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Functionalization and exfoliation of graphite into mono layer graphene for improved heat dissipation

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ABSTRACT

A novel synthesis procedure is presented for preparing single layer graphene (SGr) with high specific surface area (SSA). The SGr has been introduced as a promising additive for preparing super conductive nanofluids for high performance heat transfer applications. Owing to high SSA and unique degree of functionalization with hydrophilic groups, the SGr demonstrates an excellent colloidal stability and thermo-physical properties as an additive in aqueous media. Using UV-vis spectrometer, a highest dispersion stability of 0.866-relative concentration was reached after 30 days from preparation. Water-based SGr nanofluids illustrated fairly Newtonian behavior. Nanofluid containing SGr sheets with SSA of 761 m²/g and weight concentration of 0.01% showed the largest increase in thermal conductivity, i.e., from 0.748 to 0.795 W/m K as the temperature increased from 20 to 50 °C. The average heat transfer coefficient ratio ($(h_{nf}/h_{bf})_{ave}$) showed a downward trend with increasing of Reynolds number, decreasing from 233.5% at *Re* of 2000 to only 83.8% at *Re* of 16,000 at the constant weight concentration of 0.01%. Finally, the water-based SGr nanofluids were shown to be dramatically more effective in the duct with a backward-facing step in terms of overall thermal performance e.g., performance index and pumping power, and rheological properties including effective viscosity in comparison to distilled water.

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1. Introduction

The separation of flow and its reattachment takes place in sudden expansion of different geometries or backward-facing step [1]. This type of separation and reattachment plays a key role in designing equipment for different industrial applications which require heating or cooling operations. The occurrence of a mixture of high and low energy flow in the reattachment region may directly influence the amount of heat transfer rate in different equipment e.g., combustion chambers, environmental control systems, cooling systems for electronic equipment, high performance heat exchangers, cooling passages in turbine blades etc. Interestingly, the separation appears in the upper sharp corner of the step, resulting a recirculating region behind the step [2]. Thus numerous efforts have been given on study of separation flow of which the major por-

tions are numerical investigation of the effects of different parameters on the heat transfer performance and the actual mechanism of flow separation and reattachment [2–6]. One of the effective factors for enhancing heat transfer rate in the separated regions can be the thermo-physical properties of the working fluids, in particular, the thermal conductivities of the working fluids. Nanofluids as a new version of highly-conductive fluid open a new gateway for enhancing performance of different heating and cooling systems [7]. To address this issue, numerous researchers have been employing different nanostructures such as metal and metal oxide nanoparticles to enhance the heat transfer performance of different thermal equipment [8].

As a pioneer in investigating the heat transfer rate of nanofluids in a backward-facing step, Abu-Nada [9] prepared different water-based nanofluids including CuO, Al₂O₃, Ag, Cu and TiO₂ to study convective heat transfer coefficient in the presence of different nanofluids with different thermal conductivities. He concluded that the thermal conductivity of the working fluid plays a significant role in increasing the Nusselt number inside the recirculation zone. Kherbeet et al. [10] had investigated experimentally the effects of

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laminar water-based SiO₂ nanofluid flow over the backward-facing step on the heat transfer characteristics. The experimental results revealed that the water-based SiO₂ nanofluid at the concentration of 0.01 had the highest Nusselt number in comparison to that at the lower concentration. Mohammed et al. [11,12] investigated the effects of different nanofluids on the mixed convective heat transfer coefficient in the vertical and horizontal backward-facing steps. They observed that the nanofluids with secondary recirculation regions have shown lower Nusselt number. They also concluded that the diamond-based water nanofluids have shown significantly higher Nusselt number than that of Di-water. In another similar work, Kherbeet et al. [13] studied the heat transfer rate of Al₂O₃-, CuO-, SiO₂- and ZnO-based water nanofluids in the laminar regime over a microscale backward facing step. They observed no recirculation region behind the step for all the four nanofluids at various concentrations. They have also concluded that the SiO₂-based water nanofluid has shown the best performance among the prepared nanofluids.

With reference to the previous and the recent published information [14], most of studies on the backward facing step were performed in the presence of water-based metal or metal oxide nanofluids. It can also be seen that the lack of experimental study in the transitional and turbulent flow regimes is the second drawback in this field of study. Also, a majority of the mentioned works were numerical and there was no even numerical study on nanofluids loaded with carbon nanostructures. In other geometries, on the other hand, lots of researches were performed to enhance heat transfer rate using nanofluids loaded with different carbon based-nanostructures such as single-walled carbon nanotubes [15,16], multi-walled carbon nanotubes [17,18], graphene [19,20] and nitrogen doped graphene [21]. The unique properties of the few layer graphene such as outstanding thermal conductivity and superior specific surface area (SSA) have attracted the attention of a large number of scientists in different fields [21–23]. In particular, huge SSA, and high thermal conductivity make mono layer graphene an appropriate candidate for heat transfer applications. To this end, introducing a quick and easy method for mass-production of mono layer graphene functionalized with hydrophilic group is critical for its large-scale applications. Covalent-functionalization with appropriate degree of functionalization can provide stable suspension of the mono layer graphene.

