Fabrication and Stabilization of Oxidized Carbon Black Nanoparticle Dispersion in Aqueous Solution for Photothermal Conversion Enhancement

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ABSTRACT: This study aims to explore oxidized carbon black nanoparticles (OCB-NPs) capped with an inorganic surfactant dispersed in water, as a carbon black water-based nanofluid, on photothermal conversion enhancement. We used ultraviolet–visible (UV–vis) absorption spectroscopy and zeta potential analyzers to identify the optimal concentration of sodium hexametaphosphate (SHMP) as an inorganic surfactant for OCB-NPs in order to determine the maximum value of UV–vis light absorption and absolute zeta potential. Then, the concentrations of 0.025−0.1 wt % OCB water-based nanofluid with SHMP were formulated by an ultrasonic bath for the examination of rheological behavior, thermal conductivity, and heating rate. The results indicated that the heating rate improvement of the water-based nanofluid involving 0.1 wt % OCB-capped with SHMP after irradiation by UV–vis light with wavelengths ranging from 220 to 380 nm, which is included in the solar spectrum, and an intensity of 205 W/m² increased by approximately 66%, compared to the base fluid in the cyclic flow system. Furthermore, after a 1 month storage period, the dispersion stabilization of water-based nanofluid including 0.1 wt % OCB-capped with SHMP reached 98%, as estimated by the UV–vis spectrophotometer.

1. INTRODUCTION

Due to specific surface area effects of inorganic nanoparticles with high thermal conductivity, enhanced convective heat transfer of nanofluids can be realized for higher efficiency and lower operating costs in the heating or cooling process.1−3

Figure 1. XPS spectrum of OCB with SHMP.

Figure 2. FT-IR spectra of OCB (a) without and (b) with SHMP.
Carbon nanomaterials are of particular interest because they are black in color, which makes them ideal for solar absorption as a result of their extremely high thermal conductivity in nanofluids. Several carbon nanomaterials, including their crystalline and amorphous phases, have been developed for thermal nanofluids. Sani et al. studied the graphite/nanodiamond suspension in ethylene glycol (EG) for solar direct absorbance collector (SDAC) and solar vapor generation. Thermal conductivity, rheological behavior, and surface tension of EG-based nanofluids containing titanium nitride (TiN) with various sizes of particles were experimentally explored to fill reliable and universal theoretical relationships for thermophysical properties of nanofluids. Recently, Tam et al. conducted a survey of using carbon nanotube and graphene water-based nanofluids for direct thermal absorption. They summarized some significantly enhancement of these nanofluids for SDAC, which depends on the type of nanoparticle suspension and the base fluid, the particle volume fraction, the size and shape of the nanoparticles, and the temperature.

In order to raise economic benefit and reduce environmental pollution, the research and development of carbon black nanoparticles dispersed in aqueous solution as a water-based nanofluid are essential topic for photothermal conversion. The dispersion of carbon black nanoparticles in water presented difficulties, including wetting, dispersion, and stabilization, leading to serious disadvantages in the practical applications. As a result, how to overcome these difficulties are very important study issues.

Surfactants can improve the stability of inorganic nanoparticle dispersion in aqueous solution; however, some concerns, such as contamination, foaming, and loss of thermal properties, can reduce nanofluid performance. Surface functionalization of an organic surfactant-free approach can deliver long-term nanofluid stability without the problems normally associated with organic-based surfactants, whereas it limits the working temperature of nanofluid as organic compounds can degrade at higher temperatures. As a result, mechanical/chemical reaction techniques were used to deposit hydrophilic functional groups onto carbon nanotubes to prevent aggregation in aqueous solution, and an organic surfactant-free technique was used to deposit hydroxyl groups onto double- and single-walled carbon nanotubes. Furthermore, in the case of carbon nanotube water-based photothermal fluids, the surface hydroxyl groups were found to disperse both double- and single-walled carbon nanotubes in water to promote nanofluid stability. Polar groups have been found to improve the dispersion properties of carbon nanotubes in water.

