Original Research Paper

Wettability and Interfacial Tension Alteration of Rare-Earth Doped Yttrium Iron Garnet under Influence of Electromagnetic Waves

[1]Lau Zhen Yin, [1]Lee Kean Chuan, [1]Beh Hoe Guan, [1]Hassan Soleimani and [2]Martin Weis

[1]*Fundamental and Applied Sciences Department, Universiti Teknologi PETRONAS, Bandar Seri Iskandar, Malaysia*
[2]*Institute of Electronics and Photonics, Slovak University of Technology, Ilkovicova 3, 81219 Bratislava, Slovakia*

Abstract: Yttrium Iron Garnet (YIG) doped with rare-earth elements have shown to alter the magnetic properties of garnet nanoparticles (NPs), which is believed to have direct influence on the wettability, interfacial tension and viscosity alteration of garnet nanofluids. In this study, $Y_{2.8}R_{0.2}Fe_5O_{12}$ (R = Lanthanum (La), Neodymium (Nd) and Samarium (Sm)) NPs were synthesized by using the sol gel auto-combustion method followed by annealing treatment at 1000°C for 3 hours. The $Y_{2.8}RE_{0.2}Fe_5O_{12}$ nanoparticles synthesized had grain size ranging from 100 to 200nm with high crystallinity properties. X-ray Diffraction peaks showed varying shifting with the size of the rare-earth ions in the $Y_{2.8}R_{0.2}Fe_5O_{12}$ crystal system, suggesting that structural distortion is due to replacement of bigger ions. Sm-YIG exhibited the highest magnetization saturation among all samples, with the value of 23.54 emu/g. Wettability data of $Y_{2.8}R_{0.2}Fe_5O_{12}$ (RE-YIG) nanofluids showed that oil-wetting contact angle has an overall reduction under the influence of electromagnetic wave. Whereas the interfacial tension and viscosity data showed that doped garnet nanofluids has lower value than that of garnet nanofluids, but the magnitude of interfacial tension and viscosity value decreased with the rare-earth ionic size. Sm-YIG also has the highest interfacial tension across all samples, as the stronger magnetization saturation may contribute to higher surface tension exerted on the oil-nanofluid interface.

Keywords: Rare-Earth, Garnets, Nanoparticles, Wettability

Introduction

Enhanced Oil Recovery (EOR) using Nanoparticles (NPs) method has been a major novel application research topic to further improve current oil recovery rate. According to Bennetzen and Mogensen (2014) and Peng *et al*. (2017), nanoparticles has shown promising solution to both upstream and downstream of oil and gas field, among which are the ability to alter wettability, Inter Facial Tension (IFT) and viscosity of the multiphase fluids within the reservoir formation. Injection of nanoparticles as nanofluids into reservoir formation under the influence of Electromagnetic (EM) wave, will activate the nanoparticles to cause thermodynamics disturbance within the reservoir structure (Bhatia and Chacko, 2011). Low dimensionality of nanoparticles enhances the magnetization or became paramagnetic, which makes alteration of surface force of fluids achievable (Pilkington

and Wuge, 2012) and improve the recovery rate through oil-water IFT reduction (Soleimani *et al*., 2018). To further improve oil recovery rate, the magnetic aspect of nanoparticles is crucial. Oscillating magnetization forces experienced by the magnetic nanoparticles has shown to initiate interfacial movement at the oil-water interface and altering the apparent viscosity of the nanofluids due to their magnetorheological and electrorheological properties, which improve the oil mobilization rate (Ryoo *et al*., 2012; Yahya, *et al*., 2014; Zaid *et al*., 2014; Zitha and Wessel, 2002). Soares *et al*. (2014) simulation study, high magnetic susceptibility nanoparticles were recommended as the magnetic perturbation caused by nanoparticles within the pore wall will facilitate oil blob detachment, thus improving the oil recovery rate. As studied by Soleimani *et al*. (2014), higher magnetic susceptibility nanoparticles shown better oil recovery rate in a core flooding test under EM wave irradiance.

