

## Dispersion and Stability Behaviour of Graphene and MWCNT Based Nano-Lubricant for Vapour Compression Refrigeration (VCR) System

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### Abstract:

Incorporating nanoparticles into oil compressors marks a significant advancement in scientific research. It has been widely demonstrated that the inclusion of nanoparticles can markedly improve the system's efficiency by reducing energy consumption, decreasing evaporator temperatures, and increasing condenser temperatures. Nanoparticles made of carbon, including those of metal, metal oxide, and metal hybrids, are particularly valued for their superior properties and are deemed to have considerable potential for achievements. This study focuses on examining the stability of graphene and multi-walled carbon nanotubes (MWCNT) in polyolester (POE) oil as a precursor to replacing conventional lubricating oil in refrigeration systems separately. Graphene nano-lubricants were tested at concentrations ranging from 0.1 to 0.3 g/L with the selected surfactant, cetyltrimethylammonium bromide (CTAB), in a 1:1 ratio, while MWCNT nano-lubricants were tested from 0.5 to 0.7 g/L. All samples were prepared using a two-step method that involved magnetic stirring at 1250 rpm for 45 minutes, followed by ultrasonication for 30 minutes. The samples underwent evaluation through three methods of observation. Initial visual observation was conducted immediately after production, then after a 24-hour settling period, and finally, after seven days, the three most optimal samples were identified. For MWCNT, an additional stability test was necessitated due to the high concentration levels, making it challenging to determine the three most stable concentrations. The UV-VIS spectrophotometer played a crucial role in analyzing the stability of the samples, providing data that enabled more precise results. The absorption peak at a wavelength of 294 nm reached its highest absorption value of 0.892 (a.u.) at a concentration of 0.2 g/L. In the case of MWCNT, after testing all samples, concentrations of 0.5, 0.55, and 0.65 g/L were identified as the best options based on peak absorption at different wavelengths, with their peaks at 4.87, 4.045, and 4.116 (a.u.), respectively. The Zeta Potential Analyzer confirmed the stability of these concentrations, indicating a moderate stability with values around -37.4 mV. This level of stability was also observed in MWCNT samples, with the three chosen concentrations showing excellent stability at 88.4, -84.9, and -137 mV, respectively. Among all tested nano-lubricant samples, those with concentrations of 0.2 g/L and 0.65 g/L for Graphene/CTAB/POE and MWCNT/POE, respectively, demonstrated the highest

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stability and optimum performance. This suggests that these concentrations have the potential to serve as replacements for POE oil in refrigeration systems.

**Keywords:** nanoparticles, nanofluids, VCR system, nano-lubricant, hybrid

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## 1. Introduction

In the vapour compression refrigeration (VCR) system, many experiments have been conducted by researchers over the years to improve the performance of the system (Nabati & Saadat-Targhi, 2021; Vashisht & Rakshit, 2021). One possible method to enhance the system is by having innovative lubricant oil (Górny et al., 2022; Kumar et al., 2023; Prabakaran et al., 2022). Using the pure lubricant oil and pure refrigerant in the refrigerant system already had results such as low temperature in the evaporator and high temperature in the condenser part. However, the system would be more efficient if the refrigerant were changed or additive materials such as nanoparticles were added to the lubricant oil. That is because nanoparticles can help increase the heat transfer coefficient and thermal conductivity value, which lowers energy consumption (Vamshi et al., 2022, p. 202). Recently, researchers have paid much attention to mixing nanoparticles in lubricant oil solution as advantageous, and it has been proven by the high system efficiency value displayed (Joshi et al., 2021; Salem, 2020).

