCl_3.6H_2O$), Ferrous Chloride ($FeCl_2.4H_2O$), Sodium Hydroxide (NaOH), Ethylene Glycol ($C_2H_6O_2$), Polysorbate 80 ($C_{64}H_{124}O_{26}$) and Potassium Hydroxide (KOH), Deionized Water (H_2O) and *in natura* vegetable oils extracted from seeds of *C. Guianensis*.

Synthesis of Magnetic Nanoparticles (Fe_3O_4): The nanoparticles were synthesized by the hydrolysis co-precipitation method in alkaline medium, which is also known as the *Massart* method. It was mixed in a beaker $FeCl_3.6H_2O$, $FeCl_2.4H_2O$, ratio 1:2, and deionized H_2O. Afterwards, the sample was taken to a magnetic stirrer at 1200 rpm, maintaining a temperature of 70 °C for 20 minutes. Next, NaOH 1M was added to the initial solution, causing the color change to dark color. The precipitates were then washed with deionized water to remove residues from the process, placed for drying in a greenhouse at room temperature, macerated to separate the nanoparticles and sieved, using micrometric sieves (CHIN; YAACOB, 2007; LAURENT *et al*., 2008; SANTOS *et al*., 2012).

Polymerization of Vegetable Oil: The vegetable oil was polymerized using the polycondensation or condensation polymerization method. The method uses free fatty acids as precursor monomers that will be diluted to smaller monomers, in this case $C_2H_6O_4$, in thermal stress environment, promoting a polymerization reaction. In order to obtain the free fatty acids from the essential oil, the plant sample will undergo the transesterification process of the triglyceride chain. At the end, the polymerized material will be centrifuged and lyophilized (SILVA *et al*., 2020; MIAO *et al*., 2013; SENIHA GÜNER; YAĞCI; TUNCER ERCIYES, 2006; SHARMA; KUNDU, 2008);

Preparation of Nanoemulsions: Nano emulsions were synthesizedthrough the top-down process known as Ultrasonic Homogenization, where Fe_3O_4 nanoparticles were diluted in different concentrations in the oil polymer of *C. guianensis*, after homogenization a magnetic fluid was formed, which was dispersed in a solution containing the surfactant compound (Polysorbate 80) diluted, with different concentrations, in deionized water and homogenized by mechanical agitation at 10000 rpm for about 10 min. After this step we have formation of emulsions that were observed in the optical microscope. To reduce the sample size, for nano scales, the material went through an Ultrasonic Sonicator at 20 kHz for 10 min ((MEHMOOD *et al*., 2017; BRUXEL *et al*., 2015). After stabilization of sampling at room temperature (25°C), the nano emulsion is ready for characterization processes;

Characterization of samples: To determine the optical, morphological and magnetic properties, the samples went through the following tests: Uv-Vis, NIR, MET, MEVe SM;

Scanning Electron Microscopy (MET)*:* The fluid containing the nano emulsions was evaluated through MET, with an Electronic Microscope called JEOL 100 CXII. The sample is covered by a polymeric film (Formvar) and inserted into the equipment;

Scanning Electron Microscopy (SEM): The dispersion containing the nano emulsions was evaluated through SEM, brand JOL, JSM-700 1-F. The samples surface wascovered by a thin layer of Au to ensure the electrons conductivity on its surface;

UV-VIS-NIR Spectroscopy: The *Carapaguianensis Aubl*. vegetable oil, the synthesized polymer and the Nano emulsions were analyzed using a UV-VIS-NIR UV-3600 UV-NIR spectrometer, Shimadzu. The samples were diluted in different concentrations in distilled water, an aliquot of the sample was taken and inserted into the equipment.

RESULTS AND DISCUSSION

The morphological analysis of magnetic nanoparticles (NPs) and CNGNE was performed through the measurements of MET and SEM. In relation to magnetic nanoparticles, the MET measurements (Figure 1) indicate the spherical shape with several agglomeration sites, even so, it is possible to indicate that the average diameter of the nanoparticles was 10.23 nm ± 3.4 nm with a polydispersion of 0.33 ± 0.1 (GREGORIO-JAUREGUI *et al*., 2012).

