Studies on effect of various surfactants on stable dispersion of graphene nano particles in simarouba biodiesel

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Abstract. Discovery of graphene has inspired researchers a range of potential applications. Conventional fluids with nanoparticle of size 1-100nm dispersed in them are called Nanofluids. Nanofluids are found to possess increased physical and thermal properties like thermal diffusivity, viscosity, thermal conductivity and convective heat transfer coefficient. These properties can be explored in the field of IC engines as additives in improving the engine performance and reducing the harmful emissions like NOx, CO, UBHC etc. In this work, a comparative study of two anionic dispersants Sodium Dodecyl Sulfate (SDS) and Sodium Dodecyl Benzene Sulfonate (SDBS) as dispersants for stable dispersion of graphene in Simarouba biodiesel with 20% blend in diesel (SME20) was made. Nanofluids samples of graphene at 20ppm and different mass fraction of SDBS & SDS were prepared. Dispersion of graphene was achieved by ultrasonication and magnetic stirring. Dispersion was characterized with Ultra Violet Visible (UV–Vis) spectroscopy. The UV–Vis absorption spectra revealed the presence of Graphene with characteristic λmax around 250 nm. Experimental result showed that with increase in concentration of dispersant, the value of absorbance also increased. There is a linear relation between stability of dispersion and UV absorbency. An optimum graphene-to-surfactant ratio was determined. Surfactant concentration above or below this ratio was shown to decreases the stability of dispersion. For a mass fraction of 1:4, Graphene to SBDS ratio, the absolute value of UV absorbency was highest. Dispersion stability of SDBS was better than SDS at all concentration levels.

1. Introduction.

Biodiesels are renewable and eco-friendly alternative fuels for CI engine. They have higher viscosity, density and cetane number than diesel. Biodiesels are oxygenated fuels with 10–15% oxygen by weight which reduces the exhaust emissions particulate matter, carbon monoxide, sulfur oxides, and unburnt hydrocarbons compared to diesel. But due to the lesser energy content and complete combustion, it gives poor performance and increase in NOx emission. In order to improve the performance and emission especially NOx, nano additives added biodiesel have become an essential part of today’s fuels. Use of fuel additives in the blend of biodiesel, improve performance, combustion, and diminish emission characteristics and also improved fuel properties which enhance the combustion characteristics. Recent advances in nano science and nanotechnology enables production, control and characterization of nonmaterial. Another advantage of nano particle application in IC engines is their size, because of no chance for fuel injector and filter clogging as in the case of micron sized particles.

Nanofluids are engineered fluids, by dispersing nanoparticle of size 1-100nm in conventional base fluids. Due to high surface area, nanoparticle dispersed gets aggregated / agglomerate / settle down in the base fluid. Preparing a stable & Homogeneous Dispersion throughout the fluid is a very big challenge.

Graphene is a flat monolayer of carbon atoms that are tightly bonded to each other. With its unique 2-dimensional (2D) structure it has gained great interests since it was first discovered. It has large specific surface area (theoretical 2630m²/g, experimental 400-700 m²/g), and thermal conductivity of...
about 5000 W/m K. There are 2 primary methods for preparation of Nanofluids: 1-step process and the 2-step process. 2-step process is commonly used in the preparation of Nanofluids by mixing base fluid with commercially available nano powders. In this study nanofluids (nanofuels) were prepared using 2-step process.

1.1 Stability Mechanisms.

Stability of the dispersed nanofluids is most crucial issue can be hampered by particle aggregation. However, nanoparticles recombine due to attractive van der Waals forces after the energy supplied by ultrasonication is exhausted. The recombination is to be avoided either by decreasing the van der Waals forces by refractive index matching or by introducing repulsive forces (steric, electrostatic, or electrosteric forces). If force of attraction prevails over repulsive force, then the particles aggregate in clusters. Therefore enhancement of repulsive forces over attractive forces can prevent particle aggregation and ensure stability. The mechanical method adopted in this study involves ultrasonication and high-shear mixing in a magnetic stirrer. A combination of mechanical dispersion and chemical method of surface functionalization to improve their chemical compatibility with the base fluid using surfactants (surface active agents) can improve their wetting or adhesion characteristics and reduce the tendency to agglomerate in the continuous phase solvent [1]. Stabilization using surfactants can be done by two mechanisms: electrostatic stabilization using ionic surfactants and steric stabilization using nonionic surfactants. Any nanoparticle coated with surfactants is stable against re-aggregation by the presence of repulsion between nearby surfactant-coated nanoparticle [2].

2. Materials and Method

2.1 Biodiesel-Simarouba glauca

It belongs to family simaruba ceae, commonly known as “The Paradise Tree” or “King Oil Seed Tree”, is a versatile multipurpose evergreen tree. Simarouba glauca Seed oil is transesterified to obtain Simarouba Methyl Ester (SME) using UAS [4] production model methods. The properties of the oil and the biodiesel produced were tested and compared to American Society of Testing and Materials (ASTM) standards and European standards to determine quality of the biodiesel. The Simarouba glauca biodiesel was then blended with commercially available petro diesel purchased from retail outlet.