The stabilization of nanoparticle dispersion in aqueous solution can be quantitatively measured by the electrical potential between the dispersion medium and the stationary fluid layer attached to the particle. The electrical potential was defined as the zeta potential and indicated the degree of repulsion between charged particles dispersed in the fluid. A high zeta potential infers strong Coulomb repulsion forces between the dispersed particles and smaller attractive van der Waals forces. Nanofluids with a higher absolute value of zeta potential were considered to be a stable state, whereas nanofluids with a lower absolute value of zeta potential will undergo nanoparticle clustering and sedimentation. Generally, nanofluids with zeta potentials between 40 and 60 mV in absolute values were considered to have good stability, and those with zeta potentials greater than an absolute value of 60 mV were considered to have excellent stability.

For applications in solar energy, nanofluids offer several attractive and beneficial photothermal properties that can enhance the performance of direct-absorption solar thermal collectors. In particular, thermal conductivity enhancements of nanofluids have encouraged many researchers to evaluate their performance in direct absorption solar thermal collectors. However, investigators have also pointed out a number of factors that hinder the long-term stability and viability of nanofluids for practical use in solar thermal applications. Furthermore, the addition of organic surfactants and additives to alleviate nanoparticle clustering, agglomeration, and precipitation over time will alter the physical properties of the nanofluid. For example, increasing the concentration of surfactant will increase nanofluid viscosity and produce larger pressure drops throughout the collector system.
inducing larger demands for pumping power to circulate the working fluid.

As reviewed from the literature above, the addition of organic surfactants can prevent clustering and precipitation, but their use might have a detrimental impact on nanofluid properties involving viscosity and thermal conductivity. Therefore, our study intends to select the inorganic surfactant that can work in high-temperature situations for stabilizing the dispersion of OCB-NPs in water. We begin by using UV−vis absorption spectroscopy and zeta potential analyzers to identify the optimal amount of sodium hexametaphosphate (SHMP) as an inorganic surfactant for the dispersion of OCB-NPs in water to the maximum value of UV−vis light absorption and absolute zeta potential; then, the different concentrations of carbon black water-based nanofluids in the presence of SHMP are formulated by an ultrasonic bath. Finally, the resultant nanofluids will be characterized by UV−vis spectroscopy, natural sedimentation, rheology analysis, transmission electron microscopy, transient hot wire method, and thermal energy conversion to determine the optimal ratio of inorganic surfactant to CB-NPs, stability, and rheology behavior, as well as particle size distribution and photothermal conversion enhancement.

2. RESULTS AND DISCUSSION

2.1. Modification of Oxide Carbon Black. Choi et al. indicated that the main forms of SHMP were linear condensed phosphates (Na$_{n}$P$_{3}$O$_{10+n}$) that would degrade into lower phosphates in aqueous solutions. XPS measurement provided information about the presence of chemical elements and bonds for understanding the interfacial reaction between the surface of oxidized carbon black (OCB) and sodium hexametaphosphate (SHMP) O surfactant. As seen from the XPS spectra in Figure 1, typical XPS O 1s (285 eV) and C 1s (531 eV) spectra were obtained for OCB with SHMP, and it also showed O KLL (977 eV) spectra, which can be attributed to the surface oxidation of carbon black and the oxygen element provided by the phosphate ion. Furthermore, the P 2p (130 eV) and P 2s (187 eV) spectra were attributed to phosphorus, which means that the phosphate ion affected the carbon black and formed chemical bonds on the carbon black surface.

For further validation, FT-IR analysis was carried out for the surface compositions of samples. As shown in Figure 2, the peak at approximately 1200−1300 cm$^{-1}$ was C−O, and the peak at approximately 1720−1800 cm$^{-1}$ was C=O, indicating that the surface of the OCB has oxygen-bonded groups. In addition, the infrared spectrum of phosphate shows a very
strong and broad band on carbon black, appearing at 1120−940 and 550 cm−1, respectively. UV−vis analysis can also be used to show the effect of different SHMP concentrations on light absorption of carbon black nano-fluid. As displayed in Figure 3, with an increasing amount of SHMP, the light absorption of carbon nano-fluid increased until it reached a maximum value, responding to the optimal amount of 10 vol % SHMP (0.005 M) for 0.025 wt % CB-NP dispersion in water. This is because the excessive amount of SHMP brought out too much charge on the surface of the particles, causing the electric double layer to be compressed and the potential to decrease, inducing the van der Waals force between the particles to overcome the electrostatic repulsion, so that the particles would easily aggregate in the suspension, reducing light absorption of the species during UV−vis measurement.