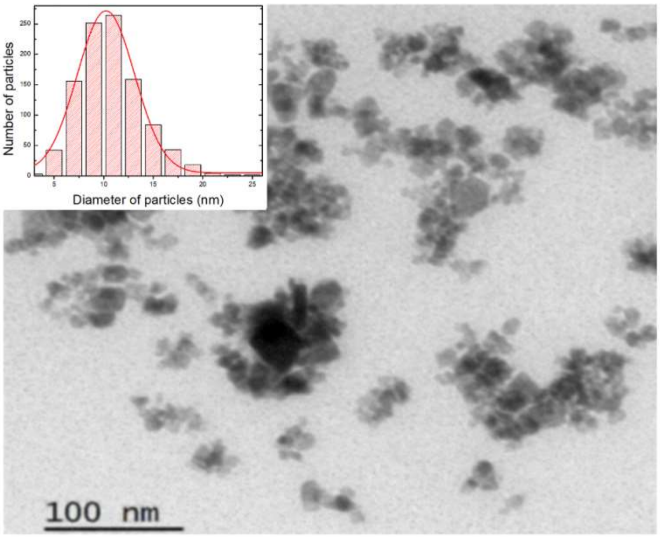

Figure 1. Micrographs made by MET of Fe_3O_4 Magnetic Nanoparticles

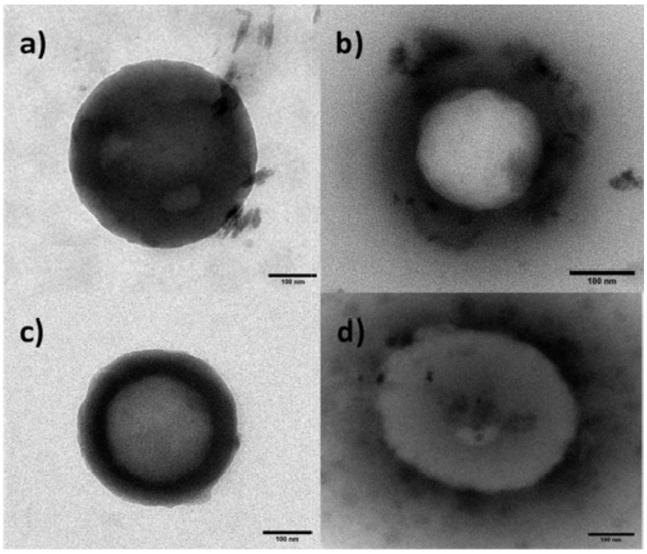

Figure 2. Micrographs made by MET. a) external part of CGNE b), c) and d) Internal part of CGNE

Micrographs made by MET indicate that the nano emulsions synthesis (CNG) was a success. The structure can be demonstrated in Figure 2 a), where the data show that the magnetic fluid is in the nucleus of the nano emulsion. The material presented a spherical shape with a diameter of 340 nm with a polydispersion of 35%. The micrographs presented in Figure 2 a) b), c) and d) indicate that the NPs, although present in the entire nanomaterial, are concentrated at their extremes. The data may indicate that magnetic nanoparticles are present and interacting, both with the nucleus of the CNGNE, formed by the polymer (CGPO), and its interface with the external environment, consisting of $C_{64}H_{124}O_{26}$.

In fact, the NPs can interact primarily with CGPO and secondarily with $C_{64}H_{124}O_{26}$ through double-link modulations, pre-existing both CGNE interfaces (PASCUAL-VILLALOBOS *et al*., 2019). The micrographs made by SEM, as a magnification of 5000x (FIGURE 3a) and 20000x (FIGURE 3b), prove the spherical shape of the Nano emulsions, which have a non-homogeneous surface, which may indicate the existence of NPs on its surface. (LAHIRI *et al*., 2017; NANDY *et al*., 2021). Figures 4 shows the UV-Vis absorbance spectrum of the Nanoparticles (NPs), Vegetable Oil (CGEO), Polymer (CGPO) and Nano emulsion (CGNE) samples. It was verified that in the NPs samples there are peaks at 394 nm, 379 nm and saturates at 200 nm.

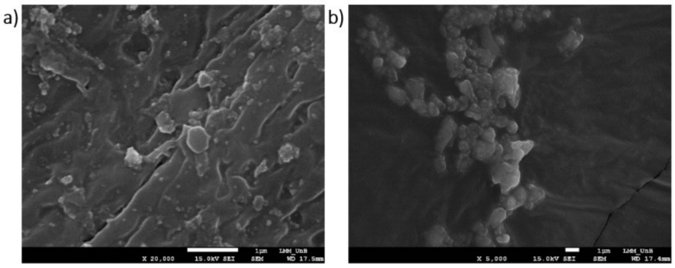

Figure 3. Micrographs made by SEM. A) Magnification of 5000x b) Magnification of 20000x

