Heat transfer and entropy generation analysis of hybrid graphene/Fe₃O₄ ferro-nanofluid flow under the influence of a magnetic field

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ABSTRACT
The heat transfer characteristics and entropy generation rate of hybrid graphene-magnetite nanofluids under forced laminar flow that subjected to the permanent magnetic fields were investigated. For this purpose, a nanoscale reduced graphene oxide-Fe₃O₄ hybrid was synthesized by using graphene oxide, iron salts and tannic acid as the reductant and stabilizer. The thermophysical and magnetic properties of the hybrid nanofluid have been widely characterized and thermal conductivity has shown an enhancement of 11%. The experimental results indicated that the heat transfer enhancement of hybrid magnetite nanofluid compared to the case of distilled water was negligible when no magnetic field was applied. Additionally, the heat transfer characteristics have been improved significantly under magnetic field. The outcome of the analysis shows that the total entropy generation rate was reduced up to 41% compared to distilled water. It appears that these magnetic hybrid nanofluids can function as good alternative fluids in the magnetic thermal engineering systems.

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1. Introduction
The investigation of the forced convective heat transfer has been attracted in many research works due to its practical relevance in turbo-machinery, heat exchangers, air conditioning systems, refrigeration and chemical reactors [1–3]. More inventive and efficient cooling technologies and fluids are now required to support this technological development [4,5]. Nanofluids, as a new class of working fluids, are a challenge for thermal fluid sciences that provided by nanotechnology. Due to their excellent thermophysical properties, nanofluids find many applications in the heat transfer enhancement [6–8]. The magnetic materials as a new class of nanoscience materials, have been used in the part of daily life since ancient eras [9–12]. Nanomagnetic materials with special properties are progressively replacing by the other material in the specific applications. Therefore, the researchers need to understand their properties at the fundamental level [10,13]. The magnetic particles can be either ferromagnetic materials including iron or cobalt, or ferromagnetic materials including magnetite (Fe₃O₄). Among them, Fe₃O₄ nanoparticles have been paying much attention because of their low toxicity and good biocompatibility [14–17]. However, the research has shown that the functionalization graphene oxide with Fe₃O₄ nanoparticles (Fe₃O₄-GO) holds vast potential in nanofluid because of their large saturation magnetization value and heat transfer capability. Graphene based materials such as graphene oxide (GO) have shown wide applications in composite materials and heat transfer applications [18–29]. A number of studies have been carried out on heat transfer properties of magnetic nanofluid and it was found that heat transfer characteristics under magnetic field are in general better than those of base fluids. Table 1 is summarized some recent work on convective heat transfer of magnetic nanofluids.

Even though, the performance of any thermal management systems such as heat exchangers can be evaluated by the other thermodynamic performance analysis, including entropy generation rates [32–34]. The heat transfer improvement is a non-monotonic parameter and maximizing of this ratio does not really show the improvement of thermodynamic performance due to irreversibilities involved. Therefore, these irreversibilities work can be measured by entropy generation rates of a heat transfer system [35]. The irreversibility analysis of different systems was investigated by several researchers [35–37] and they have shown that this is a powerful tool to decide which process or installation
is more efficient. The irreversibility analysis (entropy generation rate) is the measurement of entropy that created by the irreversibilities such thermal and the frictional loss [1].

This research work has presented a synthesize method for a hybrid reduced graphene oxide (rGO)-Fe₃O₄ nano fluid with TA (tannic acid) as the reductant and stabilizer. The stability, thermal conductivity and viscosity of magnetic nano fluid have been tested at several conditions. Based on the literatures overview, the energy management is analyzed by studying the entropy generations due to the proportions of the irreversibilities works. The aim of the present study is to analyze entropy generation and the advantage or disadvantage of rGO-Fe₃O₄ nano fluids over base fluids in the laminar flow regime will be investigated thoroughly.

2. Experimental method and process

2.1. Materials

Graphite flakes were purchased from Ashbury, Inc. Hydrochloric acid (HCl, 37%), Sulfuric acid (H₂SO₄, 98%), potassium permanganate (KMnO₄, 99.9%) and hydrogen peroxide (H₂O₂, 30%) were purchased from Merck Chemicals. Tannic acid (TA), FeCl₂·4H₂O and FeCl₃·6H₂O were purchased from Sigma-Aldrich.