Numerous studies predominantly focused on experimenting with metal oxide and metal hybrid nanoparticles. Despite the impressive outcomes from nano-lubricant experiments that utilized metal, metal oxide, and hybrid nanoparticles, carbon-based nanoparticles stand out due to their superior thermal conductivity and heat transfer coefficient (Ali et al., 2021). Graphene and multi-walled carbon nanotubes (MWCNT) have garnered significant interest among researchers for their distinctive features, including unparalleled thermal conductivity (up to 5300 W/mK), nanoparticle size, straightforward synthesis methods, prolonged suspension stability, extensive surface area, heightened surface energy and activity, reduced erosion, and lower pumping power requirements (Le Ba et al., 2020; Yang et al., 2020). For example, certain studies have explored the effects of using minimal concentrations of graphene/MO nano-lubricant in systems with 60g-R600a and 50g-LPG, finding a reduction in compressor work and an increase in refrigerant effect by approximately 20%, with a COP enhancement of 52.38% for the 60g-R600a system and 47.83% for the 50g-LPG system, at a working concentration of 0.2 g/L (T. Babarinde et al., 2020, 2021). Furthermore, Akhtar & Rajput (2023) performed an additional study to assess the MWCNT/POE at various concentrations (ranging from 0 to 1 g/L, in 0.2 intervals). At a concentration of 0.8 g/L, they observed a notable improvement in R134a's performance, which corresponded to a 21.46% decrease in compressor work. In another investigation, Babarinde et al. (2021) examined the effects of substituting mineral oil with MWCNT nanoparticles on the performance of 60g-R600a at different concentrations. Operating at the highest concentration of 0.6 g/L led to an increase in the COP value to 2.9.

Achieving a homogeneous solution with nano-lubricants in refrigerant systems presents a significant challenge. One inherent issue is the nanoparticles' propensity to aggregate, which

compromises the quality of the nano-lubricant (Said et al., 2023). The stability of nano-lubricants is profoundly affected by variables such as the type of nanoparticles, the lubricating oil used, surfactants, concentrations, and the methods employed in their production. Consequently, stability testing becomes essential to ensure that nanoparticles are evenly dispersed within the solution, thus preventing pipeline blockages and the inefficient function of nanoparticles in the compressor section. The most common methods for assessing stability include visual inspection, ultraviolet-visible (UV-vis) spectrophotometry, and zeta potential analysis

The surfactant type significantly influences a sample's appearance and condition, as noted by Al-Janabi et al. (2021). The use of zeta potential testing verified the quality of each sample, reinforcing the outcomes of the experimental investigation. The stability of the graphene/POE nano-lubricant (0.1 vol%) notably improved, reaching a zeta potential of -49.4 mV, with the addition of CTAB, compared to a -47.3 mV zeta potential observed at a different surfactant concentration. Conversely, integrating MWCNT with POE oil resulted in a zeta potential of -45.5 mV, with the nano-lubricant's quality diminishing upon adding surfactants such as CTAB, SDBS, or Span-80. Therefore, determining the optimal surfactant-to-nanoparticle ratio is critical, as the surfactant type and its quantity directly impact the nano-lubricants' quality.

Most experiments utilized a variety of raw materials at distinct concentrations, incorporating surfactants and employing different preparation methods, all of which significantly influence the outcomes. This ongoing interest in nano-lubricants for refrigeration systems drives researchers to continually seek optimal results before fully replacing traditional lubricant oils. In this study, graphene and MWCNT concentrations ranged from 0.1 to 0.3 g/L and 0.5 to 0.7 g/L, respectively, with increments of 0.05 g/L. Specifically, CTAB was chosen as the surfactant for graphene. This strategic selection of concentrations and surfactant, aligned with prior research, aimed to enhance the experimental results.

## **Experimental methods**

### **Materials**

The graphene used in the experiment was a pure powder with a purity of over 98% and a size of less than 10  $\mu\text{m}$ . The colour of the graphene was grey, and it consisted of approximately 20-40 layers with 100% graphite carbon. Additionally, pure, dark-grey MWCNT powder with a purity of over 99% and 100% carbon was used as another nanoparticle. It measured about 20 nm/ 10  $\mu\text{m}$ . Both nanoparticles were produced by Btcorp Generique Nano Private Ltd. The type of compressor oil mixed with the nanoparticle was POE oil (Emkarate RL 32H). The viscosity of the lubricant oil was 32.5 cSt at 40°C and 5.8 cSt at 100°C.