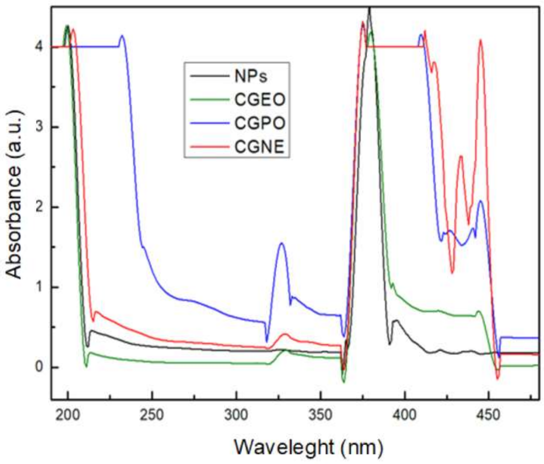

Figure 4. UV-Vis spectrophotometry of Nanoparticles, Vegetable oil, Polymer and Nano emulsions

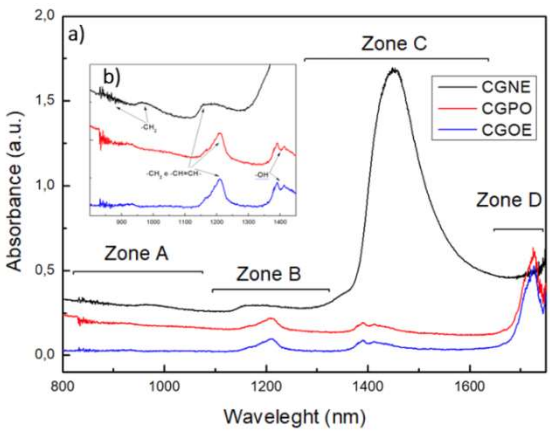

Figure 5. NIR spectrophotometry of Natural Oil, Polymer and Nano emulsions with ranges: a) from 800 nm to 1750 nm. b) from 800 nm to 1420 nm

Whereas in the samples CGEO, CGPO and CGNE they show interaction peaks at 450 nm, 410 nm and 325 nm with different intensities. CNGE presented unique peaks at 432 nm and 418 nm and saturates at 200 nm. While the CGPO showed peak saturation at 232 nm. CGEO, CGPO and CGNE showed interaction peaks at 370 nm, this interaction may be linked to the main composition of the natural oil (ZHANG *et al*., 2015). Figure 5 shows the NIR spectrophotometer of CGNE, CGPO and CGOE samples in a spectrum ranging from 800 nm to 1750 nm. The chart was separated into electromagnetic spectrum zones. In the first electromagnetic spectrum Zone (zone A) ranging from 800 nm to 1090 nm, it is also known as the transition zone in the infrared, for this sector it was possible to observe weak interactions in all the samples, these peaks may be associated with molecular vibrations of bonds – CH_2. For the second interval (zone B), which ranges from 1090 nm to 1260 nm, we have interactions in 1210 nm, which is associated with the bonds -CH_2 and - CH=CH- characteristic of unsaturated fatty acids linked to the essential oil of *C. Guianensis* studied.In the CGNE sample there is a displacement of the interaction peak to a position of 1160 nm. Upon observing the C zone, which ranges from 1760 nm to 1630 nm, a peak at 1450 nm is noticeable in the CGNE sample, and this peak may be associated with -OH bonds; resulting from the dilution of nano emulsions in distilled water. The CGOE and CGPO samples showed interaction peaks at 1390 nm and 1421 nm, which are also associated with the bonds – OH. Probably these interaction peaks were superimposed in the GNE sample. For the last zone of the electromagnetic spectrum (zone D), ranging from 1650 nm to 1750 nm, there was a peak of interaction at 1727 nm in the CGOE and CGPO samples that may be associated with the -CH_2,CH_3 AND -CH=CH- bonds. The low intensity vibrations found in the CGNE sample can also indicate the associations described above (ABU-KHALAF *et al*., 2020).

CONCLUSIONS

With the results listed we can suggest that the synthesis process was a success, the NPs present good stability, a spherical shape and diameter that facilitates interactions in different environments and magnetic properties. Whereas the polymerized phase presents characteristics originated from the vegetable oil of *Carapa guianensis Aubl.*, which has also been transposed to CGNE. This new developed nanomaterial presents unique properties, indicated in the UV-Vis measurements. Due to the abundance of raw material, good versatility, easy scalability and unique properties this new hybrid nanomaterial can have several applications as an example: contrast agent in Magnetic Resonance by Image and Transdermal Diffusivity in Stratum Corneum membrane. However, new studies should be carried out to explain bioactivity, toxicity and biocompatibility.

Acknowledgments: We are grateful for the financial and academic support offered by the Graduate Program in Nanoscience and Nanobiotechnology of the University of Brasilia, Optics and Nanoscopy Group (GON) – UFAL and Federal Institute of Education, Science and Technology of Rondônia – IFRO.

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