# Microwave Interactions between Magnetic Nanoparticles and Magnetic Graphene Oxide and their Applications as Electromagnetic Heating Facilitators for Heavy Crude Oil

Interacción con microondas de las nanopartículas magnéticas y óxido de grafeno magnético y sus aplicaciones como susceptor en el calentamiento electromagnético de crudo pesado

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**Abstract**

**Objective:** This work proposes and evaluates a technology for improving heavy crude oil mobility through the addition of magnetic nanoparticles and external electromagnetic fields. **Methodology:** The study first individually characterizes heavy crude oil, magnetite nanoparticles, and magnetic graphene. Then, the magneto-rheological behavior of heavy crude oil and its mixtures with nanoparticles and magnetic graphene assessed using 0.5 to 10 wt.% compounds. Subsequently, these mixtures were subjected to electromagnetic radiation within the microwave range at different radiation times to achieve different compositions. These treatments were conducted in an electromagnetic cavity using a cylindrical waveguide with powers ranging between 0.8 and 1 kW. **Results:** Initially, the crude oil did not respond to the electromagnetic radiation treatment as it is transparent to this treatment. However, after adding magnetic nanoparticles, comprising both magnetite and magnetic graphene, the mixture strongly interacted with the electromagnetic field. The higher the concentration of these particles, the greater the temperature increase experimented by the mixture. For the experiments in the electromagnetic cavity, an increase from the original temperature of 22°C to the 58.2°C–60°C range was evidenced in 60 s for the magnetite at a 0.5 wt.% concentration. For experiments using the cylindrical waveguide as an applicator at the same previous experimental conditions, temperatures above 90°C were reached. Using magnetic graphene and the same electromagnetic cavity, temperatures between 81.9°C and 96.5°C were reached. **Conclusions:** Both materials notably increased heavy crude oil heating rates (decreasing its viscosity), when subjected to electromagnetic radiation within the microwave range. In fact, both materials can be considered as candidates for improving crude oil extraction and transportation processes.

**Keywords:** Heavy Crude Oil, Magnetite Nanoparticles, Magnetic Graphene Oxide, Microwave Radiation.

**Resumen**

**Objetivo:** Proponer y evaluar una tecnología que mejore la movilidad de crudos pesados, mediante la adición de nanopartículas magnéticas y la aplicación de campos electromagnéticos externos. **Metodología:** Se caracterizaron individualmente el crudo pesado, las nanopartículas de magnetita y el grafeno magnético. Se evaluó el comportamiento magneto-reológico del crudo pesado y de sus mezclas con nanopartículas y grafeno magnético en un rango de composiciones en el rango 0.5 %wt a 10 %wt. Posteriormente, estas mezclas se sometieron a radiación electromagnética en el rango de las microondas, para diferentes composiciones y tiempos de radiación. Estos tratamientos se realizaron en una cavidad electromagnética y en una guía de onda cilíndrica, con potencias entre 0.8 kW y 1 kW. **Resultados:** El crudo original no respondió al tratamiento con radiación electromagnética, siendo transparente a éste. Sin embargo, con la adición de nanopartículas magnéticas, tanto de magnetita como de grafeno magnético, la mezcla interactuó fuertemente con el campo electromagnético. A mayor concentración de estas partículas, mayor fue el incremento de la temperatura de la mezcla. Para experimentos en la cavidad electromagnética, se evidenció un incremento desde la temperatura inicial de 22 °C hasta el rango 58.2 °C – 60 °C para el caso de la magnetita a una concentración de 0.5 % wt en 60 s. Para experimentos utilizando como aplicador la guía de onda cilíndrica y para las mismas condiciones experimentales anteriores, se alcanzaron temperaturas por encima de los 90 °C. Utilizando el grafeno magnético y la misma cavidad electromagnética se alcanzaron temperaturas en el rango de 81.9 °C-96.5 °C. **Conclusiones:** Ambos materiales incrementan notablemente el calentamiento del crudo pesado (disminuyendo su viscosidad), cuando se someten a radiación electromagnética en el rango de las microondas, y podrían considerarse candidatos para mejorar los procesos de extracción y transporte de crudo.