2.2. Material preparation

Water soluble graphene oxide (GO) that used in this experiment was synthesized by a simplified Hummers’ method [38,39]. For the hybrid magnetic nano fluid preparation, 50 mg of GO was mixed with 100 mL distilled water (DW) and then placed in a three-neck round flask. 1.3 g of FeCl₃·6H₂O and 475 mg of FeCl₂·4H₂O predissolved in 20 mL DW were added into the flask, which was continuously stirred and purged with high-purity N₂ (Nitrogen gas) for 3 h. After addition of tannic acid, pH of mixture was adjusted to 10 using ammonia solution. Then, the mixture was heated up to 80 °C and refluxed in a nitrogen atmosphere for 8 h. Subsequently, the hybrid rGO-Fe₃O₄ was collected after washing with DW for several times. The blackish precipitates were added into the beaker and homogenized by stirring and the concentrations of magnetic nano fluid were maintained at 0.5 wt%.

2.3. Characterization method

Transmission electron microscopy (TEM) measurements were conducted on a CARL ZEISS-LIBRA120 microscope. X-ray diffraction (XRD)
patterns were measured on an Empyrean PANALYTICAL diffractometer. Room temperature magnetization measurements were carried out using a Lakeshore 7400 series vibrating sample magnetometer (VSM). Fourier transform-infrared (FT-IR) absorption spectra of the composites were recorded using a Bruker FT-IR (Bruker Tensor 27) spectrometer at room temperature in the range 4000–1000 cm⁻¹ using FT-IR mode. An X-ray photoemission spectrometer, XPS (PHI 5400 ESCA) with an Al-Kα (hv = 1486.69 eV) X-ray source was used to identify bonding of the elements. The rheological behavior of nanofluids was measured using a Thermo Scientific (KD2 Pro, Decagon Devices, Inc., USA) was used to measure the thermal conductivity. The light transmission method was used to study of stabil-}

### Table 3

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimensions</td>
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<tr>
<td>Maximum operating temperature</td>
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</tr>
<tr>
<td>Surface field</td>
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</tr>
<tr>
<td>Residual flux density (Bmax)</td>
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<tr>
<td>Maximum energy product (Bmax)</td>
<td>42 MGOe</td>
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<tr>
<td>Relative permeability of NdFeB permanent magnet (µr)</td>
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</tr>
<tr>
<td>Magnetic permeability of free space (µ0)</td>
<td>4π x 10⁻⁷</td>
</tr>
</tbody>
</table>

2.4. Estimation of heat transfer coefficient

Fig. 1 shows the laminar flow system that employed to measure the convective heat transfer coefficient and it consists of a flow loop, a heating unit, a cooling unit and measuring instruments. The details of experimental setup are presented in the Table 2.

An investigation of the heat transfer behavior was done by evaluating the heat transfer coefficient and the surface temperature. The measurements were performed under different laminar bulk velocities ranging from 0.05 to 0.3 L/m (equal to the Reynolds number between 294 and 1765). The effect on the convective heat transfer coefficient was described under a constant heat flux of 3.5 kW/m², an inlet temperature of 30 °C and various magnetic field arrangement [40].

The effect of the permanent magnetic field as well as the arrangement and number of magnets on entropy generation rates of the hybrid magnetite nanofluid have been investigated by adding magnetic bar on the surface of test tube. The magnetic field was controlled by NdFeB, grade 42 block (permanent magnets) that purchased from K&J Magnetics. The specifications of the permanent magnets are summarized in Table 3.

The magnetic bars were used to apply permanent magnetic field along the test section, as shown in Fig. 2. These magnets were directly positioned on the both side of the test section at certain points, so that a perpendicular magnetic field was applied to the rGO-Fe₃O₄ nanofluids flow.