The nanoparticle and the surfactant were measured accordingly, and each sample was mixed in 100 ml of lubricant oil using a magnetic stirrer at 1250 rpm for 45 minutes. After that, the samples were sonicated for 30 minutes. Then, each sample in a beaker was divided into three different bottles to undergo different stability testing. All samples were tested by visual inspection, zeta potential, and UV-vis. Visual observation was the simplest and most cost-effective way, in which all samples were investigated after preparation. The samples were

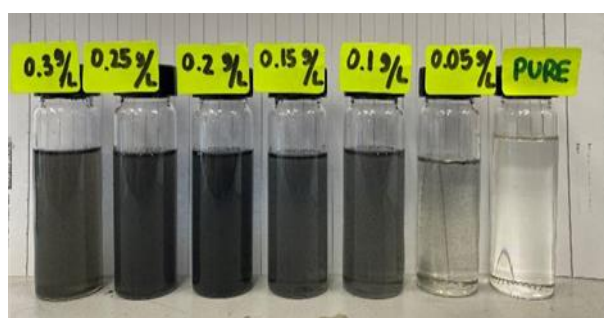
observed after 24 hours and after seven days at room temperature in stable condition and on a flat surface. The longer period of time would be applied if there was no sedimentation visually. Next, the three samples that were chosen after the visual observation results were injected into their cuvettes for the UV-VIS spectrophotometer testing, and the wavelength range was 200–700 nm. Additionally, the Malvern zeta sizer was used to determine the value of zeta potential for those three concentrations to confirm the UV-vis value results.

## Results and discussion

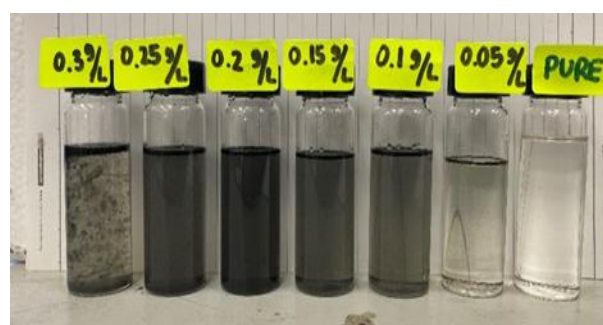
### Visual Observation

The investigation of each sample began after all samples were produced. Based on the samples in Figure 1a, it has been observed that addition of CTAB in graphene-lubricant enhance the stability of the nanofluid solution. From the visual observation, it has been observed that increasing the graphene concentration in the lubricant makes the nanofluid becomes darker. After 24 hours, most of the samples are stable (Figure 1b). The positively charged head group of CTAB creates additional charges on the graphene surface and increase the repulsive force which led to the stability of graphene in the lubricant oil and minimised the agglomeration. Besides that, the stability of the graphene in the lubricant oil also depends on the graphene concentration, Van der Waals forces attraction and electrical repulsion forces (Alexander et al., 2021). In addition, appropriate production methods, such as the duration of magnetic stirring and ultrasonication process, can also help reduce agglomeration and improve the dispersion of nanoparticles in all samples of graphene nano-lubricant (Atif & Inam, 2016).

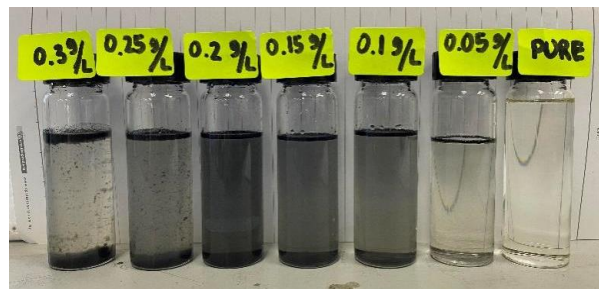
However, for 0.3 g/L sample, the graphene starts to agglomerate after being left for 24 hours. Then, after 7 days, sample with 0.3 g/L graphene showed sedimentation at the bottom of the vial (Figure 1c). Presence of higher graphene concentration in lubricant makes the graphene particles closer to restack as the Van der Waals force becomes greater. Thus, graphene starts to aggregates and form cluster, which makes it unstable and sediment at the bottom of the fluid. The aggregated graphene in lubricant oil is not accepted as it can results in clogging and abrasion problems in the VCR system.