A core flooding test with Yttrium Iron Garnet (YIG) nanofluid with the presence of EM waves has shown a yield of 43.64% (Soleimani *et al*., 2016), making them a promising candidate for the application. Rare-Earths (RE) doped YIG has shown promising ability to alter the magnetic properties of YIG, changing the structural, morphological and dielectric properties as well (Akhtar *et al*., 2017). However, the dopant alteration of the magnetic properties of YIG is dependent on the magnetic moment of both rare-earth ions and yttrium ions, observed neodymium doped YIG has a higher magnetic property than YIG (Wolf, 1961). Low dimensionality of YIG nanoparticles shows superparamagnetic behavior with high magnetic susceptibility as well (Sanchez *et al*., 2002).

In this study, Lanthanum (La), Neodymium (Nd) and Samarium (Sm) were doped into YIG at the concentration of 0.2 moles to form $Y_{2.8}R_{0.2}Fe_5O_{12}$ (RE-YIG) NPs. The samples were synthesized via sol-gel auto-combustion method and characterized by Field-Emission Scanning Electron Microscope (FESEM), Energy-dispersive X-ray spectroscopy (EDX), X-ray Diffractometer (XRD) and Vibrating Sample Magnetometer (VSM). The properties of RE-YIG nanofluids were evaluated through inverted sessile drop method for contact angle and IFT measurements. Viscosity of the RE-YIG nanofluids was performed using a rheometer. All nanofluids properties were investigated with or without the presence of EM waves at 100 MHz. The aim of the study is to relate the wettability, IFT and viscosity of RE-YIG nanofluid with the magnetization properties under the influence of EM waves.

Experimental Methodology

Materials and Methods

Yttrium (III) Nitrate ($Y(NO_3)_3.6H_2O$), Lanthanum (III) Nitrate, Neodymium (III) Nitrate, Samarium (III) Nitrate and Iron (III) Nitrate ($R(NO_3)_3.6H_2O$) were used as raw material to synthesize the RE-YIG nanoparticles via sol-gel auto-combustion method. Citric acid was added as chelating agent at the ratio of metal nitrates to citrates to be 1:1 (Vajargah *et al*., 2007) and the pH of the mixture solution was adjusted to 2 using ammonia solution (Vajargah *et al*., 2006). The resulting solutions were then heated at 90°C and stirred with magnetic stirrer until a viscous gel is formed. The gels were then heated up to 250-300°C for combustion and the burnt powder were then grounded. Annealing treatment at 1000°C for 3 h were followed. For complete annealing treatment and the formation of single-phase high crystallinity YIG nanoparticles, high annealing temperature is required (Akhtar *et al*., 2014; Baqueiro *et al*., 1997; Janifer *et al*., 2017). Unlike ball milling methods, the mixtures of Y_2O_3 and α-Fe_2O_3 powders in molar ratio 3:5 were ball-milled using a planetary mill and requires lesser amount of raw materials.

Table 1: Mass of each precursor material required

RE-YIG	Amount (g)			
	Y	RE	Fe	Citric Acid
YIG	15.57	0.00	27.37	20.83
Nd-YIG	14.32	1.17	26.97	20.52
La-YIG	14.34	1.16	27.01	20.55
Sm-YIG	14.30	1.19	26.93	20.49

However, the annealing temperature is higher (1200°C) which leads to bigger grain size (Ristić *et al*., 2003). Table 1 depicts the amount of precursor materials needs in order to yield 10 g of YIG NPs.

Characterization of RE-YIG Nanoparticles

Once RE-YIG nanoparticles were synthesized, they were sent to FESEM coupled with EDX to characterize their morphology and element composition. XRD were then used to evaluate the phase and crystallinity of the synthesized RE-YIG nanoparticles. As for the magnetization properties of the samples, they were characterized using VSM.