2.5. Data reduction

The accuracy and reliability of the experimental set up have been verified by the Shah correlation in laminar fluid flow regime with an error rate of < 6.3% for the DW as a base fluid [1]. The convective heat transfer coefficient (h) has been calculated as follows:

\[ h(x) = \frac{q}{T_b(x) - T_w(x)} \]

The convective heat transfer coefficient of a fluid can be expressed in the form of the Nusselt number (Nu), which was calculated as:

\[ Nu(x) = \frac{h(x)D}{k} \]

Also, with a steady state one dimensional energy balance in the tube, the inner wall temperature was calculated based on the measured outer wall temperature, which was defined as:

\[ T_w(x) = T_{w,0} - \frac{q}{2\pi L K_w} \ln \left( \frac{D}{D_0} \right) \]

In conjunction with the heat transfer measurements, the viscous pressure loss was measured along the test section for DW and magnetic nanofluids. The experimental results were compared with the pressure loss predictions:

\[ f = \frac{\Delta P}{L \left( \frac{\rho v^2}{2} \right) } \]

The friction factor results for DW are validated by the Hagen–Poiseuille correlation [41] and the error rate of < 5.4% was observed.

### Table 4

<table>
<thead>
<tr>
<th>Variable name</th>
<th>Uncertainty analysis</th>
</tr>
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<tr>
<td>NuLocal</td>
<td>± 7%</td>
</tr>
<tr>
<td>TwLocal</td>
<td>± 9%</td>
</tr>
<tr>
<td>hLocal</td>
<td>± 10%</td>
</tr>
<tr>
<td>f</td>
<td>± 10%</td>
</tr>
</tbody>
</table>
Additionally, the friction factor was verified by \( f = 64/Re \) equation and it was shown to be accurate within ±6% error.

The total entropy generation rate \( (\dot{E}_{\text{gen}}) \) is the sum of the frictional entropy generation rate \( (\dot{E}_{\text{gen,fl}}) \) and the thermal entropy generation rate \( (\dot{E}_{\text{gen,th}}) \) and it can be determined as follows [37]:

\[
\dot{E}_{\text{gen}} = \dot{E}_{\text{gen,th}} + \dot{E}_{\text{gen,fl}}
\]

\[
\dot{E}_{\text{gen,th}} = \frac{\pi d^4 L q - 2}{k_n T_a T_{av}}
\]

\[
\dot{E}_{\text{gen,fl}} = \frac{32 m_f^3 \rho}{\mu_f n^2 d^3 T_{av}}
\]

\[ T_{\text{avg}} = \frac{T_{\text{in}} - T_{\text{out}}}{\ln \left( \frac{T_{\text{in}}}{T_{\text{out}}} \right)} \] (8)

An uncertainty analysis of the measured data and other selected relevant parameters was carried out using the method of Taylor [42] and the data reduction process. The results are presented in Table 4 and show the maximum uncertainty ranges for each parameter.

3. Results and discussion

3.1. Characterizations and properties of magnetic nanofluid

The structure of the prepared rGO-Fe\(_3\)O\(_4\) nanocomposite was examined by means of TEM. A large amount of Fe\(_3\)O\(_4\) nanoparticles distributed on rGO sheets as demonstrated in Fig. 3(a) and (b). The as-prepared
ferromagnetic nanoparticles are mono-dispersedly anchored onto the rGO nanosheets. The chemical composition of Fe₃O₄ nanoparticles, investigated by an EDS spectrum. Fig. 3(c) indicates that the main contributions from elemental C, Fe and O only, hence confirming the chemical purity of the sample. The peak corresponding to Cu in the EDS spectrum arises from the TEM grid used for preparing the TEM specimen.

The FT-IR absorption spectra of the dried GO and rGO-Fe₃O₄ composite are shown in Fig. 4(a). As shown in Fig. 4(a), a new IR band at 567 cm⁻¹ in the spectrum of the rGO-Fe₃O₄ hybrid is because of the Fe-O vibration of Fe₃O₄ nanoparticles, which suggested that Fe ions have been successfully oxidized into Fe₃O₄ nanoparticles. The IR bands of carbonyl (1730 cm⁻¹), epoxy (1230 cm⁻¹) and hydroxyl (3450 cm⁻¹) groups of the graphene oxide were considerably diminished due to the reduction process to produce rGO-Fe₃O₄ hybrid. Fig. 4(b) represents the XRD profiles of GO and rGO-Fe₃O₄. The GO indicates a strong peak located at 2θ = 9.7°, liaising with the (002) interplanar spacing of 0.91 nm. For the magnetic graphene, the peak at 2θ = 24.5° can be observed, with the disappearance of 2θ = 9.7°, confirming the successful reduction of the GO in the reaction. The pattern of rGO-Fe₃O₄ displayed obvious diffraction peaks of Fe₃O₄ and the peak positions and relative intensities match well with the standard XRD data for magnetite (JCPDS file #01-1111). The average crystallite size of Fe₂O₃ on GO surface was calculated to be ~10.9 nm by using the Debye–Scherrer formula (\(D = \frac{0.9\lambda}{\beta\cos\theta}\)), where, D is the crystallite size, \(\lambda\) is the wave length of X-ray, \(\beta\) is the value of full width at half maximum and \(\theta\) is the Bragg’s angle) which was lower than crystallite size of bare Fe₂O₃ ~13 nm.