In addition, according to Beer’s law,28

$$A = -\log T = \log \frac{I_0}{I} = aIc$$

(1)

where A indicates light absorbance, T expresses light transmittance, I0 and I are intensities of incident light and transmitting light, respectively, I represents the width of a sample cell (0.001 m), and C represents the concentration of OCB in base fluid (0.0025 g/dm³). At a wavelength of 400 nm, the extinction coefficient (α) of carbon black nano-fluid was significantly increased from 267.6 to 552.4 dm³/(m·g) as OCB was capped with SHMP. This suggests that the addition of an appropriate amount of the surfactant can effectively increase the light-absorbing ability of the carbon black nano-fluid due to well dispersed OCB-NPs in the base fluid.

2.2. Stabilization of Carbon Black Nano-fluid. The primary particle size of carbon black is approximately 20 nm; however, since the surface energy between the particles is large, it makes the particles tend to aggregate, forming secondary particles, called as the secondary particle size. The effect of ionic surfactant on the average secondary particle size of OCB was measured by TEM (Figure 4). As estimated from the figure, the average secondary particle size of OCB capped with SHMP was 25.67 nm, which was evidently smaller than 124.10 nm of OCB without modified with SHMP. In addition, we applied a zeta potential analyzer to measure the surface potential of OCB dispersion in water with different concentrations of SHMP. As shown in Figure 5a, the absolute zeta potential clearly increased with increased surfactant, reaching a maximum negative value of −67.55 mV with the addition of 10 vol % SHMP (0.005 M) in water containing 0.025 wt % OCB, which means that the absolute value of the interface potential is the highest, leading to excellent stability of CB-NP dispersion in base fluid with an optimal amount of surfactant. Additionally, Figure 5b shows that the zeta potential of 0.025 wt % OCB in water with 10 vol% SHMP (0.005 M) varied according to the pH value. The absolute zeta potential was found to increase substantially with the increasing pH value, reaching a maximum value of −78.53 mV at pH 8.45, which results from OH− ions in the alkaline liquid also adsorbing to the surface of OCB, thereby increasing the negative zeta potential.

Furthermore, we used the natural sedimentation method and UV−vis absorption spectroscopy to quantify the amount of OCB particles suspended in a liquid after standing for a period of time. As illustrated in Figure 6, after storage for 30 days, the normalized light absorbances of OCB dispersion in water with and without SHMP can be used to confirm that the OCB-NPs have 85.82 and 97.83%, respectively, in liquid compared to the original suspension solution. This suggests that the inorganic surfactant can significantly enhance the stabilization of oxidized carbon black nano-fluid. According to the optimal ratio of OCB to SHMP, we increased the
concentration of OCB from 0.025 to 0.1 wt % in water to confirm whether the higher concentration affects the stability of the dispersion. As shown in Figure 7, no difference in the zeta potential was found, indicating that the oxidized carbon black water-based nano fluid remained in a very stable state due to the effect of the inorganic surfactant.

2.3. Thermophysical Properties. 2.3.1. Viscosity. In DASC, nano fluid was circulated flowing inside a glass tube, where the nano fluid absorbs heat from irradiation, and then it conveyed the absorbed heat to the cold water through a heat exchanger. Therefore, the viscosity of the as-prepared nano fluid was measured to evaluate the effect the wall surface friction force of tube in this work. As shown in Figure 8a, as the concentration of carbon black was increased from 0.025 to 0.1 wt %, the viscosity of nano fluid was raised from 1.05 to 1.09 cp, indicating that the concentration of carbon black increased by four times, but the increase of viscosity was only 4.31%; and as-prepared carbon black water-based nano fluid belonged a shear thinning fluid, as exhibited in Figure 8b, meaning that the dispersed nanoparticles would not be aggregated during the flowing state, which in turn will facilitated for the practical application.

