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The effects of ultrasonication power and time on the dispersion stability of few-layer graphene nanofluids under the constant ultrasonic energy consumption condition

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ABSTRACT

Few-layer graphene (FLG) nanofluids have received widespread interest in recent years due to their excellent thermal and optical properties. However, the low dispersion stability is one of the main bottlenecks for their commercialization. Ultrasonication is an effective method and almost an essential step to improve the stability of nanofluids. This work aimed to determine the optimal ultrasonication process for preparing stable FLG nanofluids, particularly under the constant ultrasonic energy consumption condition. For this purpose, FLG nanofluids were prepared under various amplitudes (20%–80%) and times (33.75–135 min) and evaluated by both sedimentation and optical spectrum analysis techniques. It was found that ultrasonication treatment at 30% amplitude for 90 min was sufficient for proper dispersion of FLG, and a further increase in the ultrasonication power would not benefit the stability enhancement much. However, for FLG nanofluids prepared at amplitudes higher than 30% under the constant ultrasonic energy consumption condition, their stability deteriorated seriously due to the reduced ultrasonication time, while for FLG nanofluids prepared at 20% amplitude for 135 min, they showed the higher stability, which indicates that the stability of FLG nanofluids is more sensitive to ultrasonication time than power. Therefore, a relatively longer ultrasonication time rather than a higher amplitude is recommended to prepare stable FLG nanofluids for practical applications at given ultrasonic energy consumption.

1. Introduction

Nanofluids have shown prospects in widespread applications such as heat transfer [1,2], solar energy harvesting [3–5], and lubrication [6,7] since the concept was firstly proposed in 1995 [8]. By far, nanofluids have not been commercialized yet. One of the main bottlenecks is their poor stability, especially the long-term dispersion stability [9].

One of the effective methods to improve the dispersion stability of nanofluids is ultrasonication treatment, which breaks the nanoparticle clusters and lowers their size for proper dispersion by the cavitation process [10–13]. Ultrasonication time and power are two crucial factors affecting the performance of ultrasonication treatment [14], and various investigations have been conducted to find the optimal ultrasonication process for stable nanofluid preparation [15–178]. For example, Mahbubul et al. [18] tested the stability of TiO_2 nanofluids for different ultrasonication times by zeta potential analysis and electron microscopies. The results indicated that the dispersion of TiO_2 nanoparticles improved with the ultrasonication time up to 150 min, and the longer time would lead to re-agglomeration of nanoparticles. Asadi et al. [19] investigated the stability of water-based MWCNT nanofluids via zeta potential measurement and sedimentation technique. They reported that MWCNT nanofluids presented the highest stability and thermal conductivity by 60 min ultrasonication, and the stability would be deteriorated by further prolonging the ultrasonication time. It can be inferred that there existed an optimal ultrasonication time for some nanoparticles, and further prolonging the ultrasonication time would deteriorate the stability of nanofluids. For some other nanoparticles, the longer ultrasonication time would result in higher stability [20,21]. Compared to ultrasonication time, the effects of ultrasonication power are less investigated. Graves et al. [22] prepared the methanol-based capped copper nanofluids through ultrasonication. They found that the z-average size decreased from 330 nm to 190 nm as the ultrasonication amplitude increased from 20% to 80%. There is also some research regarding the combined effects of ultrasonication time and power [23–25].

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Nomenclature

CTAB	cetyltrimethylammonium bromide
FLG	few-layer graphene
RC	relative concentration
RSM	response surface method
TEM	transmission electron microscopy

Based on the literature review, it can be concluded that both ultrasonication time and power affect the dispersion stability of nanofluids significantly, and the optimal ultrasonication process for different nanofluids is material-dependent. In recent years, investigations on graphene family nanofluids have presented a rapidly increasing trend. However, water-based graphene nanofluids suffer from relatively poor stability due to the hydrophobic nature of graphene [26]. Although various investigations have been conducted to improve the dispersion stability of graphene nanofluids through ultrasonication, most of these studies focus on the impacts of ultrasonication time [12]. Moreover, the ultrasonication power and time effects are commonly studied under the varying energy consumption condition; in other words, the ultrasonic energy consumption differs from case to case. For fair comparisons of the performance of ultrasonication treatment, the total ultrasonic energy consumed should be fixed, which is beneficial to find the most effective ultrasonication process at given ultrasonic energy consumption. In this work, water-based flew-layer graphene (FLG) nanofluids were prepared by a two-step method through ultrasonication. Both sedimentation and optical spectrum analysis methods were applied to systematically examine the effects of ultrasonication power and time on the stability of graphene nanofluids. The mono effect of ultrasonication power was first examined to determine the referential ultrasonic energy consumption. Then the synergetic effects of ultrasonication power and time under the constant ultrasonic energy consumption condition were evaluated. The finding of this work would reference the preparation of stable FLG nanofluids through ultrasonication treatment.