**Palabras claves:** Crudo pesado, nanopartículas de magnetita, óxido de grafeno magnético, radiación de microondas.

## Introduction

Given the continuous increase in the energy demand, heavy oils become an alternative to overcome this need. It is also known that among oil sands, heavy and extra-heavy oils account for 70 % of global fossil fuel reserves. However, one major disadvantage of heavy oils is their high viscosity, feature that increases transportation, operation and processing costs. Hence, the need to develop alternatives that decrease heavy and extra-heavy oil viscosity. To heat the oil as to increase its mobility efficiently, would prevent excessive energy consumption. High frequency electromagnetic fields (HF-EMF) heat a fluid through vibrations in the chemical bonds allowing direct heating, unlike conventional methods where the heat is transmitted, first, to the container walls and later to the fluid through thermal conduction. The main objective of HF-EMF is to achieve rapid and localized heating. However, HF-EMF only affect molecules with high dipole moments. For this reason, oils are not a good target to apply this strategy because most hydrocarbon molecules exhibit very low polarity; consequently, the addition of susceptors - materials that interact strongly with the electromagnetic radiation - is commonly used. For example, the addition of magnetite NPs to heavy oils has been considered as an option to heat the mixture, reducing its viscosity. However, magnetite NPs tend to form aggregates due to strong anisotropic dipole interactions, thus losing their dispersibility, which eventually hinders their use in many applications [1]. Therefore, it is necessary to immobilize these NPs in suitable supports to preserve their unique properties. Among a large group of materials, graphene and its derivatives are considered to have high potential for the immobilization of NPs [2]. Of interest, graphene oxide (GO) is an attractive material due to its two-dimensional structure that gives it a high surface area and the reactivity of its functional groups. Immobilized NPs not only prevent the aggregation of graphene or GO, but also preserve the properties of NPs through a synergistic effect between both components. The lack of functionalities in the surface of graphene to directly immobilize the NPs on its surface, favors the use of GO as support material in the assembly of nanocomposites based on graphene [3, 4, 5]. With this approach, in this research study, a composite based on graphene oxide and magnetite NPs was prepared. This material was added to a heavy oil and irradiated with HF-EMF to study its effects on heating the mixture and reducing its viscosity.

## Methodology

### Reagents and materials

Union Carbide Corp graphite was used. Reagents $H_3PO_4$ of 85% wt, $CH_2O_2$ of 98% wt, $H_2SO_4$ of 98% wt and $H_2O_2$ of 30% wt, were purchased from Panreac AppliChem GmbH. The $KMNO_4$ of 98% wt was acquired from Merck KGaA, and the Sigma-Aldrich $FeSO_4.7H_2O$ of 98% wt, $NaOH$ of 98% wt, and $KNO_3$ of 98% wt were used.

## Synthesis of graphene oxide

Graphene oxide was obtained by modifying the synthesis of Tour [4]. An acid mixture of H2SO4 / H3PO4 (9: 1 v / v) was prepared, and added to 1.0 g of graphite. Then, 6.0 g of KMNO4 were slowly mixed into this composition. The resulting substance was stirred at 50 °C for 24 hours, after which H2O2 at 30 % wt was slowly added. Finally, the product was neutralized and diluted in cold water, then purified by centrifugation.

## Preparation of Fe3O4@GO composite and Fe3O4 NPs

The graphene oxide composite material was prepared through the oxidation of Fe+ 2 with KNO3 under alkaline conditions, using a nitrogen atmosphere. The mixture was heated to 90 ° C while maintaining constant stirring. Later, 7.2 g of FeSO4.7H2O, 0.582 g of KNO3 and finally 2.88 g of NaOH (aqueous) were added. The result was a black dispersion, which was centrifuged and washed with ethanol, always maintaining a pH of 7. Lyophilization was performed to obtain graphene oxide with magnetic properties.

## Instrumental

The Nicolet IS50-Thermo Fisher Scientific spectrometer was used to perform the structural analysis of the synthesis products by infrared spectroscopy, complementing the structural characterization with the analysis of the diffractograms obtained in the D8 Advance-Bruker equipment. The microstructure and morphology were analyzed by way of transmission electron microscopy (TEM), in the JEM-1210-JEOL equipment. The magnetometer MPMS-XL-Quantum Design at 300 K was used to obtain the magnetization curves.