2.3.3. Heating Rate Improvement. For evaluation of photothermal conversion enhancement of oxidized carbon black water-based nano fluid, we applied the Newton’s law of cooling, Figure 10. Temperature difference between the initial state and during cyclic flow periods enlarging with the increasing flow rate of water-based nano fluid with amounts of OCB and SHMP at (a) 40, (b) 80, and (c) 160 mL/min, as well as irradiating time of UV–vis light with wavelengths ranging from 220 to 380 nm and an intensity of 205 W/m².

2.3.2. Thermal Conductivity. The thermal conductivity of a nano fluid is mainly influenced by the number, size, and material properties of solid nanoparticles and the carrier fluid. In this study, the thermal conductivity of water-based nano fluid with different concentrations of OCB capped with SHMP was measured by transient hot wire method at 298 K controlled by cyclic cooling water.

As analyzed from the results in Figure 9, the addition of OCB capped with ionic surfactant in the base fluid would indeed increase thermal conductivity. The thermal conductivity of as-prepared nano fluid would rise from 0.592 (W/m·K) to 0.606 (W/m·K) when the 0.1 wt % OCB is added into the water-based nano fluid in the presence of SHMP.

Table 1. Heating Rate Improvement of Carbon Black Water-Based Nanofluid Varying with Different Flow Rates and Compositions

| flow rate (mL/min) | sample composition         | heating rate improvement (%) |
|-------------------|----------------------------|-----------------------------|
| 40                | Di-water                   | 0                           |
|                   | Di-water/OCB-0.025 wt %    | 9.95                        |
|                   | Di-water/SHMP/OCB-0.025 wt %| 17.60                       |
|                   | Di-water/SHMP/OCB-0.050 wt %| 21.43                       |
|                   | Di-water/SHMP/OCB-0.1 wt % | 34.95                       |
| 80                | Di-water                   | 0                           |
|                   | Di-water/OCB-0.025 wt %    | 23.89                       |
|                   | Di-water/SHMP/OCB-0.025 wt %| 37.71                       |
|                   | Di-water/SHMP/OCB-0.050 wt %| 47.07                       |
|                   | Di-water/SHMP/OCB-0.1 wt % | 60.66                       |
| 160               | Di-water                   | 0                           |
|                   | Di-water/OCB-0.025 wt %    | 28.95                       |
|                   | Di-water/SHMP/OCB-0.025 wt %| 41.89                       |
|                   | Di-water/SHMP/OCB-0.050 wt %| 48.25                       |
|                   | Di-water/SHMP/OCB-0.1 wt % | 66.67                       |
where \( Q \) indicates the intensity of light radiation, \( A \) expresses the cross-sectional area, \( h \) is the heat transfer coefficient of forced convection, and \( \Delta T \) is the temperature difference of the nanofluid between the initial state and during cyclic flow periods. In this work, \( Q \) and \( A \) are specified so that the heat transfer coefficient of forced convection, which are functions of fluid properties and velocity, could be in terms of temperature difference between the initial state and during cyclic flow periods of oxidized carbon black water-based nanofluid.

In order to further understand photothermal conversion enhancement, we carried out the light absorption measurement of the as-fabricated nanofluid affected by different concentrations of the OCB and flow rate in a cyclic flow system with a setup equipped with an ultraviolet irradiation device. As demonstrated in Figure 10, the temperature difference between the initial state and during irradiation by UV–vis light with wavelengths ranging from 220 to 380 nm, which is involved in the solar spectrum, and an intensity of 205 W/m² was found to significantly enlarge with an increasing flow rate [(a) 40, (b) 80, and (c) 160 mL/min] of water-based nanofluid with amounts of OCB and SHMP in the cyclic flow system. This is because the uniform dispersion of OCB nanoparticles due to the capping agent in flowing base fluid can enhance the light absorption of nanofluids during irradiation by UV–vis light.