2. Experiments

2.1. Materials and preparation of nanofluids

Few-layer graphene (FLG) in powder form was provided by Suzhou Tanfeng Graphene Technology Co., Ltd. According to the supplier, FLG was fabricated by the physical method, and it had a purity of 98 % and 3–5 layers. Detailed properties of FLG provided by the suppliers are shown in Table 1. Deionized water was chosen as the base fluid. The cationic surfactant, cetyltrimethylammonium bromide (CTAB), was selected to stabilize FLG within the base fluid.

A two-step method was used to prepare FLG nanofluids [27]. As shown in Fig. 1, firstly, 100 mL deionized water was weighted. Then CTAB and FLG with a ratio of 1: 1 was added into the deionized water, followed by magnetic stirring for 30 min. Lastly, the suspension was dispersed by a probe-type sonicator (Scientz-1500F, 18 mm tip) at 20 kHz for various ultrasonication amplitudes and times in the pulse mode (2 s OFF and 2 s ON). The maximum output power of the sonicator is 1500 W, and the output power can be adjusted by varying the amplitude. The pulse mode was selected because ultrasonication in such a mode helps to retard the temperature rise rate [12]. In addition, the samples were placed in a flat-bottom jacket beaker, and their temperature during ultrasonication was maintained below 25 ℃ via cooling water to prevent the degradation of CTAB and minimize vaporization [28]. Two concentrations (0.01 wt% and 0.1 wt%) of FLG nanofluids were prepared. In the pre-experiment, FLG nanofluids subjected to only ultrasonication treatment would completely sediment in a short time due to the hydrophobic nature of FLG. Therefore, both physical and chemical techniques were employed to enhance the stability of FLG nanofluids. The ratio between FLG and CTAB was fixed for all nanofluids, and the primary goal of this work was to examine the effects of ultrasonication treatment.

Table 1
The properties of FLG according to the supplier.

Property	Value
thermal conductivity	5000 W/(m·K)
density	2200 kg/m^3
specific heat capacity	790 J/(kg·K)
layer	3–5
purity	98 %
thickness	1–3 nm
lateral size	10–20 μm
appearance	black powder

2.2. Characterizations

The micro morphologies of FLG nanoparticles were examined by a TEM (Tecnai G2 F20, FEI). The absorption spectra of FLG nanofluids were determined by a UV–Vis spectrophotometer (UV-2600, Shimadzu).

2.3. Evaluation of the stability of FLG nanofluids

The sedimentation method was first used to determine the stability of FLG nanofluids. Typically, the pictures of FLG nanofluids were recorded and compared over time to examine the variations of sediment in nanofluids. The optical spectrum analysis method was also applied to evaluate the stability of nanofluids. This method works based on the Beer-Lambert law that the absorbance is linearly proportional to the concentration of nanofluids [29,30]. The stability of FLG nanofluids is determined by measuring the absorbance and its variations with time. The degrading of absorbance value reflects a decrease in the concentration of nanofluids because of sedimentation. Note that this method is not applicable to directly determine the stability of dark-colored nanofluids with high concentrations due to the considerable noises in the spectrum. Therefore, in this work, the prepared nanofluids were firstly diluted in a ratio of 1:50 before measurement.

3. Results and discussion

3.1. Morphology

As shown in Fig. 2(a), FLG presents flake-like structures. From Fig. 2 (b), it is seen that FLG has about four graphene layers, which is following that provided by the supplier.

3.2. The effects of ultrasonication power

In this section, the mono effect of ultrasonication power on the stability of FLG nanofluids was examined under the constant ultrasonication time condition. The ultrasonication time of 90 min was selected according to previous work [17] and our pre-experiments. Seven amplitudes (20%, 30%, 40%, 50%, 60%, 70%, and 80%) were considered, and the corresponding ultrasonication power varies in the range of 300–1200 W. Note that the volume of FLG nanofluids is 100 mL during preparation; thus the corresponding ultrasonication power density falls in the range of 3000–12000 W/L.