(a)



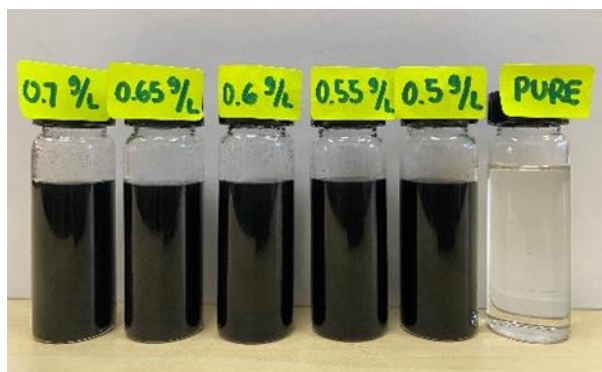
(b)



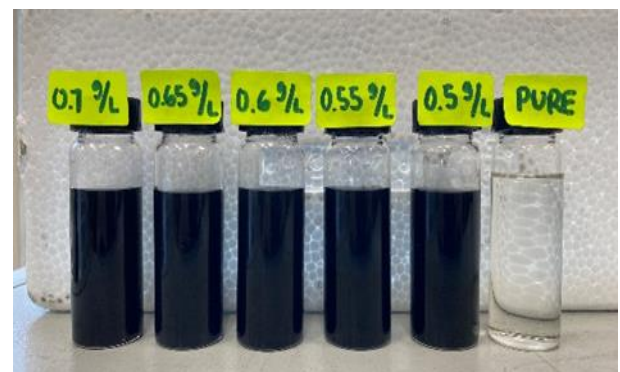
(c)

**Figure 1.** Graphene-nano lubricant (a) after preparation, (b) after 24 hours of preparation, and (c) after seven days of preparation.

Figure 2a illustrates the challenges of selecting the appropriate concentrations of MWCNT nano-lubricants to replace pure Polyolester (POE) oil as compressor oil, highlighted by the significantly dark and concentrated colour of each sample. It became apparent only after three months of observation that there was minimal sedimentation at the bottom layers of the solution (Figure 2b). This finding was confirmed by previous experiment by Choi et al. (2021), which showed that the visual appearance remained stable for 72 hours following preparation when using 0.1 vol% MWCNT/POE oil. Additionally, Salem (2020) observed no sedimentation in the solution even after 60 days of employing a 0.5 wt% MWCNT/POE nano-lubricant. In order to obtain the difference of each MWCNT sample, the small amount of solution was put into the cuvette as shown in Figure 3. The darker solutions were 0.65, 0.55, and 0.5 g/L while other samples also blended well and no agglomeration visually, but the colour of solution has less concentrated. The darkest solution was 0.5 g/L which was the lowest concentration of MWCNT samples. Based on visual assessment, 0.5 g/L was the most appropriate concentration because from the colour of the solution, it was very concentrated compared to other samples and it showed that dispersibility of less nanoparticles in the lubricant makes the solution more stable than having more MWCNT has the possibility of faster forming agglomeration or sedimentation. Even though it is not visible at the bottom or bottom of the solution, there is a high possibility of agglomeration forming in the MWCNT solution if there are too many nanoparticles in it (Joshi et al., 2022).

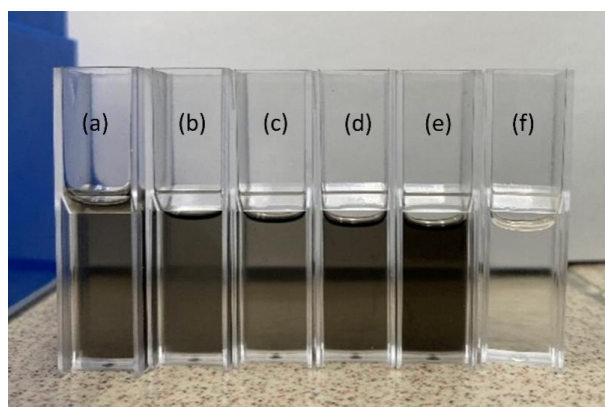


(a)



(b)

**Figure 2.** MWCNT nano-lubricant (a) After preparation, and (b) After three months of preparation.



**Figure 3.** Dispersion study (a) 0.7 g/L, (b) 0.65 g/L, (c) 0.6 g/L, (d) 0.55 g/L, (e) 0.5 g/L, and (f) pure oil

Thus, it has been observed in the early stability testing, which was conducted visually, identified 0.1, 0.5, and 0.2 g/L as the most stable concentrations for graphene nano-lubricants and 0.5, 0.55, and 0.65 g/L for MWCNT nano-lubricants.