According to the experimental results above, at a steady state and the temperature difference from the initial state of water-based nanofluid being the datum, the heating rate improvement was defined as the temperature difference from the initial state of the OCB nanofluid subtracted by the datum and then divided by the datum. Table 1 lists a summary of heating rate improvement for water-based nanofluid with different amounts of OCB and SHMP under three flow rates of 40, 80, and 160 mL/min, respectively. As shown in the table, particularly, the heat rate improvement could reach 66.67% when the water-based fluid slightly added by 0.1 wt % OCB nanoparticles capped with SHMP was irradiated by UV–vis light for 25 min at a flow rate of 160 mL/min, which is induced by a specific area effect of carbon black nanoparticles and forced convection.

\[ Q/A = h\Delta T \]

3. CONCLUSIONS

In this work, the surface of CB-NPs was first modified through oxidation and then capped with inorganic surfactant, followed by dispersal in water-based fluid via an ultrasonic bath, thereby becoming an oxidized carbon black water-based nanofluid. The zeta potential of the aqueous solution with 0.025 wt % OCB nanoparticle and 10 vol % SHMP (0.005 M) reached a maximum negative value of −78.53 mV at a pH of 8.45, exhibiting excellent stabilization of suspension. In addition, in the photothermal conversion of the cyclic flow system, the heating rate improvement of as-fabricated nanofluid (0.1 wt % OCB) increased by approximately 67%, but its viscosity increased only slightly (4%) relative to the base fluid.

4. EXPERIMENTAL SECTION

4.1. Materials. Carbon black nanopowder with a sphere-like particle and a mean primary particle size of 14 nm, trademarked by Monarch 1100, was purchased from Cabot Co. and oxidized into oxidized carbon black (OCB) in order to be compatible with water.\(^{31}\) Deionized water (18.1Ω) was used as a base fluid. Sodium hexametaphosphate (SHMP) manufactured by Sigma-Aldrich was used as an inorganic surfactant.