Fig. 3 depicts the visual inspection of FLG nanofluids just as prepared and after a month from preparation. The FLG nanofluids present a dark appearance due to the black nature of FLG. After static sedimentation for a month, there is almost no change in appearance. Also, no apparent separation between FLG and base fluid is found in the supernatants. However, a small amount of sediment is seen at the bottom, which may result from the aggregation of large size FLG due to the gravitational pull

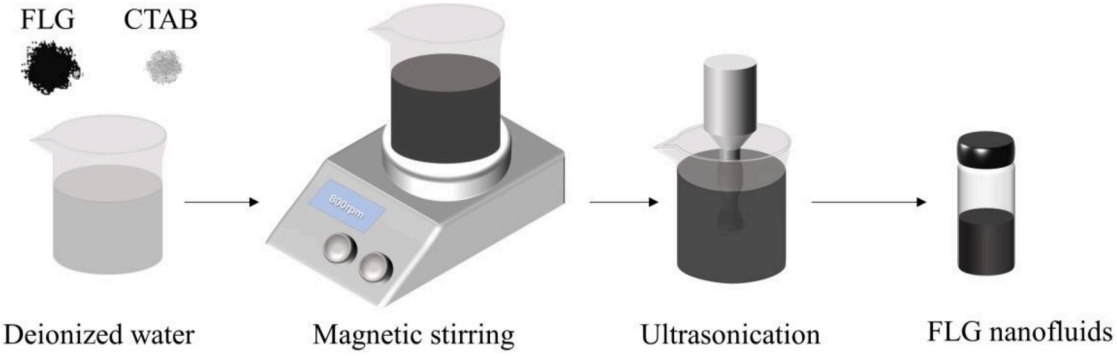

Fig. 1. Preparation of FLG nanofluids.

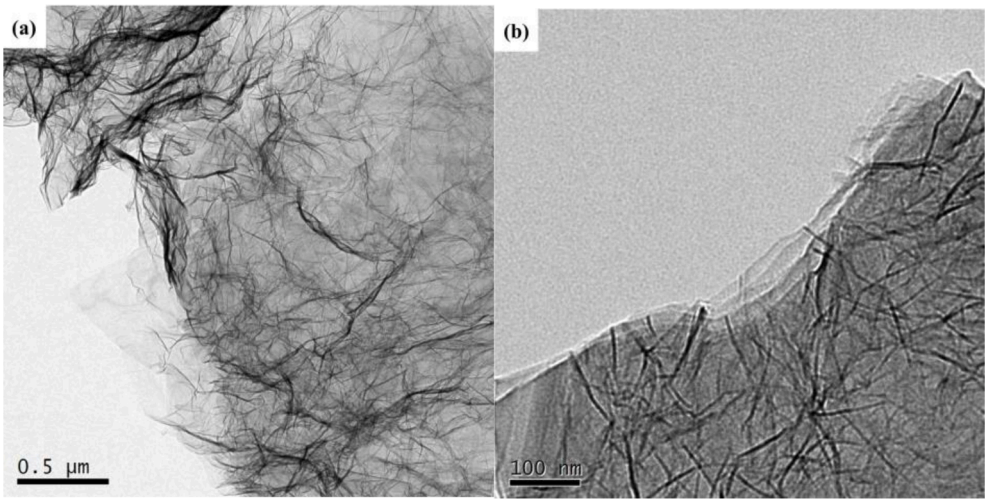

Fig. 2. TEM images of FLG.