Fig. 5 shows XPS survey scan of the GO and rGO-Fe₃O₄. The survey scan of rGO-Fe₃O₄ ascertains the existence of carbon, oxygen, and iron, related to the Fe₃O₄ nanoparticles and the underlying rGO sheet. The formation of magnetite nanoparticles is also corroborated by the narrow scan of Fe2p spectrum (Fig. 5(b)), in which the peaks at 723 and 711 eV are specs of Fe 2p₁/₂ and Fe2p₃/₂ of Fe₃O₄. Fig. 5(c) and (d) indicates the narrow scan of the C1s spectrum of GO and rGO-Fe₃O₄, respectively. For both samples the peaks centered at binding energies of 284.2, 286.1, 288.1 and 289.3 eV were assigned to the C-C/C=C, C-O, C=O and O=C=O oxygen and carbon containing bonds, respectively. During the reduction process by TA and Fe ions, the peak intensities for most of the oxygen-containing groups, exclusively C=O, reduced significantly due to the redox reaction confirming the results from FT-IR analysis.

![Fig. 5](image)

**Fig. 5.** (a) XPS survey scan of GO and rGO-Fe₃O₄; (b) narrow scan of Fe2p spectrum for rGO-Fe₃O₄; (c) C 1s spectra of GO and (d) C 1s spectra of rGO-Fe₃O₄.

![Fig. 6](image)

**Fig. 6.** (a) Magnetization hysteresis loops of rGO-Fe₃O₄ at room temperature (25 °C). The inset shows the photograph of rGO-Fe₃O₄ nanofluid and its response to an external magnetic field, (b) relative concentration (C/C₀) of nanofluids with sediment time.
The field dependence of magnetization (M − H measurement) hysteresis loop of rGO-Fe₃O₄ at room temperature is shown in Fig. 6(a). The results show that the magnetic saturation value (Mₛ) of hybrid magnetic particles was 45.9 emu/g. The stability test of a nanofluid is one of important matters for heat transfer application and the UV–vis test was extensively used to evaluating the relative concentration of the nanofluids. Fig. 6(b) illustrates the relative concentrations of rGO-Fe₃O₄ nanofluid and exhibited a sedimentation of 5.2% after 60 days that could show high stability of rGO-Fe₃O₄ nanofluid. It shows that the effect of tannic acid on the particles that could help to prevent their agglomeration and sedimentation of particles under van der Waals attraction.

Based on the theory, magnetic particles have a complex behavior including Brownian or Neel relaxation models and studies on these aspects are important for various technological applications. Magnetic nanofluids with large pyromagnetic coefficient, high saturation magnetization, low Curie temperature, high specific heat and low viscosity could be good candidates for thermo-magnetic systems [43] and rGO-Fe₃O₄ hybrid nanofluid could be a perfect nanofluid. As shown in Fig. 7(a), the thermal conductivity of rGO-Fe₃O₄ nanofluid enhanced up to 11% that was proposed by Maxwell theory [44]. Therefore, the thermal conductivity enhancement is due to the increases of Brownian motion of particles at higher temperatures and more violent in Brownian motion leads to increase the thermal conductivity.

Fig. 7(b) illustrates the viscosity versus temperature of mentioned magnetic nanofluid and it was shown the characteristic behavior of a definite liquid, which was further confirmed by the rheological measurements. This nanofluid exhibited the Newtonian behavior and viscosity of nanofluid decreased with rise of temperature and this behavior was observed above the shear rate of 200 s⁻¹ with negligible effect on the viscosity against shear rate as there were no significant interactions among the magnetic particles.