## Characterization of oil

A sample of Colombian extra heavy oil characterized by its high viscosity and low mobility was used. The SARA hydrocarbon characterization was performed through chromatographic techniques using the methodology described in the ASTM D-4124. API gravity and density were measured using the procedures of ASTM 1298. Likewise, viscosity curves were made both for the oil, and the oil with magnetite NPs for different concentrations and temperatures. We used a permanent magnet viscometer that encapsulated the sample.

## Rheology measurements of oil samples and its mix with magnetite nanoparticles

Using the Anton Paar MCR 320 rheometer we determined the viscosity of the oil at 30 °C, 50 °C and 70 °C with shear rates from 0 s-1 to 10 s-1. This equipment also has a cell that allows measuring viscosities in the presence of static magnetic fields. The ICM -magnetic field strength- values used in these experiments were 0.17 T, 0.35 T, and 0.65 T.

## Heating of oil by HF-EMF

Mixtures of oil and Fe3O4 NPs or magnetic graphene oxide composite were made in concentrations of 0.5 %wt, 1.0 %wt, 5.0 %wt and 10.0 %wt. The

samples were placed in a microwave cavity and subjected to microwave radiation (2.45 GHz). Additionally, the heating was carried out in a cylindrical waveguide using the same frequency. For both cases, a magnetron of 800 W nominal power was used. For both types of experiments, the heating of the mixtures was monitored in real time through an FISO optical fiber measurement and monitoring system at different times: 5 s, 15 s, 30 s, 45 s and 60 s. The heating with a thermal camera Fluke-50 Tis was also monitored. The short measurement times were due to the risk of damage to the optical fiber due to both, the organic solvents and the relatively high temperatures.

## Results

Figure 1 shows the infrared spectra, which were obtained from the products. The red spectrum corresponds to graphene oxide, and it shows between 3600 cm-1 to 3300 cm-1, the corresponding vibration with the flexion of the O-H group. In 1720 cm-1 the vibration of the group C=O appears. In 1626 cm-1 the characteristic signals of C=C are observed, which did not oxidize. At 1405 cm-1 the signal of the O-H link is present. In 1220 cm-1 the vibrations of the C-O-C epoxy groups can be appreciated, while in 1052 cm-1 the C-O vibrations are observed [6, 7, 8]. The spectrum for Fe3O4 nanoparticles is observed in blue. The characteristic band of this product is that of Fe–O at 540 cm-1[9]. The green spectrum corresponds to the Fe3O4 @ GO composite, which also shows the band of 540 cm-1, the main indicator of the formation of the composite material (composite).

**Figure 1.** Infrared spectra of graphene oxide (GO) (red), magnetite nanoparticles ($Fe_3O_4$) (blue) and composite (Fe3O4 @ GO) (green)
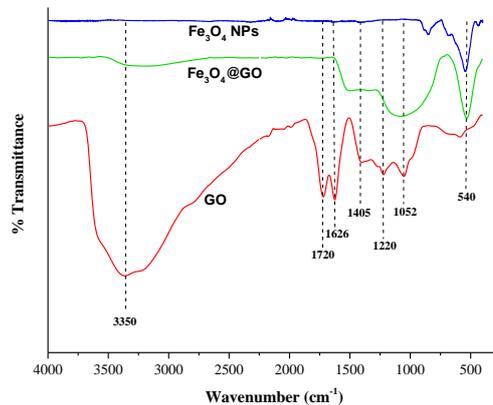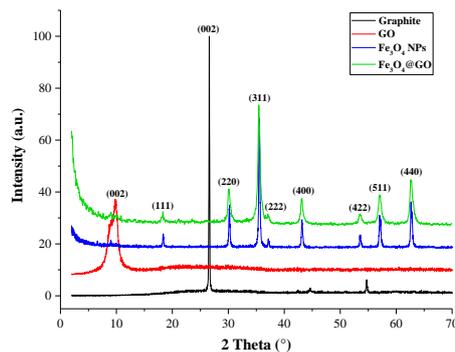