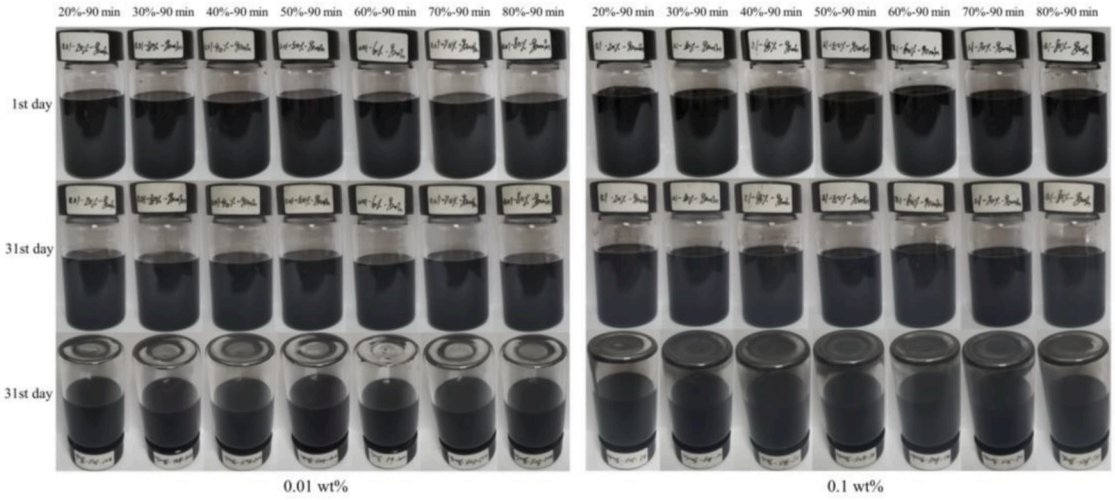

Fig. 3. Photographs of FLG nanofluids prepared at different ultrasonication powers as prepared and after a month from preparation.

under the static condition. In addition, more sediment is seen at 0.1 wt% compared to 0.01 wt%, implying that FLG nanofluids are more stable at low concentrations. At a given concentration, the amount of sediment of FLG nanofluids prepared at different ultrasonication powers is close to each other. Hence, it is difficult to distinguish the degree of stability of FLG nanofluids prepared at different ultrasonication powers via the sedimentation method.

The optical absorbance spectrum analysis method was further applied to examine the long-term dispersion stability of FLG nanofluids. Fig. 4 presents the absorption spectra of FLG nanofluids as prepared. The absorbance peak values of FLG nanofluids are located around the wavelength of 270 nm, which agrees with previous work [31], confirming the successful preparation of graphene nanofluids. Scrutinizing the figure reveals that the absorption spectra of fresh nanofluids are

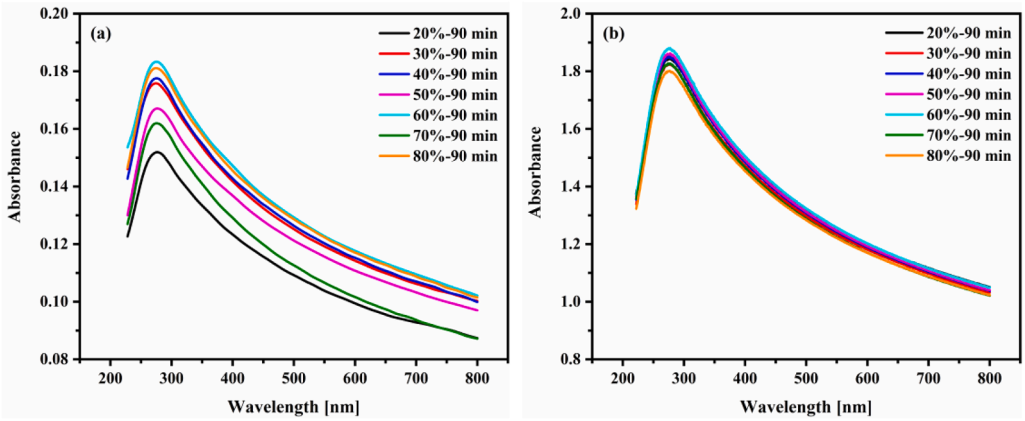

Fig. 4. Absorption spectra of fresh FLG nanofluids prepared at different ultrasonication powers for 90 min: (a) 0.01 wt% and (b) 0.1 wt%.

pretty close at 0.1 wt%, implying similar stability. The differences in the absorption spectra are relatively more considerable at 0.01 wt%, indicating that the effect of ultrasonication power on the initial stability of FLG nanofluids is more significant at low concentrations.

Relative concentration (*RC*), defined as the ratio of the absorbance of nanofluids after static sedimentation for a specific time to that of the fresh one, was used to indicate the sedimentation process quantitatively. As presented in Fig. 5, the *RC*s of FLG nanofluids subjected to various ultrasonication powers decrease with time, implying agglomeration of FLG nanoparticles, followed by sedimentation. In particular, a sharp decrease in the *RC*s occurs in the first two days after preparation, meaning the rapid sedimentation of FLG. The possible reason is that FLG nanoparticles are uniformly dispersed within the base fluid after preparation due to the influence of ultrasonic cavitation, and the initial concentration is also high so that collision of FLG nanoparticles is more likely to happen within the nanofluids due to the Brownian motion. According to the DLVO theory [32], the attractive force between FLG nanoparticles will dominate the repulsive force after the collision, which pulls FLG nanoparticles together, leading to their rapid agglomeration and subsequent sedimentation because of the increased weight. After the rapid sedimentation stage, the reduction rates of *RC*s tend to be gentle, indicating increased stability. After a month's static sedimentation, the *RC* of FLG nanofluids drops by approximately 20% and 30% for 0.01 wt % and 0.1 wt% concentration, respectively. The results confirm that the stability of FLG nanofluids reduces with the increasing concentration.