Evaluation of RE-YIG Nanofluids

0.01wt% of RE-YIG nanoparticles were mixed with 30000ppm brine solution to form nanofluid samples (Ogolo *et al*., 2012). Appropriate amount of surfactant was added to increase the suspension time of the nanoparticles within the fluid. Once the nanofluids were prepared, their densities were measured using density bottle. For the evaluation of wettability and IFT, an inverted sessile drop method was used with the help of Goniometer to measure the oil-wetting contact angle and IFT of the oil-nanofluids interface, both with and without the irradiance of EM wave (Yuan and Lee, 2013). Viscosity of nanofluids were measured using rheometer with and without EM irradiance as well.

Results and Discussions

Characterization of RE-YIG Nanoparticles

Field Emission Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy Characterization.

As shown in Fig. 1, the measure grain size of NPs synthesized were within the range of 100-200 nm. Along with the FESEM results, EDX data were collected and tabulated in Table 2. EDX results show the element composition of each sample matches well with the weight percent of rare-earths doped in YIG which is about 3% corresponding to the 0.2 mole concentration.

Referring to ICDD PDF2 RDB database, it was found that YIG with chemical formula of $Y_3Fe_5O_{12}$ (Reference code: 01-043-0507) with a cubic crystal system were formed along with small traces of YIG with chemical formula of $YFeO_3$ (Reference code: 01-086-0170) which is an orthorhombic crystal system.

Table 2: EDX weight percentage analysis of RE-YIG samples

Element	Samples (Weight %)			
	YIG	La-YIG	Nd-YIG	Sm-YIG
Y	34.37	31.44	33.19	33.38
Fe	29.89	30.95	26.05	30.18
O	35.73	34.11	38.15	32.64
R	0	3.49	2.61	3.8

Figure 2 shows the XRD patterns of YIG and RE-YIG NPs respectively, where *hkl* values were obtained from the database. High intensity peaks shown in Fig. 2 indicate high crystallinity properties of the NPs.

Based on the highest peak (420) of the spectrum, the 2θ, Full-Width Half-Maximum (FWHM) and d-spacing values crystalline size was calculated using Scherrer equation:

$$D = k\lambda \ / \ \beta cos\theta \tag{1}$$

Where:
D = The crystalline size
k = The shape factor (0.89)
λ = The laser source wavelength (1.5406Å for Cu Kα)
β = The FWHM value of the peak
θ = The Bragg's angle

$$d_{hkl} = a \ / \ \sqrt{(h^2 + k^2 + l^2)} \tag{2}$$

Where:
d_{hkl} that = The distance between the planes of atoms that give rise to the diffraction peaks
a = The lattice parameter of the crystal lattice
h, k and l = The Miller indices of the plane

Equation 2 was then used to calculate the lattice parameter of the synthesized samples. Value of k was chosen for 0.89 for spherical crystal. Both the measured and calculated values were tabulated in Table 3. It was observed that the crystalline size of the RE-YIG NPs was within the range of 45-53 nm. Besides that, we also found there has been a variation in 2θ and lattice constant value with different rare-earths dopants. It is speculated that the different ionic size of the rare-earth metals and yttrium contributes to such variation (Akhtar *et al.*, 2017). Lattice constant increased in the sequence of YIG, Sm-YIG, Nd-YIG and La-YIG, corresponding to the increase in ionic size of the metal ions.
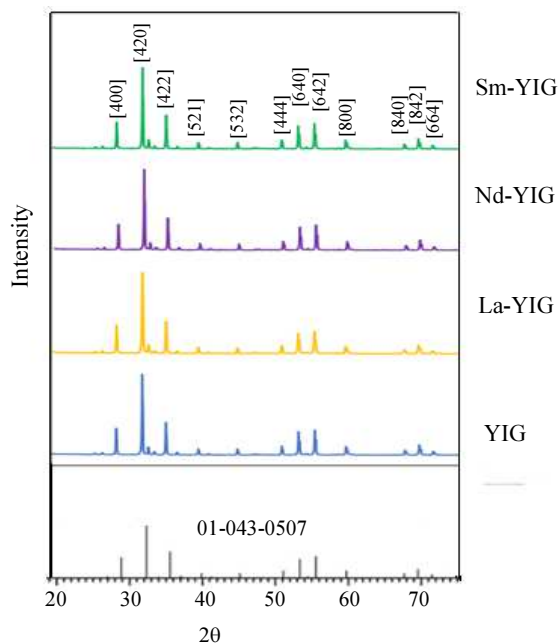
Vibrating Sample Magnetometer Characterization