#### Dispersion and stability analysis via UV-vis

Ultraviolet-visible spectrometry (UV-vis) is a reliable technique for assessing the stability of nanofluid. It involves emitting light of varying wavelengths through a solution and measuring the absorbance at each wavelength (Mohd Zaki et al., 2017). A higher level of absorbance (y-axis) at the peak wavelength indicates excellent dispersion of nanoparticles in the base oil (Awang et al., 2019). Additionally, UV-vis spectroscopy can be used to estimate changes in absorbance of the suspension over time.

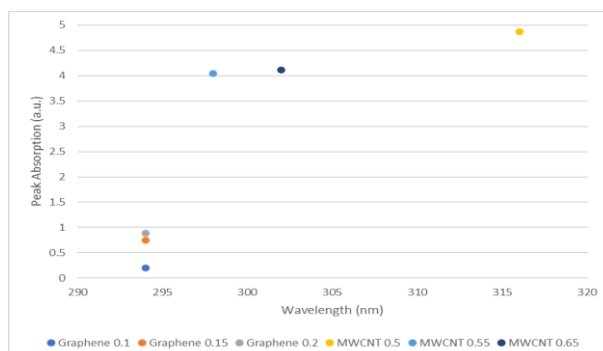
Based on Tabel 1 and Figure 4, it is noticeable that all the peak absorption of graphene samples was at the same wavelength, 294 nm. Meanwhile, all MWCNT samples reached their peak absorption at different wavelength. In graphene nano-lubricant result, the effect of adding more nanoparticles in base oil has the great improvement to reach the higher absorption value. The same trends also happened in Mohd Zaki et al., (2017) and Aljuwayhel et al., (2023) experiment while working with Nanodiamond/POE and SiO<sub>2</sub>/PAG, respectively. This was proven by having 0.2 g/L, which was the highest concentration in graphene samples, the value was at 0.892 a.u at 294 nm.

If compared to MWCNT result, the result was five times higher than graphene result. In MWCNT data, by having a higher concentration value does not necessarily mean that it will produce a higher peak absorption value as well. The MWCNT result actually has a opposite trend with graphene. According to the findings in table 1, the lower the concentration value of the MWCNT sample, the higher the peak absorption value obtained. It indicates that the nanoparticles in the POE oil were distributed more uniformly, and increasing the concentration value caused the nanoparticles ineffective in solution. As mentioned earlier, this statement was related to the visual observation (Figure 3), even after preparation some MWCNT nanoparticles started to agglomerate due to the higher amount of nanoparticle. So that the samples were tested in the UV visible spectrophotometry was not very concentrated more.

More details, the peak absorption of 0.55 and 0.65 g/L almost had the same value, but had the large gap with 0.5 g/L. The presence of adding more nanoparticles had the disadvantage in dispersibility and stability solution and this situation also happened in Singh et al., (2022) while working with SiO<sub>2</sub> in Madhuca Indica oil. By working with 0.4, 0.8, and 1.2wt%, the higher peak absorption was reached with 0.4wt% which was the lowest concentrations. It occurred due to the higher amounts of nanoparticles that made the nanoparticles tend to agglomerate quickly.

**Table 1.** UV-visible of graphene and MWCNT nano lubricant

|                    | Samples concentration<br>(g/L) | Peak Absorption<br>(a.u.) | Wavelength<br>(nm) |
|--------------------|--------------------------------|---------------------------|--------------------|
| Graphene-lubricant | 0.1                            | 0.197                     | 294                |
|                    | 0.15                           | 0.751                     | 294                |
|                    | 0.2                            | 0.892                     | 294                |
| MWCNT-lubricant    | 0.5                            | 4.87                      | 316                |
|                    | 0.55                           | 4.045                     | 298                |
|                    | 0.65                           | 4.116                     | 302                |