Moreover, the *RC* of FLG nanofluids prepared at 20% amplitude is lower than those of nanofluids prepared at higher amplitudes, especially at 0.1 wt% concentration, implying that ultrasonication at 20% amplitude for 90 min is not enough for breaking down the FLG clusters, and using the relatively higher amplitudes benefits the dispersion of FLG nanoparticles. For FLG nanofluids prepared at 30%–80% amplitudes, variations in the *RC* are found pretty limited, indicating that a further increase in the ultrasonication power will not significantly improve the stability. Therefore, ultrasonication at 30% amplitude for 90 min is believed sufficient for dispersing FLG nanoparticles, and there is no need to use excessively high ultrasonication power, which will not enhance the stability but increase energy consumption.

3.3. The synergetic effects of ultrasonication power and time

In the section above, ultrasonication power and total ultrasonic energy consumption in preparing FLG nanofluids are different. It is unknown whether the ultrasonication power or ultrasonic energy determines the stability of FLG nanofluids. As an improvement, the ultrasonic energy consumption for preparing nanofluids was fixed for more fair comparisons in this section. As previously discussed, ultrasonication treatment at 30% amplitude of sonicator power (450 W) for 90 min was sufficient for preparing stable FLG nanofluids. Therefore, the total ultrasonic energy consumption at such treatment was selected as the baseline. For the newly prepared nanofluids, the ultrasonication time was accordingly extended for the cases at amplitudes lower than 30%, while for cases over 30%, the ultrasonication time was correspondingly reduced to maintain the constant ultrasonic energy consumption. The modified ultrasonication time and power for preparing FLG nanofluids under the constant ultrasonic energy consumption condition are presented in Table 2.

As illustrated in Fig. 6, FLG nanofluids prepared under the constant ultrasonic energy consumption condition present a black appearance,

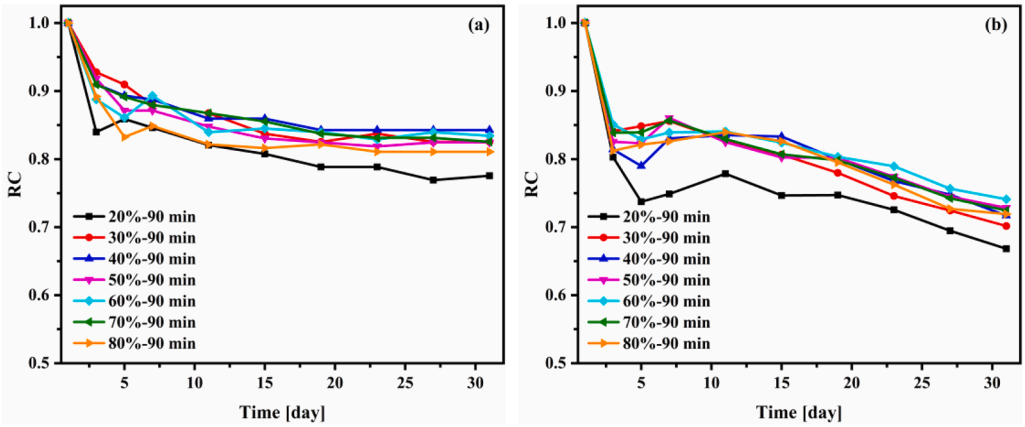

Fig. 5. Variations of the *RC*s of FLG nanofluids prepared at different ultrasonication powers with time: (a) 0.01 wt% and (b) 0.1 wt%.

Table 2
Ultrasonication power and time under the constant ultrasonic energy consumption condition.

Amplitude	20%	30%	40%	50%	60%	70%	80%
Power	300 W	450 W	600 W	750 W	900 W	1050 W	1200 W
Time	135 min	90 min	67.5 min	54 min	45 min	38.5 min	33.75 min

and no visual variations can be found after sedimentation for a month. By reversing the bottles, some sediment can be seen at the bottom, indicating the variation in the stability of FLG nanofluids. Also, it is not difficult to distinguish the amount of sediment for different concentrations. However, it is not easy to distinguish the amount of sediment for nanofluids prepared under various combinations of ultrasonication power and time only via visual inspection at a given concentration.