Magnetic properties were studied by using VSM measurements performed at room temperatures. As observed in Fig. 3, RE-YIG shows soft magnetic material features, indicating the existence of superparamagnetic and single domain particles in the samples (Herzer, 1990; Kneller and Luborsky, 1963; Lu *et al.*, 2007).
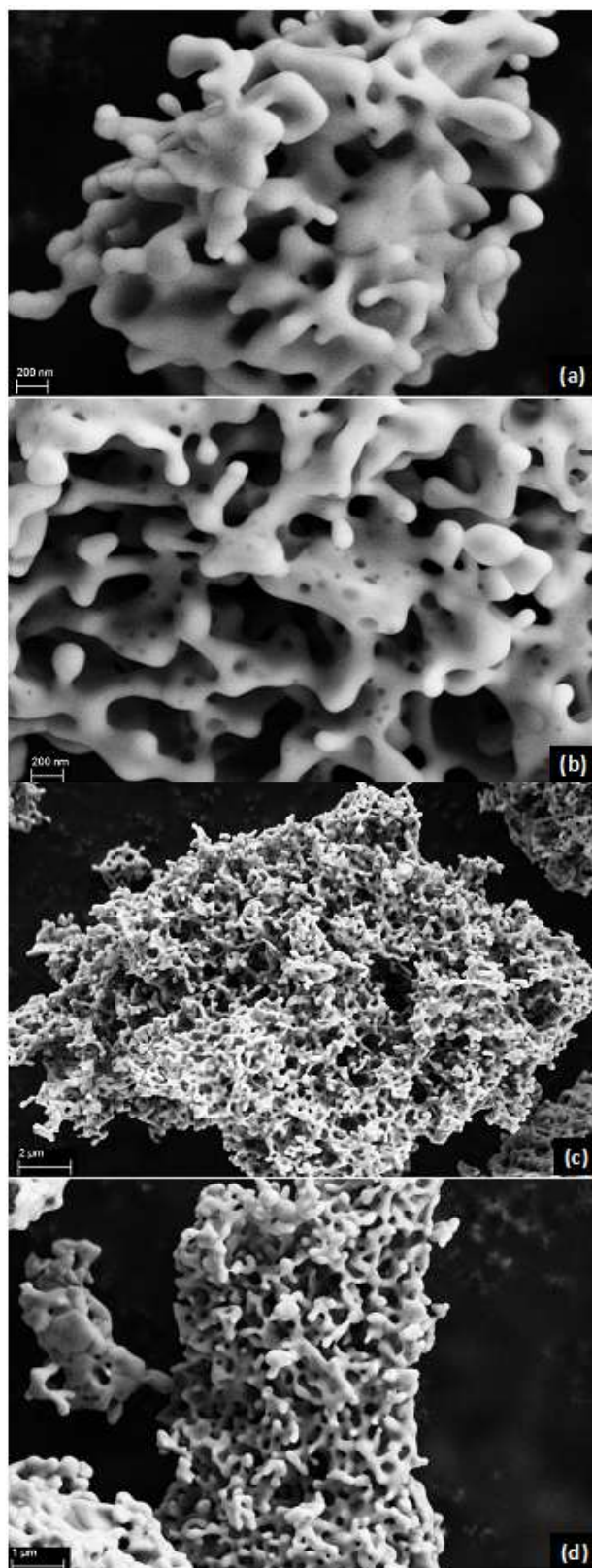
Fig. 1: FESEM Images for (a)YIG, (b) La-YIG, (c)Nd-YIG and (d)Sm-YIG

Furthermore, as tabulated in Table 4 and Fig. 4, the magnetic saturation, M_s of YIG decreases with the doping of La and increases with the doping of Nd and Sm, indicating Nd and Sm improve the parallel

magnetic moment alignment with Y ion in YIG structure (Uhm *et al.*, 2016; Wolf, 1961).