It must be mentioned that the thermal conductivity analysis is not the only effective parameters of nanofluid ability in the heat transfer system and heat transfer and entropy generation analysis could define the efficiency of the nanofluid. The efficiency of the nanofluids is depending on the fluid thermal properties and the flow regime (laminar or turbulent modes). Fig. 8(a) shows the average Nusselt number of
DW and rGO-Fe3O4 nanofluid versus fluid velocities in the thermal developing region. The results show that the Nusselt number of magnetic nanofluid is less than the DW. The reason is the Nusselt number waslargely influenced by the particle’s Brownian motion and the thermophysical properties of the nanoparticles. In this case, the heat transfer coefficient enhancement ($\geq 4\%$) of magnetic nanofluid is less than thermal conductivity enhancement ($\approx 11\%$). Based on the Nusselt number formulation (Eq. (2)), the Nusselt number of magnetic nanofluid will be less than the base fluid. As shown in Fig. 8(b), the average heat transfer coefficient increases by the fluid velocities and DW and magnetic nanofluid in the absence of magnetic field have the almost same trend for the heat transfer coefficient. The results indicated that the convective heat transfer coefficient increased up to 4% and it did not exceed the thermal conductivity enhancements. This phenomenon could be defined as a macroscopic parameter of a nanofluid that is flowing across a solid surface with high temperature gradient in a laminar regime [45].

In order to verify the performance of nanofluid, the entropy generation of nanofluid was calculated. The entropy generation analysis of nanofluids is known as a useful application to analyze thermal design optimization through minimizing it, and then better working conditions can be performed for heat exchangers [46–48]. Fig. 8(c) shows the total entropy generation rate of DW and rGO-Fe3O4 nanofluid in the absence of magnetic field. The results indicated that the total entropy generation increases from flow velocities of 0.05 m/s to 0.1 m/s and decreases above a velocity of 0.1 m/s due to the reducing the temperature difference between wall and bulk fluid and enhancement of heat transfer between the wall and the fluid. Also, it shows that the show that the total entropy generation decrease compare to the case of DW. Entropy generation shows the irreversibility of the system, therefore by reducing it, a more efficient system is obtained.

Fig. 8(d) shows the increment of pressure drops for different ranges of flow velocity. The rise in pressure drop was based on the viscous drag effects of the rGO-Fe3O4 nanofluids and this phenomenon could be validated by the pressure drop equation (Eq. (4)) [40]. It also can be explained by the combination of the flow rate and temperature, which could lead to decrement in viscosity of fluid and pressure drops. Additionally, frictional entropy generation has a minor effect, in the form of a slight reduction, on the total entropy generation because the maximum value of the frictional entropy generation remains $<1$ for all velocities and concentrations.

Further investigations have been carried out on the entropy generation of the hybrid magnetite nanofluid to examine the effect of the permanent magnetic field as well as the number and arrangement of magnetic bars. As shown in Fig. 9, the magnetic particle has chainlike structure in the flow pattern under constant magnetic field and these chainlike structures were happening without clumping in the carrier fluid flow [49]. The size, length and pattern of this chain are related to the magnetization of particles and magnetic bars flux density. Therefore, increase of the heat transfer may be expected due to the increase in the size and patterns of the chainlike particles under constant magnetic field. The entropy generation reduction of magnetic nanofluid in the presence of magnetic field can be explain by the interactions between the nanofluid flow and the aggregations of magnetic particles that formed at the inner surfaces of the tube corresponding to each magnetic bar location. Entropy generation reduces when using nanofluids due to their improved thermal transport mechanism.

The influence of magnetic field on entropy generation rates is shown in Fig. 10. Fig. 10(a) shows that the thermal entropy generation rate of magnetic nanofluid decreases with applying magnetic field and the thermal entropy generation decreases with velocity. Fig. 10(b) shows that frictional entropy generation rate and it was increased with velocities and the enhancement of frictional entropy generation was extremely low and can be neglected. Additionally, Fig. 10(a) shows that the magnetic forces could increase the heat transfer and best one was seen in case (I) at a fluid velocity of 0.3 m/s. Additionally, it shows that the total entropy generation rate was reduced up to 41% compared to distilled water (Fig. 11). There are various factors that can explain the enhancement of heat transfer after applying the constant magnetic field. The reason is thermal conductivity and viscosity increased due to the accumulation of magnetic particles near the inner wall surface and it led to change in the thermal boundary layer thickness. At lower