As shown in Fig. 7, the absorbance peak values of FLG nanofluids prepared under the constant ultrasonic energy consumption condition are also located around the wavelength of 270 nm. The absorbance of FLG nanofluids almost increases with the increasing ultrasonication time or decreasing ultrasonication power. The results indicate that both ultrasonication time and power significantly affect the initial stability of FLG nanofluids, and FLG nanofluids prepared at lower ultrasonication power with longer time present relatively higher initial stability.

Fig. 8 shows that the *RC*s of FLG nanofluids drop with time for all the combinations of ultrasonication power and time, indicating the continuous sedimentation process of FLG nanoparticles. Besides, the sedimentation rate is relatively high in the first few days and then drops gradually with time, meaning that the stability of FLG nanofluids improves with the sedimentation time. Another feature that can be seen is that variations in the *RC* of FLG nanofluids are easier to distinguish, and the *RC* of FLG nanofluids increases with ultrasonication time at a given time. After a month's sedimentation, the *RC*s of FLG nanofluids prepared at 20%-135 min, 30%-90 min, 40%-67.5 min, 50%-54 min, 60%-45 min, 70%-38.5 min, and 80%–33.75 min at 0.1 wt% concentration are 0.77, 0.70, 0.66, 0.58, 0.52, 0.45, and 0.44, respectively. In particular, the *RC* of FLG nanofluids prepared at 40%-67.5 min is 5.7 % lower, while the value of nanofluids prepared at 20%-135 min is 10 % higher than that of nanofluids prepared at 30%-90 min. The results indicate that the loss in the stability of FLG nanofluids caused by the reduction in ultrasonication time cannot be compensated by the corresponding increase in ultrasonication power to keep the constant ultrasonic energy consumption. In other words, the positive influence of prolonging ultrasonication time is superior to that of increasing ultrasonication power on the stability of FLG nanofluids. A similar variation trend can also be seen at the 0.01 wt % concentration.

Moreover, variations of the *RC*s of FLG nanofluids with ultrasonication power and time at different days are presented in Fig. 9. It is observed that FLG nanofluids prepared at lower amplitude with correspondingly longer time present the higher *RC*. Besides, the reduction rate of *RC*s for FLG nanofluids prepared at lower amplitude with longer time is much lower than that of other nanofluids, indicating that

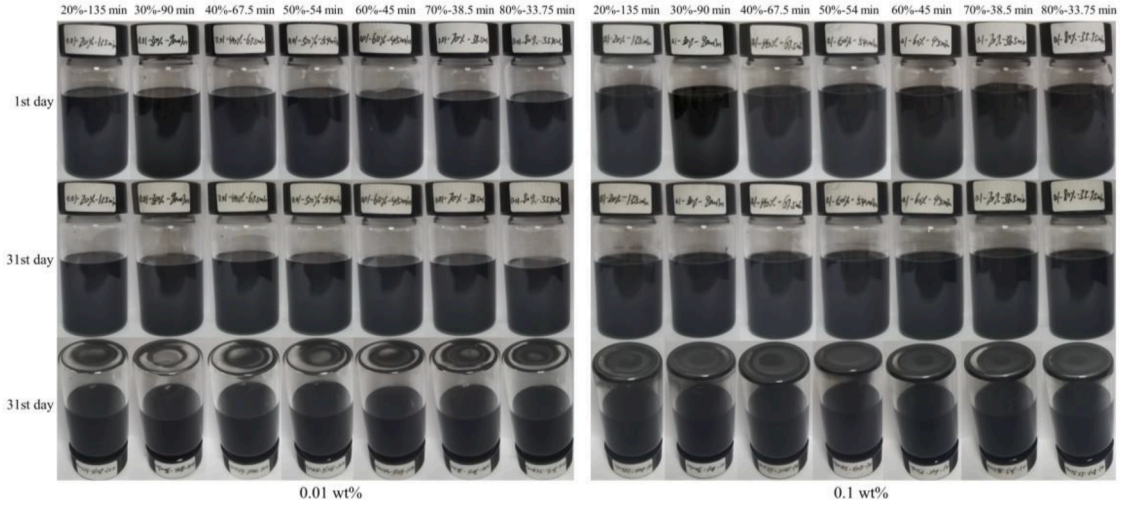

Fig. 6. Photographs of FLG nanofluids as prepared and after a month from preparation under the constant ultrasonic energy consumption condition.