To achieve all the goals mentioned above, *in situ* liquid phase exfoliation and functionalization of graphene [24,25] opened a new gateway for mass-production of mono-layer graphene. To increase the efficiency of exfoliation, the chemical functionalization of graphite with different functional groups, such as 4-bromophenyl, provides a new approach for improving its solubility in polar, aprotic, organic solvents, allowing the exfoliation of bulk graphite with fewer problems [26]. For example, Sun et al. [27] concluded that graphite can be changed to graphene by covalent functionalization of the graphite and the synthesis of a stable suspension in the presence of DMF without any added surfactant or stabilizer. Also, they achieved both exfoliation and functionalization with a fast procedure and obtained suitable edge-functionalization and intact pristine graphene structure in the interior basal planes. Thus, finding a method for mass production of mono layer graphene functionalized with hydrophilic groups and preparing highly conductive nanofluids can be the effective novelties for increasing the heat transfer rate of backward-facing step.

To this end, three phases of study have been performed to study the heat transfer behavior of water-based mono layer graphene nanofluids as well as water over a backward facing step. First phase comprises of the introduction of an *in situ* exfoliation and functionalization of graphene for mass-production of single layer graphene (SGr) flakes with the large SSA. Note that the method of synthesizing SGr seems to be novel, simple, and cost-

effective and chemically functionalized SGr flakes have the capability of good stability in aqueous media. The SGr flakes with nanoscopic porous morphology are of high SSA based on BET analyses of nitrogen cryo-adsorption method. Secondly, water-based SGr nanofluids were prepared at different low weight concentrations, and subsequently thermo-physical properties and colloidal stability in the presence of non-covalent functional group were investigated. Lastly, heat transfers parameters such as Nusselt number (Nu) and the convective heat transfer coefficient (*h*) over a backward-facing step in both of the transitional and turbulent flow regimes were investigated for the prepared coolants and compared with water data as the basefluid. The results suggest that water-based SGr nanofluids can be considered as the highly-conductive nanofluids even at very low weight concentrations.

2. Material and methods

2.1. Exfoliation and functionalization

In order to synthesize SGr, typically, the pristine graphite (10 mg) and 185.4 mg of AlCl₃ as a Lewis acid were ball-milled for 1 h by a planetary ball mill (PM 100) and subsequently the gray mixture poured into a Teflon reaction vessel with the volume of 250 ml. Then, 200 ml of tetrahydrofurfuryl polyethylene glycol (PEG) were gradually added during sonication for 30 min at room temperature to obtain a homogeneous suspension under nitrogen atmosphere. Also, 0.5 mL of concentrated hydrochloric acid (HCl) were added dropwise to the mixture over the sonication procedure. In the next step, the Teflon reaction vessel was sealed and instantly transferred into an industrial microwave and irradiated at 150°C and an output power of 700 W for 20 min. To increase the degree of functionalization, the resulting sample had sonicated for 5 min and then 5 min placed under microwave irradiation. This procedure repeated 4 times continuously. An electrophilic addition reaction happened between the PEG groups and the bulk graphite, resulting in the covalent bond between the PEG groups and hydroxyl groups to the exposed edges and side of the graphite flakes, thereby producing functionalized, expanded graphite. Microwave irradiation was employed for decreasing the reaction time and increasing the degree of functionalization. Regarding the mechanism of reaction, with a Lewis acid (AlCl₃) as a catalyst and trace concentrated hydrochloric acid to protonate alcohols, electrophilic addition reactions were carried out between PEG and graphene through a microwave-induced method. A primary alcohol in the presence of Lewis acids and concentrated hydrochloric acid can be protonated and resulted in an electrophilic species (carbocation or protonated alcohol). This electrophile (in the case of poly ethylene glycol) is a stabilized cationic electrophilic reagent with significantly superior reactivity in the presence of microwave irradiation [28]. These types of carbocations were described by George Olah [29]. By protosolvation of a cationic electrophile, an active electrophilic reagent is suitable for electrophilic aromatic substitution, thus in the case of the graphene flakes, an electrophilic addition reaction was occurred. The formed cations in the first step of electrophilic addition reaction on flakes reacts with nucleophiles [29,30]. Also, the reaction intensifies under microwave irradiation. When carbon nanostructures are exposed to microwave irradiation, strong absorptions are obtained which produces intense heating. Although the utilization of microwaves for the activation of carbon nanostructures has not been fully explored, the strong absorptions can open the door to the similar electrophilic addition reactions [28,31]. In addition, the edges of the expanded graphite were more available for reaction with the functional groups than the basal plane surfaces, which were stacked with strong π - π interactions. The resulting black ink-like dispersion was left to sit 24 h to separate large unstable graphite aggregates. The functionalized

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