Magnetic remanence (M_r), coercivity (H_c) and saturation (H_s) has shown a general increase from YIG, La-YIG, Nd-YIG and Sm-YIG, following the order as per mentioned. The coercivity was observed to be increased as well. The M_s for Sm-YIG from our previous study was only 20.3 emu/g for x=1.0 (Lee *et al.*, 2019). However, RE-YIG NPs are still considered as soft magnetic materials with H_c less than 100 Oe.

Evaluation of RE-YIG Nanofluids

Wettability Alteration Analysis

Oil-wetting alteration were measured using inverted sessile drop contact angle measurement method with Goniometer. Based on Fig. 4, it is shown that La-YIG and Nd-YIG nanofluids have a slightly higher initial contact angle measurement than YIG nanofluid, whereas SM-YIG nanofluid shown significant reduction, without the irradiance of EM wave. It is suspected that the electrorheological properties of Sm-YIG nanofluids contributed to the significant difference. Overall, RE-YIG has shown a general reduction in oil-wetting angle, indicating a better performance of oil sweeping rate (Mohajeri *et al.*, 2015).

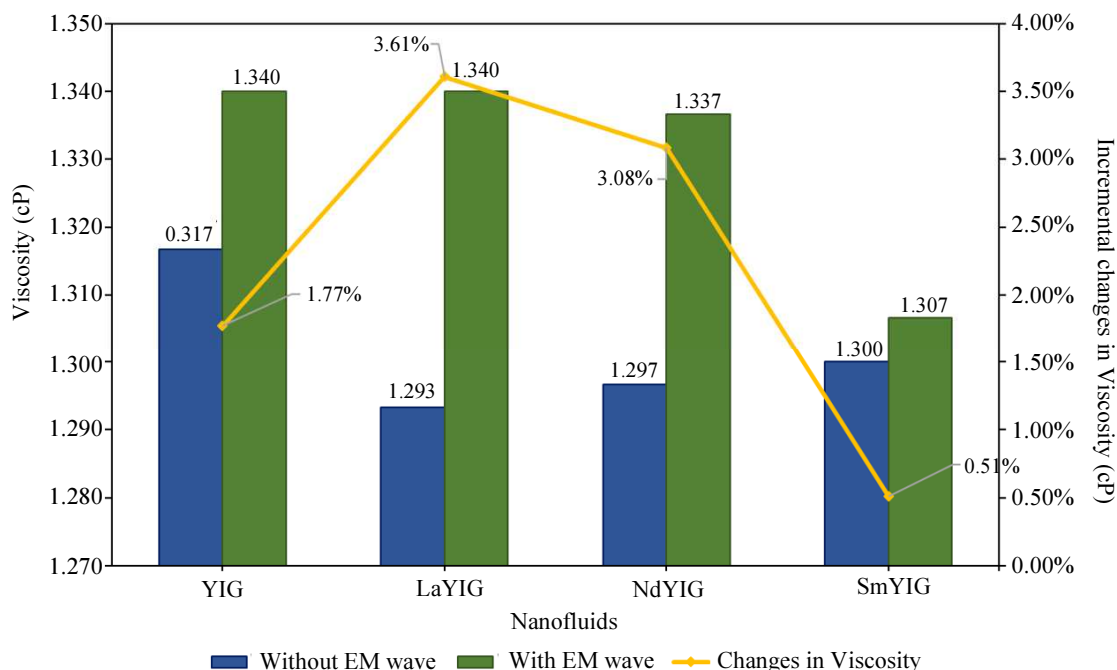**Fig. 2:** XRD pattern of RE-YIG nanoparticles with YIG XRD reference
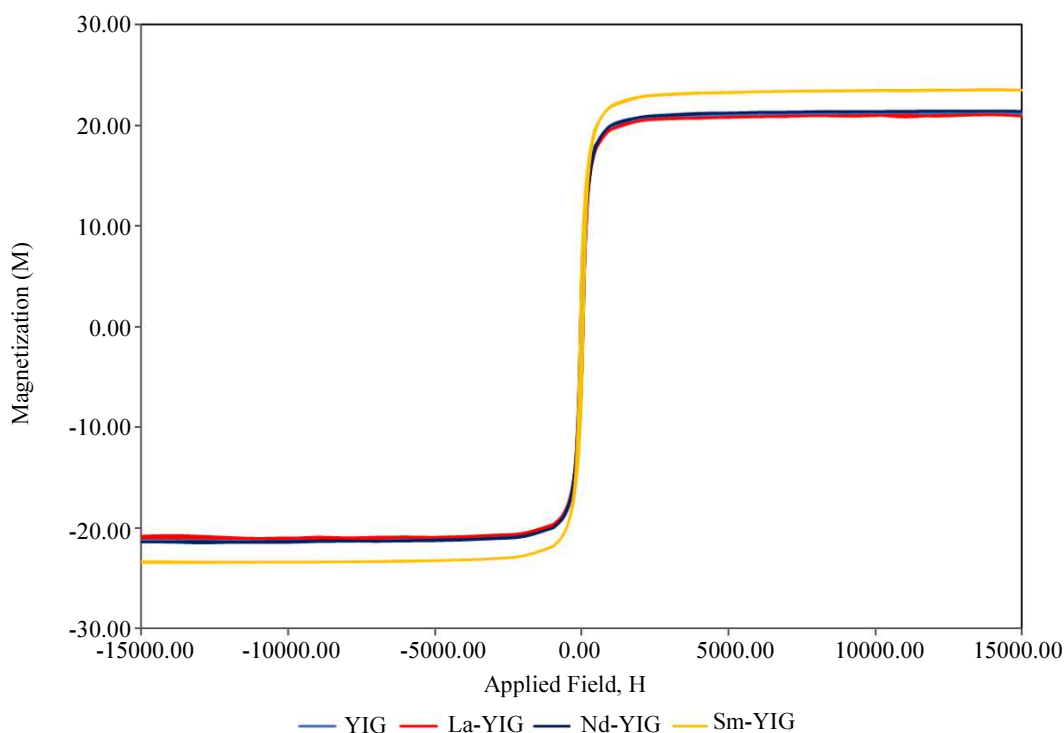
Fig. 3: Magnetic hysteresis loop of RE-YIG nanoparticles
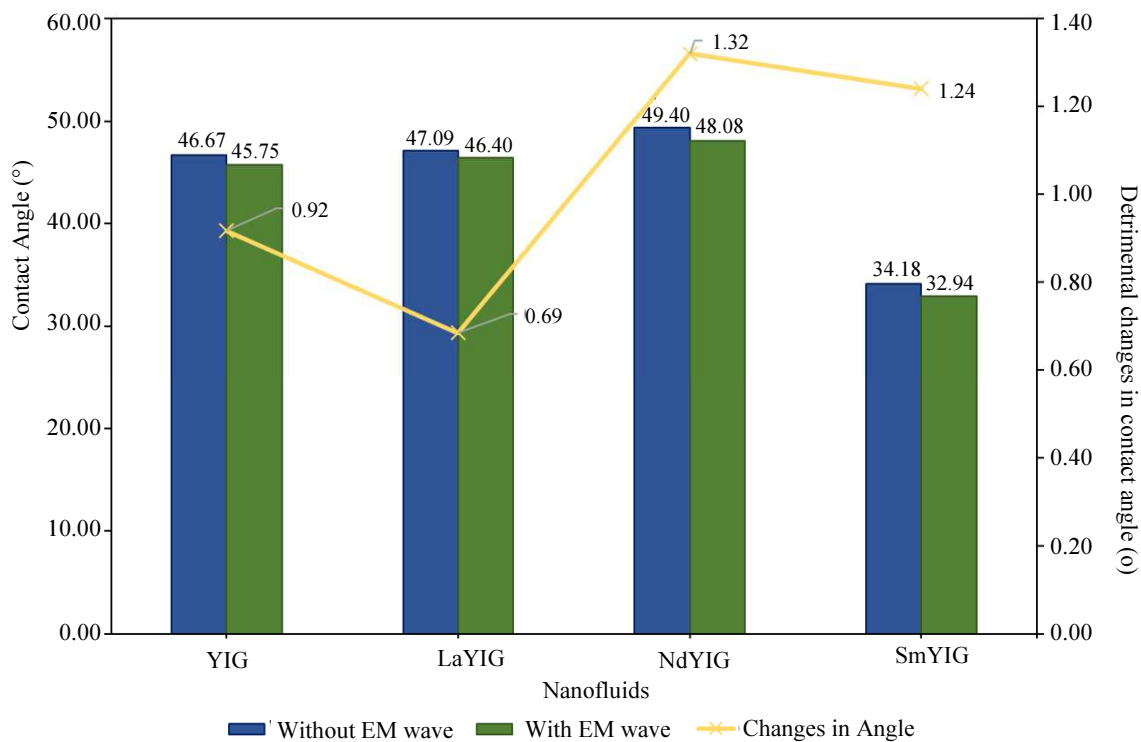
Fig. 4: Wettability alteration with RE-YIG nanofluids and influence of EM waves.
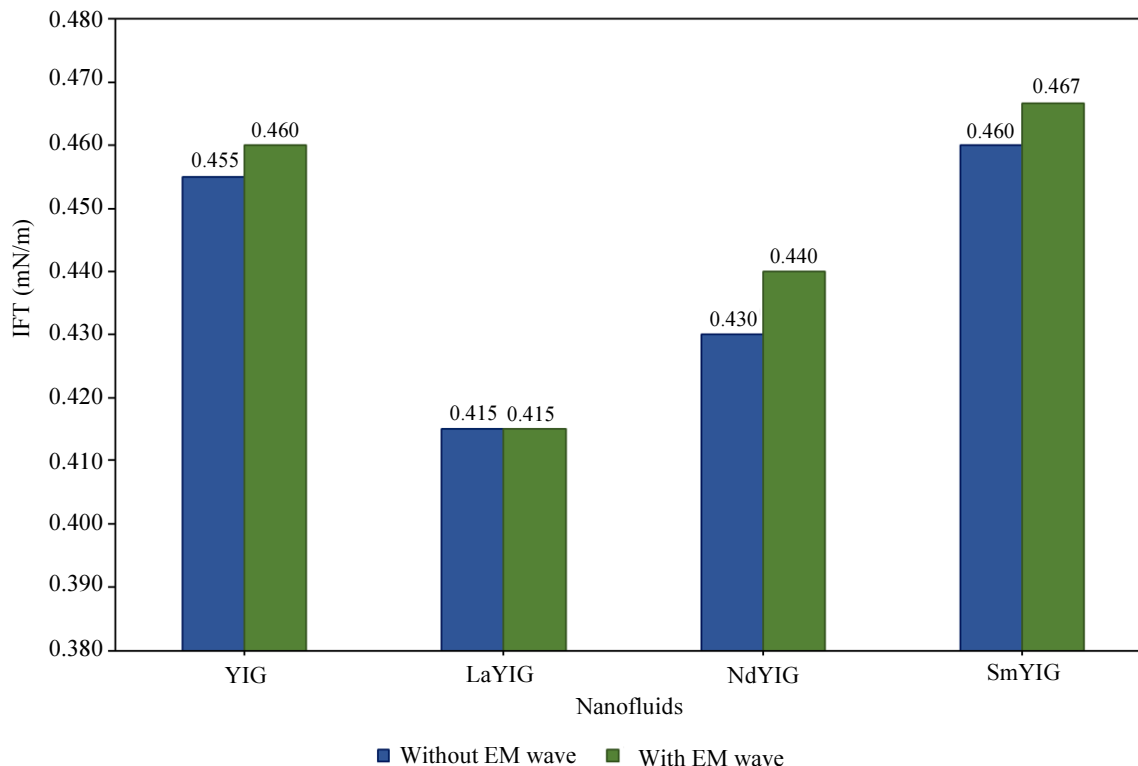
Fig. 5: IFT alteration with RE-YIG nanofluids and influence of EM waves