**Figure 4.** The peak absorption for each concentration nano-lubricants.

### Zeta Potential Analysis

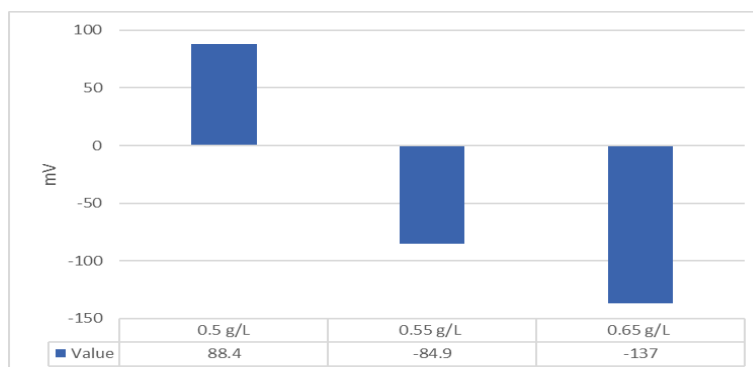
Zeta potential is a parameter used to determine the surface charge of nanoparticles in a colloidal solution. It was observed that samples with concentrations of 0.1 g/L and 0.15 g/L had unstable mixing solutions. However, upon visual inspection, these solutions appeared better than those with concentrations of 0.3 g/L, 0.25 g/L, and 0.05 g/L. The instability of the graphene was due to different concentration or particle size in a lubricant (Sandya et al., 2021). In addition, sonication process may affect the stability of the graphene nanoparticles in the lubricant. As the graphene concentration increased, the zeta potential value was found to decrease to -37.8 mV for the graphene-lubricant concentration of 0.2 g/L indicating the highest stability. The graphene particles break and disperse freely in the solvent after sonicated which subsequently reduce the particle charge density in zeta potential (Demirkır & Ertürk, 2020). Therefore, this result should be considered before replacing pure compressor oil (T. Babarinde et al., 2020; Madyira et al., 2022) for long-term use in VCR systems. This has been confirmed through

experiments by various researchers, who found that the application of 0.2 g/L in different mass R600a led to outstanding coefficient of performance (COP) compared to higher concentrations in domestic refrigeration systems (Babarinde et al., 2020; Madyira et al., 2022).



**Figure 7.** The result of Zeta Potential in graphene nano-lubricants

In contrast, based on the MWCNT result, it shows that all three concentrations had the excellent stability because of their values, which were more than  $\pm 60$  mV. The stability of MWCNT/POE 0.1–0.5 wt% was also examined by Salem, (2020); the results ranged from +30.34 to +31.5 mV. Furthermore, Al-Janabi et al., (2021) conducted an additional test with MWCNT/POE 0.1 v% without surfactant and the zeta potential result was -45.5 mV. Therefore, compared to the previous experiment, the current MWCNT and POE oil mixture results showed greater stability. Moreover, in the long time period, MWCNT solutions have the potential to replace pure compressor oil since they were more stable than graphene nano-lubricant.



**Figure 8.** The result of Zeta Potential in MWCNT nano-lubricants

## Conclusion

All the experiments and tests were conducted to determine the most stable concentrations of graphene and MWCNT nano-lubricants, specifically at 0.1 - 0.3 g/L and 0.5 - 0.7 g/L, with interval 0.05 g/L respectively. The initial observation after preparing all the graphene samples was the appearance of sedimentation on the surface. Among the samples, the concentrations of 0.2 g/L, 0.15 g/L, and 0.1 g/L showed the most stability with minimal sedimentation and better dispersion. However, in MWCNT samples, there was no sedimentation visually, but the difference of concentrated in each sample was indicated after put the samples into the cuvette.

The most concentrated MWCNT nano-lubricants were 0.5, 0.55, and 0.65 g/L. After visual inspection, the UV-vis testing was undergone and showed that the highest peak absorption among graphene samples was belong to 0.2 g/L and for MWCNT samples was belong to 0.5 g/L. The zeta potential testing supported the UV-vis results by showing their values where the 0.2 g/L was categorized as moderate stability and all three MWCNT solutions had the excellent stability. The highest value was achieved with the use of 0.65 g/L with significant differences with other MWCNT samples. It can be concluded that the 0.2 g/L graphene nano-lubricant and 0.65 g/L MWCNT nano-lubricants have the high possibility of replacing the POE oil as the compressor oil in HVAC system.

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