![Fig. 9. Schematic of magnetic field on the flow pattern of fluid flow inside tube.](image)

![Fig. 10. Entropy generation rate as a function of velocity for rGO-Fe3O4 nanofluid for several cases: (a) thermal; (b) frictional.](image)

![Fig. 11. Total entropy generation rate of rGO-Fe3O4 nanofluid as a function of velocity for several cases.](image)
velocity, the magnetic particles move lower, therefore, the magnetic field has time to exert enough forces on the magnetic particles that causes transverse motion and then dragging magnetic particle toward the tube wall surface [9]. Accordingly, at higher velocity, the positive effects of thermal boundary layer disturbance compare with the negative effects of the viscosity are significant and it could enhance the heat transfer. This again indicates that nanofluid cooling provides a better efficiency than pure water, as entropy generation is related to energy loss. Fig. 11 shows the total entropy generation rate of rGO-Fe3O4 nanofluid with the fluid velocities for several cases. It found that the total entropy generation decreases as the rise of fluid velocities and the effect of frictional entropy generations were low (the maximum value of frictional entropy generations were < 1 in all cases). According to the fluid dynamic theory [9], the heat transfer enhancement (minimizing the entropy generation rates) of a nanofluid is a relative to the preoperational of \( \frac{k}{\delta_t} \) (thermal conductivity/thinness of boundary layer). Additionally, the movement of magnetic particles and their attraction to the tube wall surface due to the magnetic field could be reduced the thermal boundary layer thickness and also thermal conductivity enhancement of magnetic nanofluid due to the applied magnetic fields, led to an increase in heat transfer characteristic [50]. In addition, the efforts in developing flow and heat transfer correlations as well as in analyzing the entropy generation of nanofluid flow were discussed. Nanofluids seem to provide better heat transfer performance than base fluids with little penalty in pressure drop. However, for a better understanding of nanofluid convective heat transfer, further experimental and numerical studies are needed to identify the heat transfer mechanisms and provide accurate predictions.

Additionally, the thermal efficiency analysis can be used to evaluate the rGO-Fe3O4 nanofluids performance for thermo-magnetic heat transfer system and is defined as follow [10]:

\[
\eta_{th} = \frac{h_{avg} \cdot \theta_{avg}}{h_{avg} \cdot \theta_{avg}}
\]  

The effect of magnetic field arrangements on the thermal efficiency index is shown in Fig. 12. It shows that the maximum thermal efficiency index was only 1 in the absence of the magnetic field. After applying a magnetic field, this amount increased between 1.1 and 1.7. Therefore, use of hybrid magnetic nanofluids could be beneficial for enhancement of heat transfer coefficient under a magnetic field. A more dramatic improvement in thermal efficiency is expected as a result of decreasing the surface temperature of test section after applying magnetic field.

Table 5 is summarized some recent work on the entropy generation of nanofluid flow.

4. Conclusions

This research has investigated a synthesize method for a hybrid mixture of rGO-Fe3O4 nanofluid with tannic acid as the reductant and stabilizer. The magnetic nanofluid was highly stable and a sedimentation of 5.2% after 60 days was observed. The thermal conductivity was enhanced up to 11% and this hybrid magnetic nanofluid exhibited Newtonian behavior. Effects of external permanent magnetic field on rGO-Fe3O4 hybrid nanofluids on the laminar forced convective heat transfer in a long heated circular tube have been experimentally studied. The local convective heat transfer coefficient increased up to 4% but it was not exceeding the thermal conductivity enhancements. The thermal efficiency index was only 1 in the absence of the magnetic field and it was increased between 1.1 and 1.7 after applying a magnetic field. Additionally, the effects of the fluid velocities, arrangement and number of magnets on total entropy generation rates have been extensively investigated and the total entropy generation rate was reduced up to 41% compared to distilled water. The rGO-Fe3O4 hybrid nanofluids can enhance the heat transfer under the influence of magnetic fields that depends on the three reasonable factors, including increases of thermal conductivity, viscosity increment and thermal boundary layer disturbance. Therefore, using rGO-Fe3O4 nanofluid in a thermal management system can enhance its thermal performance and provide an opportunity for reducing the size of heat exchangers.

Acknowledgements

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