**Source:** Own production

Figure 2 shows the diffraction pattern for 4 products. In red, the graphite that was used as raw material in the synthesis of graphene oxide. A signal is observed around 2θ=26.53°, which after performing the oxidation process, is observed in the case of graphene oxide (red) at 2θ=9.42°, indicative of

graphite oxidation. The blue diffraction pattern corresponds to magnetite nanoparticles, where the signals observed in 2θ: 18.36°, 30.15°, 35.56°, 37.17°, 43.17°, 53.59°, 57.11°, and 62.70° correspond to planes (111), (220), (311), (222), (400), (422), (511), and (440), respectively [5, 6]. These signals match JCPDS reference 89-3854, for magnetite [10, 11].

The green diffraction pattern corresponds to the Fe3O4@GO composite. It shows the same signs of magnetite, indicating that the functionalization of graphene oxide was carried out. It is also possible to observe in this pattern a very slight signal for the band (002), this is because the crystallinity of the magnetite is more intense than that of graphene oxide [7, 8]. Therefore, it can also be concluded that magnetite is dispersed in the oxide, [12].

**Figure 2.** Diffraction patterns for: graphite, graphene oxide, $Fe_3O_4$ nanoparticles and for the ($Fe_3O_4$@GO) composite



**Source:** Own production

**Figure 3.** Transmission micrographs (TEM), a) Graphene oxide sheets, b) Graphene oxide SAED standard c) Composite material ($Fe_3O_4$ @ GO), d) SAED composite pattern ($Fe_3O_4$ @ GO)
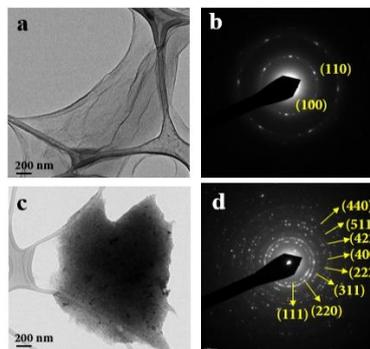


**Source:** Own production

Figure 4 shows the magnetization curves for the Fe3O4 nanoparticles and the Fe3O4@GO composite. Their magnetization values were 66 emu/g and 86 emu/g respectively. The hysteresis presented by both materials allows to describe them as ferromagnetic [13, 14, 15, 16, 17].

**Figure 4.** Magnetization curves of $Fe_3O_4$ NPs and $Fe_3O_4$@GO compounds
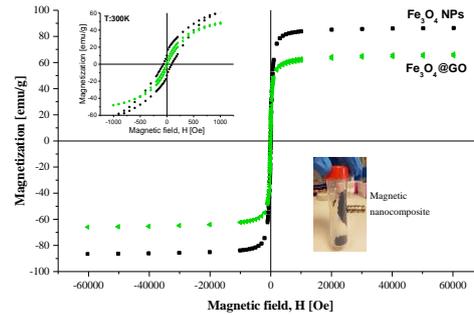
## Characterization of oil

Table 1 shows the characterization of oil. The SARA analysis revealed that the composition of the sample is rich in resins and asphaltenes. The presence of these molecular families are characteristic of heavy oil, and the fractions of higher molecular weight are the main contributors to the high viscosity of the fluid. This table also includes API gravity measured at 25 °C and fluid density at 60 °C. Both values confirm that oil is a high viscosity fluid, with a very low API degree.

**Table 1.** Characterization of oil

| Oil Characterization | | | | |
|---|---|---|---|---|
| SARA | Saturates | Aromatics | Resins | Asphaltenes |
| | %wt | | | |
| | 16.6 | 29.3 | 37.3 | 16.8 |
| API Gravity (25 °C) | 8.5° API | | | |
| Density (60 °C) | 0.9853 g/cm³ | | | |

## Rheological Curves of Oil

The viscosity of the oil at 30 °C, 50 °C and 70 °C was determined with shear rates from 0.1 s-1 to 100 s-1 using the Anton Paar MCR 320 rheometer. This equipment contains a cell that allows measuring viscosities in the presence of static magnetic fields. The values of the Magnetic Field strength (MFS) used in these experiments were 0.17 T, 0.35 T and 0.65 T. Figure 5 shows the experimental results (without a magnetic field) of the variation of viscosity with the temperature for different values of the shear rate up to 100 s-1.