4.2. Sample Preparation. To determine the optimal amount of inorganic surfactant for dispersion stabilization of the water-based nanofluid, 5 mg of OCB was dispersed in 25 mL deionized water with different amounts of 0.005 M SHMP (0, 5, 10, and 15 vol %) by an ultrasonic bath (Branson 3510, 40 kHz, 143 W) for 15 min under ambient temperature controlled by recycle cooling water. To investigate the rheological behavior, thermal conductivity, and heating rate improvement of the water-based nanofluid, at room temperature, the nanofluids with different concentrations (0.025, 0.05, and 0.1 wt %) of OCB in the presence of SHMP were created by ultrasonic bath dispersion for 60 min.
is included in the solar spectrum, and an intensity of 205 W/A4000) with wavelengths ranging from 220 to 380 nm, which respectively, in which the equipment are sketched in Figures procedures from the manuals of the DV-II+Pro and KD2 Pro, ductivity both are repeated three times based on operating device. The measurements of viscosity and thermal conductivity was recorded on a Decagon KD2 Pro with a Nano Brook Zeta PALS. The viscosity was recorded by analysis was performed with a JEOL JSM-7800F scanning spectroscopy data were collected with a Shimadzu UV-1800 spectroscopy, and samples were contained in 1 mL spectroscopy (FTIR; FT-720) spectroscopy was performed form infrared (FTIR) and ethylene glycol-based nanofluids. Rashidi, A. M. Sunlight absorbing potential of carbon nanoball water incorporation in direct solar thermal collectors: a review. Poinern, G. Nanofluid types, the ir synthesis, properties and behavior of carbon nanomaterials when interfacing neuronal cells: How far have we come? Carbon 2019, 143, 430–446. (7) Karami, M.; Raisee, M.; Deliani, S.; Akhavan Bahabadi, M. A.; Rashidi, A. M. Sunlight absorbing potential of carbon nanoball water and ethylene glycol-based nanofluids. Opt. Spectrosc. 2013, 115, 400–405. (8) Hordy, N.; Rabilloud, D.; Meunier, J.-L.; Coulombre, S. High temperature and long-term stability of carbon nanotube nanofluids for direct absorption solar thermal collectors. Sol. Energy 2014, 105, 82–90. (9) Das, S.; Giri, A.; Samanta, S.; Kanagaraj, S. Role of graphene nanofluids on heat transfer enhancement in thermosyphon. J. Sci.: Adv. Mater. Devices 2019, 4, 163–169. (10) Hazra, S. K.; Ghosh, S.; Nandi, T. K. Photo-thermal conversion characteristics of carbon black-ethylene glycol nanofluids for applications in direct absorption solar collectors. Appl. Therm. Eng. 2019, 163, 114402. (11) Chamsa-ard, W.; Brundavanam, S.; Fung, C. C.; Fawcett, D.; Poinern, G. Nanofluid types, their synthesis, properties and incorporation in direct solar thermal collectors: a review. Nanomaterials 2017, 7, 131. (12) Zyla, G.; Fal, J.; Estellé, P. Thermophysical and dielectric profiles of ethylene glycol based titanium nitride (TiN–EG) nanofluids with various size of particles. Int. J. Heat Mass Transfer 2017, 113, 1189–1199. (13) Tam, N. T.; Phuong, N. V.; Khoi, P. H.; Minh, P. N.; Afrand, M.; van Trinh, P.; Thang, B. H.; Zyla, G.; Estellé, P. Carbon nanomaterial-based nanofluids for direct thermal solar absorption. Nanomaterials 2020, 10, 1199. (14) Han, D.; Meng, Z.; Wu, D.; Zhang, C.; Zhu, H. Thermal properties of carbon black aqueous nanofluids for solar absorption. Nanoscale Res. Lett. 2011, 6, 457. (15) Ghadimi, A.; Metselaar, I. H. The influence of surfactant and ultrasonic processing on improvement of stability, thermal conductivity and viscosity of titania nanofluid. Exp. Therm. Fluid Sci. 2013, 51, 1–9. (16) Al-Waeli, A. H. A.; Chaichan, M. T.; Kazem, H. A.; Sopian, K. Evaluation and analysis of nanofluid and surfactant impact on photovoltaic-thermal systems. Case Stud. Therm. Eng. 2019, 13, 100392. (17) Halaful, S.; Maré, T.; Estellé, P. Efficiency of carbon nanotubes water based nanofluids as coolants. Exp. Therm. Fluid Sci. 2014, 33, 104–110.

4.3. Characterization. To analyze the optimal conditions of the addition of SHMP, X-ray photoelectron spectroscopy (XPS) was carried out on a PHI 5000 Versa Probe X-ray photoelectron spectroscopy (ULVAC-PHI) and Fourier transform infrared (FTIR; FT-720) spectroscopy was performed and scanned over the range of 400–4000 cm⁻¹. UV–vis spectroscopy data were collected with a Shimadzu UV-1800 spectrophotometer, and samples were contained in 1 mL quartz cuvettes. Transmission electron microscopy (TEM) analysis was performed with a JEOL JSM-7800F scanning electron microscope. Zeta potential analysis was performed with a Nano Brook Zeta PALS. The viscosity was recorded by a rheometer on a programmable DV-II+Pro viscometer, and thermal conductivity was recorded on a Decagon KD2 Pro device. The measurements of viscosity and thermal conductivity both are repeated three times based on operating procedures from the manuals of the DV-II+Pro and KD2 Pro, respectively, in which the equipment are sketched in Figures 11 and 12. Ultraviolet visible light source (EXFO Acticure A4000) with wavelengths ranging from 220 to 380 nm, which is included in the solar spectrum, and an intensity of 205 W/m² was applied to radiate the as-prepared nanofluid in the cyclic flow system under ambient temperature in a closed cabinet.

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Notes
The authors declare no competing financial interest.

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