Fig. 6: Viscosity alteration with RE-YIG nanofluids and influence of EM waves

Table 3: XRD parameter of RE-YIG nanoparticles based on (420) peak

Samples	2θ	FWHM (°)	Crystalline size, D (nm)	d-spacing (Å)	Lattice constant, a(nm)
YIG	32.324	0.163	50.058	2.767	12.374
La-YIG	32.272	0.182	44.879	2.771	12.393
Nd-YIG	32.298	0.158	51.870	2.769	12.383
Sm-YIG	32.324	0.154	53.062	2.768	12.377

Table 4: VSM analysis of RE-YIG nanoparticles

Samples	Magnetic parameters			
	M_s (emu/g)	M_r (emu/g)	H_c (Oe)	H_s (Oe)
YIG	21.30	4.45	37.09	1395.97
La-YIG	21.10	4.41	39.68	1429.42
Nd-YIG	21.50	5.25	46.05	1464.67
Sm-YIG	23.54	6.02	47.03	1503.88

Interfacial Tension Alteration Analysis

Based on Fig. 5, it is observed that the oil-nanofluid IFT results does not shows significant changes under the influence of EM wave. However, the IFT value does differ for different rare-earth dopants. La-YIG has the lowest IFT value whereas Sm-YIG has the highest IFT value. The increment in IFT value correspond to the increment of rare-earth metal ionic size, suggestive that the structural properties of RE-YIG is one of the contributors to the IFT alteration, where further studies is required.

Viscosity Alteration Analysis

As shown in Fig. 6, a similar trend of viscosity of RE-YIG without the influence of EM wave were observed as in IFT section, further suggesting that the structural properties of RE-YIG should be investigated. Under the irradiance of EM wave, each RE-YIG nanofluids shows different magnitude in changes of viscosity as well, with La-YIG having the largest increment, which is 3.61% followed by Nd-YIG. It is suspected that the structural distortion experienced by the YIG structure due to the doping of bigger ionic size metal ions and the magnetic coercivity of RE-YIG particle, contributes to the variation of incremental changes in viscosity.

Conclusion

A high crystallinity $Y_{2.8}R_{0.2}Fe_5O_{12}$ (R = La, Nd, Sm) with grain size ranging between 100-200 nm were synthesized successfully via the sol-gel auto-combustion method. XRD results showed variation in lattice parameter, suggesting the ionic radii of rare-earth metal ions contributed to structural distortion within the YIG structure which increased the overall lattice constant of the crystal structure. Nd-YIG and Sm-YIG shown improvement in YIG magnetization saturation value, with Sm-YIG being 23.54 emu/g. Wettability data showed general improvement in reduction of oil-wetting contact angle, suggesting potential improvement in oil sweeping rate. Both IFT and viscosity data showed that the dopant of the YIG nanoparticles contributes to the changes in IFT and viscosity value without the irradiance of EM waves. Under the influence of EM waves, IFT value does not altered significantly. La-YIG showed the highest increment in viscosity value at 3.61% followed by Nd-YIG, YIG and Sm-YIG. Both structural and magnetic properties of RE-YIG are suspected to contribute to such changes, where further research studies are recommended.

In conclusion, the doping of rare-earth metal ions into YIG structure will change the various parameters and properties of the YIG nanoparticles and hence alter the IFT, wettability and viscosity when applied as nanofluids in EOR applications.

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Author's Contributions

Lau Zhen Yin, Lee Kean Chuan, Beh Hoe Guan and Martin Weis: Conceptualization.

Lee Kean Chuan and Beh Hoe Guan: Funding Acquisition/Resources.

Lau Zhen Yin, Lee Kean Chuan and Beh Hoe Guan: Investigations/Methodology/Visualization/Writing.

Lee Kean Chuan, Beh Hoe Guan and Martin Weis: Supervision/Validation/Review:

Hassan Soleimani: Superivision/ Valdation.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

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