**Figure 5.** Variation of the viscosity of heavy oil with respect to shear rate and temperature (30 °C, 50 °C and 70 °C)
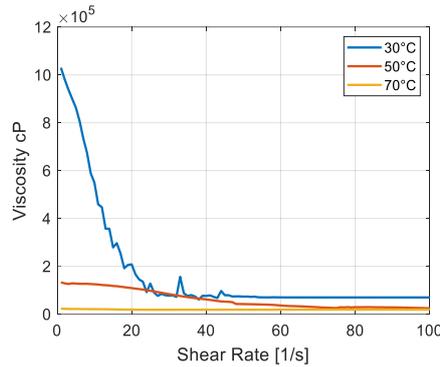


**Source:** Own production

As expected for this type of fluids, viscosity decreases considerably with the increase in temperature, reaching its lowest value at 70 °C. Figure 6 shows the results of the variation of viscosity with the shear rate, but at a constant temperature of 30 °C and for different values of the MFS. The viscosity decreases consistently due to the presence of the magnetic field, although the effect of changing its intensity at high shear rate values is not highly marked.

**Figure 6.** Variation of the viscosity of the heavy oil with respect to the shear rate and intensity of the magnetic field (MFS) maintaining a temperature of 30 °C
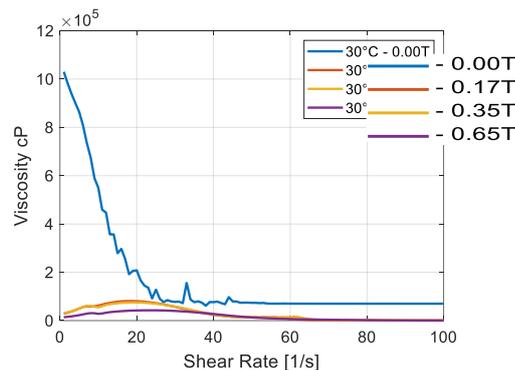


**Source:** Own production

Figure 7 includes the results of the viscosity variation for an operating temperature of 70 °C. The trend remains and the increase in the MFS applied to oil decreases its viscosity, regardless of its operating temperature. Nevertheless, certain deviations are appreciated for a low range of the shear rate when a low MFS is applied.
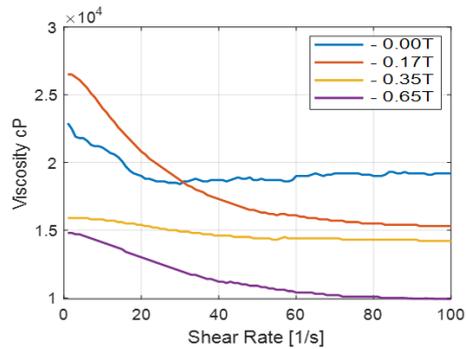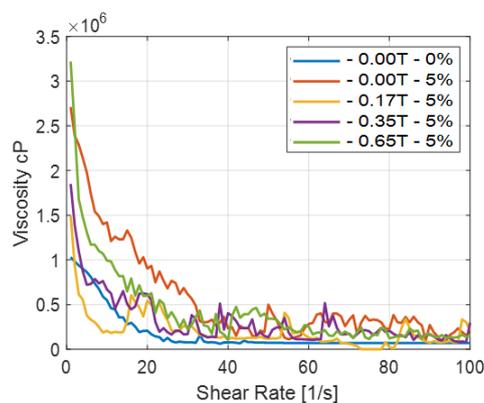
**Figure 7.** Variation of the viscosity of the heavy oil with respect to the shear rate and intensity of the magnetic field (MFS) maintaining a temperature of 70 °C



**Source:** Own production

Subsequently, we show the effect of the presence of magnetite at different MCI, temperatures and shear rates. Figure 8 presents the results at a temperature of 30 °C and with the addition of 5 %wt of magnetite NPs. There is evidence of an increase in the value of the viscosity of oil compared to the original oil. Although the presence of magnetic fields decreased the viscosities for all MFS levels, in none of the cases it was lower than in the original sample. This may be due to both temperature, and the effect on mobility caused by the magnetite nanoparticle concentration level and, therefore, its non-homogeneous distribution. A somewhat different situation is observed in Figure 9.