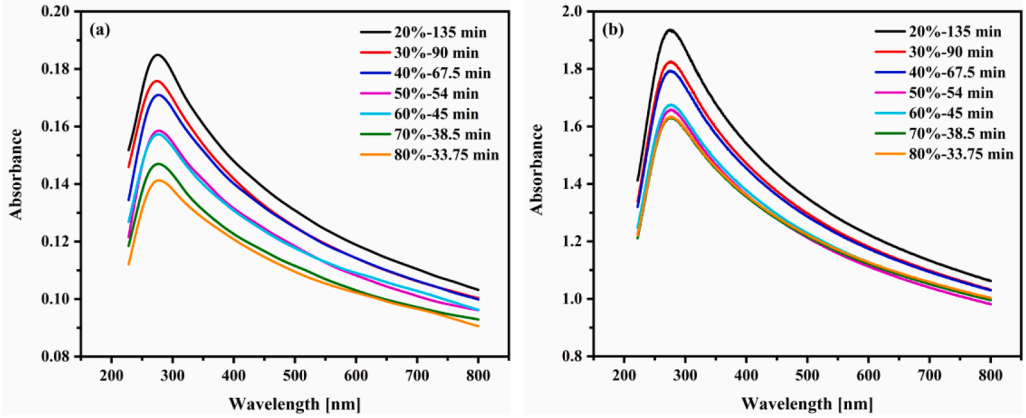

Fig. 7. Absorption spectra of FLG nanofluids prepared under the constant ultrasonic energy consumption condition: (a) 0.01 wt% and (b) 0.1 wt%.

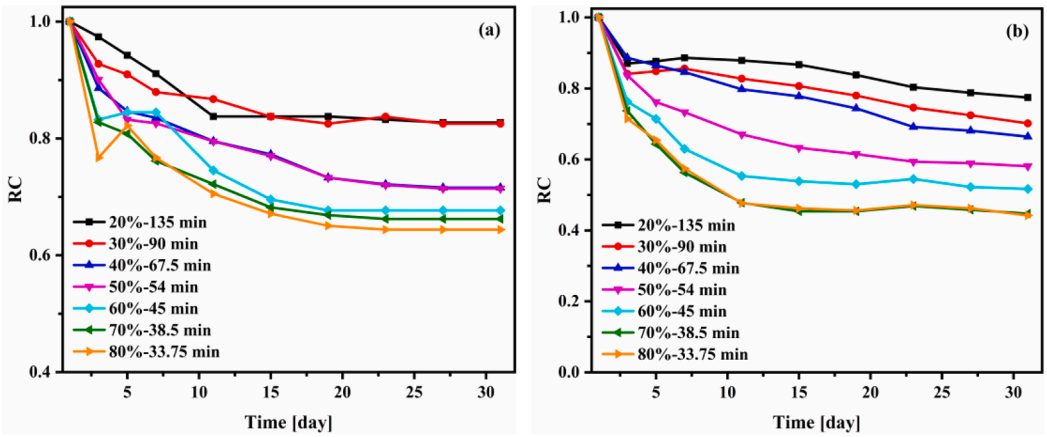

Fig. 8. Variations of the *RC*s of FLG nanofluids prepared under the constant ultrasonic energy consumption condition with time: (a) 0.01 wt% and (b) 0.1 wt%.