**Figure 8.** Variation of the viscosity of the heavy oil with respect to the shear rate and intensity of the magnetic field (MFS) maintaining a temperature of 30 °C. and a nanoparticle concentration of 5% wt



**Source:** Own production

**Figure 9.** Variation of the viscosity of the heavy oil with respect to the shear rate and intensity of the magnetic field (MFS) maintaining a temperature of 70 °C. and a nanoparticle concentration of 5% wt
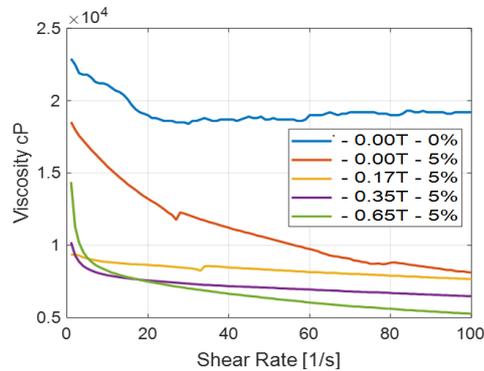
Figure 9 shows different rheological behaviors. In most cases the hydrocarbon behaves like a pseudo-plastic fluid. In the case of the original oil, this behavior reaches 20 s-1. From this point on, its behavior is that of a Newtonian fluid. The presence of the magnetite nanoparticle decreases the viscosity, and even prolongs the pseudo-plastic behavior to 100 s-1. As analyzed in previous curves, the presence of a magnetic field decreases viscosity as it increases. This decrease is due to the synergy between the temperature and the action of the fields in the magnetite nanoparticles [18].

## Heating of oil with microwaves

Table 2 shows the effect of the presence of magnetite NPs in oil. From these experimental data it can be seen that for the case of 0 %wt of NPs, the heating is minimal, and it reaches up to 36.4 °C. However, as the concentration of these nanoparticles is increased, temperatures increase up to 69 °C in 60 s at a concentration of 10 %wt. Similarly, it is appreciated that the NPs alone reach 15 s of radiation for a power of 800 W at 186 °C. It can be interpreted that the heat transfers between the NPs and the oil is very high due to the conductivity of the magnetite. The heating was not continued as to preserve the integrity of the optical fiber measurement system.

**Table 2.** Temperature variation of oil with the addition of magnetite nanoparticles NPs (in %wt) during heating in an electromagnetic cavity (800 W)

| Time (s) | Temperature (°C) oil with the addition of magnetite nanoparticles NPs | | | | | |
|---|---|---|---|---|---|---|
| | %wt | | | | | |
| | 0 | 0.5 | 1 | 5 | 10 | 100 |
| 0 | 24.7 | 24.7 | 24.7 | 24.7 | 24.7 | 25 |
| 5 | 24.8 | 29.2 | 30.2 | 32.0 | 37.8 | 40,7 |
| 15 | 26.8 | 32.7 | 34.5 | 35.2 | 43.0 | 94,6 |
| 30 | 28.4 | 38.0 | 38.2 | 42.0 | 58.7 | 194 |
| 45 | 32.2 | 47.8 | 47.8 | 52.2 | 66.0 | - |
| 60 | 36.4 | 58.2 | 58.2 | 60.2 | 69.0 | - |

**Source:** Own production

Figure 10 shows a Thermophotograph for a mixture of oil, but in this case in a cylindrical waveguide and after 60 seconds with an 800 W magnetron. The mixture reaches a temperature of 98.9 °C for a magnetite nanoparticle concentration of 10 %wt.

In this case, the difference of temperatures in relation to the microwave cavity occurs because the mixture receives all the energy generated by the magnetron, unlike the cavity, where not all the radiation affects the mixture, but it is reflected multiple times in the walls of the cavity, losing energy over time. Also, in this case, the sample has a very small volume compared to the total volume of the electromagnetic cavity. In this thermophotograph the effect of the instantaneous heating that occurs in the mixture can be appreciated, due to the effect of the magnetite NPs contained in the oil. Heating is also observed in the zones that are in contact with the mixture container. The areas of the waveguide that do not have direct contact with the container (glass) do not heat [19].

**Figure 10.** Thermography oil mixed with magnetite nano particles during heating in a cylindrical waveguide with 800 W power
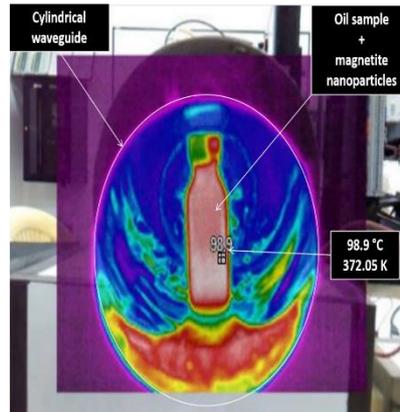


**Source:** Own production

## Heating in the presence of magnetic graphene oxide composite

Table 3 shows the effect of microwave heating for these mixtures according to the concentration of magnetic graphene oxide composite. An evident increase in temperature is observed, up to the point of reaching 137 °C in 60 seconds with a concentration of 10 %wt. Likewise, the heating of Fe3O4@GO in the absence of oil was evaluated. After 30 seconds it reached a temperature of 194 °C, but the radiation was discontinued, given the limitations of the fiber optic measurement system. Finally, comparing the effectiveness of the heating of the oil, in other words, the interaction of an electromagnetic field with a frequency of 2.45 GHz, either with magnetite NPs alone or with those of magnetic graphene oxide, it can be concluded that the temperatures reached with the latter are consistently higher for the same times of irradiation and concentration.

The effect of Fe3O4 @ GO, in the process of microwave heating of heavy oil, outlines it as a suitable alternative for the implementation of mobility improvement technologies of high viscosity hydrocarbons, applying electromagnetic fields.

**Table 3.** Increase of the temperature of the oil modified with the magnetic graphene M2 during its heating in an electromagnetic cavity

| Time (s) | Temperature (°C) oil modified with the magnetic graphene | | | | | |
|---|---|---|---|---|---|---|
| | %wt | | | | | |
| | 0 | 0.5 | 1 | 5 | 10 | 100 |
| 0 | 24.7 | 24.7 | 24.7 | 24.7 | 24.7 | 25,9 |
| 5 | 24.8 | 36.0 | 37.5 | 41.0 | 45.6 | 67,7 |
| 15 | 26.8 | 42.3 | 45.2 | 48.2 | 59.6 | 186 |
| 30 | 28.4 | 50.0 | 53.2 | 58.5 | 75.3 | - |
| 45 | 32.2 | 64.5 | 68.3 | 77.2 | 97.7 | - |
| 60 | 36.4 | 81.9 | 88.5 | 96.5 | 137.7 | - |

**Source:** Own production

## Conclusions

The rheological behaviors of heavy crudes are affected by magnetic fields, generating a marked decrease in their viscosity. This effect is proportional to the intensity of the magnetic field. In this research study, an unconventional application of magnetic nanoparticles (magnetite and Fe3O4@GO) was analyzed. These types of compounds interact with electromagnetic fields, due to their chemical composition, which provides them with a non-zero dipole moment, showing high sensitivity to electromagnetic fields. This interaction is reflected in the increase of its internal thermal energy and, therefore, generating instant heating in them during their release, as observed in the experimental results. This rapid increase in temperature generates a heat flow to the continuous phase, in this case, the heavy oil, that dissipates it and uses it as sensible heat, raising its temperature and decreasing its viscosity as a consequence. These observations are valid for both the type of electromagnetic cavity and the cylindrical waveguide applicator. On the other hand, when Fe3O4@GO is used, greater interaction is achieved and, therefore, faster warming than with magnetite NPs was observed, for the same concentrations and irradiation times. The temperature is homogenized much faster with this magnetic composite material due to its physicochemical properties, such as the high thermal conductivity of graphene. Also, part of this heating occurs due to the interaction of the electric field (of the electromagnetic wave) with the magnetic graphene oxide.

The results obtained hereby are the starting point for testing with other materials of a magnetic nature, in order to scale the experiments to pilot plant in the future.

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