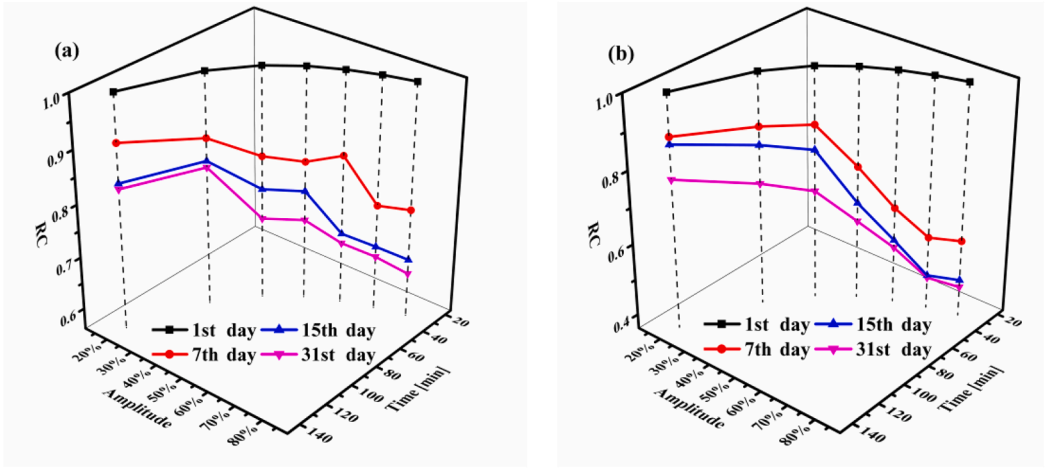

Fig. 9. Variations of the *RC*s of FLG nanofluids with ultrasonication power and time under the constant ultrasonic energy consumption condition: (a) 0.01 wt% and (b) 0.1 wt%.

ultrasonication time plays a more crucial role in enhancing the stability of FLG nanofluids than ultrasonication power. Therefore, it is recommended to use the relatively lower ultrasonication power with a longer time for preparing more stable FLG nanofluids under the constant ultrasonic power consumption condition.

3.4. Discussion

The primary purpose of ultrasonication treatment in the preparation of nanofluids by a two-step method is to use sound energy to break down the clustered nanoparticles into small-sized nanoparticles for better dispersion through the cavitation process. It is significant to find the optimal ultrasonication power and time for standardizing the preparation process of stable FLG nanofluids while saving energy. At the given ultrasonication time of 90 min, ultrasonication at 30% amplitude (450 W) is sufficient for dispersing FLG nanoparticles. Compared to ultrasonication power, ultrasonication power density may be more accurate because the volume of nanofluids would vary in different preparation processes. However, most previous work only considered ultrasonication power rather than ultrasonication power density, which lacks coordination for nanofluids' preparation process. Therefore, it is recommended to use the volume-independent parameter (ultrasonication power density) to characterize the intensity of the ultrasonication process.

Moreover, under the constant ultrasonic energy consumption condition, ultrasonication power seems to present a less crucial influence on the stability of FLG nanofluids than ultrasonication time. The main possible reason is that the ultrasonic cavitation process plays the role of breaking down FLG clusters and dispersing them in the base fluid simultaneously. Breaking down FLG clusters relies more on the local ultrasonication power density while dispersing the separated FLG nanoparticles to achieve the homogeneous dispersion relies more on the duration. Likely, the ultrasonication power considered in this work is at a high level, and the local power density around the probe is strong enough to break down the FLG clusters for all the cases. Thus, ultrasonication power seems an insignificant parameter, and ultrasonication time becomes the dominant factor for preparing stable FLG nanofluids in the present work.

4. Conclusions

In this work, FLG nanofluids were prepared using a two-step method under different ultrasonication conditions. Both sedimentation and optical spectrum analysis techniques were applied to evaluate the effects of ultrasonication power and time on the dispersion stability of FLG nanofluids. We found that the fresh FLG nanofluids after ultrasonication treatment had the highest concentration but were less stable. After undergoing rapid sedimentation in the first few days, FLG nanofluids became practically stable. Besides, ultrasonication treatment at 30% amplitude for 90 min was found sufficient for proper dispersion of FLG

nanoparticles. Under the constant ultrasonic energy consumption condition, the stability of FLG nanofluids prepared at higher amplitudes than 30% with reduced ultrasonication time deteriorated significantly, while for FLG nanofluids prepared at 20 % amplitude with longer ultrasonication time, they presented superior stability to the referential case. The optimum ultrasonication treatment for stable FLG nanofluids preparation was found at 20% amplitude (3000 W/L) for 135 min. This study indicates that the stability of FLG nanofluids is more sensitive to the ultrasonication time than power, mainly due to the relatively high ultrasonication power density considered in this work. To our best knowledge, the synergetic effects of ultrasonication power and time on the stability of FLG nanofluids under the constant ultrasonic energy consumption condition were examined for the first time. Our results would reference the preparation of stable FLG nanofluids through ultrasonication treatment. However, the considered ultrasonication power is relatively high. Further work should focus on the lower ultrasonication power to determine the minimum ultrasonic energy needed for preparing stable FLG nanofluids.

CRediT authorship contribution statement

Nianben Zheng: Conceptualization, Investigation, Supervision, Data curation, Formal analysis, Writing – review & editing, Funding acquisition. **Long Wang:** Investigation, Validation, Data curation, Formal analysis. **Zhiqiang Sun:** Supervision, Funding acquisition, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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