Table 2.1 Fuel properties of Simarouba biodiesel blend SME20

| Sl. No | Properties    | Units  | SME20 |
|--------|---------------|--------|-------|
| 1      | Viscosity     | Cst    | 2.83  |
| 2      | Density       | Kg/m³  | 831   |
| 3      | Flash point   | °C     | 67    |
| 4      | Fire point    | °C     | 74    |
| 5      | Calorific value | KJ/Kg | 41458 |
2.2 Graphene

The nanoparticle used in our study is graphene. The commercially available graphene was purchased from sigma Aldrich, Germany. Graphene is a two-dimensional (2D) material, formed of a lattice of hexagonally arranged carbon atoms. The properties of graphene used in or study are given in Table 2.2.

Table 2.2 Properties of graphene.

| Sl.No | Parameter                  | Property          |
|-------|----------------------------|-------------------|
| 1     | Manufacturer               | Sigma Aldrich     |
| 2     | Average Particle Size      | 22.5 -26nm        |
| 3     | Surface area               | 492 m²/gm         |
| 4     | Purity                     | 99.5 %            |
| 5     | Thermal Conductivity       | 3000 W/m K        |

2.3 Surfactants.

The role of a surface active agent (surfactant) is to provide an effective and efficient coating to induce electrostatic or steric repulsions that can counterbalance van der Waals attractions. Based on the composition of head, surfactants can be classified into three classes: nonionic surfactants - without charge groups in their head, anionic surfactants with negatively charged head groups and cationic surfactants with positively charged head groups. The stabilization of dispersions in ionic surfactants results from the electrostatic repulsion between the ionic hydrophilic heads that coat adjacent graphene sheets. In this study, the electrostatic repulsion provided by adsorbed surfactants stabilizes graphene against the strong van der Waals interaction between graphene surfaces, and prevents agglomeration. In the present study a comparative analysis of 2 surfactants for graphene dispersion and dispersing power of surfactants has been analyzed experimentally. This study provides insights into some parameters for optimization of dispersion of graphene using SDS and SDBS surfactants. The significance of using a particular ratio of surfactants and graphene has been established for obtaining optimum dispersion, which may be cited as a relatively new finding in this area of research. From our study, the optimum graphene to SDS and SDBS surfactant ratio turns out to be the most important parameter in nanotube dispersion. We have optimized this ratio for these surfactants.

We systematically studied effects of these two readily available anionic surfactants, Sodium Dodecyl benzene Sulfonate (linear alkyl benzene Sulfonate) is a series of organic compounds with the formula C₁₂H₂₅C₆H₄SO₃Na. It is a colorless salt with useful properties as a surfactant.

![Figure 2.1 Structure of Sodium Dodecyl Benzene Sulfate](image)

Sodium Dodecyl Sulfate (SDS). Anionic head groups of SDS include long-chain fatty acids; alkyl sulfate.

![Figure 2.2 Structure of Sodium Dodecyl Sulfate](image)
Both SDS & SDBS are excellent electrostatic stabilizers with high affinity to absorb onto nanoparticle particle surfaces. They are commercially available and manufactured by SD-Fine-Chem Ltd, Mumbai, are purchased.

3. Preparation of Nanofluids:

When inorganic nanoparticle is used for an inclusion of particles as reinforcement, dispersion of nanoparticle is indispensable because of nanoparticle in various liquids tending to agglomerate. The prepared Nanofluids should be an agglomerate-free and stable suspension without sedimentation for long durations. The ultrasonication technique is most suited method for [3] preparing any nanofluid, as it facilitates possible agglomerate nanoparticle back to nanometer range. In this technique, ultrasonic waves are supplied to the liquid suspension in 2 ways. In the probe type sonicater (direct sonication) by immersing an ultrasound probe into the fluid with particles dispersed, or in bath type sonicater (indirect sonication) by introducing the sample container with the suspension into a bath containing a liquid through which ultrasonic waves are propagated. Ultrasonication of fluids leads to 3 physical mechanisms: Cavitation of the fluid, localized heating, and formation of free radicals. Cavitation, formation and implosion of bubbles, can cause dispersion and fracture of solids. Frequency of ultrasound waves decides the bubble size in the fluid. Low frequency waves produce large bubbles while high energy forces occur as they collapse. Increased frequency reduces bubble size and nucleation, so that cavitation is decreased. Bubbles nucleating at the solid surfaces, rapidly expanding can push particles apart. Dispersed solid particles can remain separated after bubble collapse if they are wetted by the fluid phase and if the volume fraction of nanoparticle in the fluid phase is low enough so that solid movement is possible.

The Simarouba Methyl Ester (SME-20) Biodiesel blend is prepared with petrodiesel in volume fraction of 80:20 Diesel-SME. The dosing level of the graphene nanoparticle (by weight) in the base fluid was taken as 20 ppm. To find the optimum graphene to-surfactant ratio for each surfactant, the concentration of surfactants was varied from 1.2 to 1.5% in steps of 0.1%, keeping the amount of graphene constant. Required quantity of nanoparticle for dosing level was measured using a precision electronic balance. The surfactant- graphene ratios 1:2, 1:3, 1:4 and 1:5 were prepared.

The sonication was done on a bath type digital ultrasonic cleaner at an ultrasonic frequency of 20 KHz with 5w power shown in Figure 3.1. Sonication is done at a constant temperature of 30°C, for 60 minutes, followed by stirring on a magnetic stirrer at a constant speed of 750rpm for a period of 30 minutes. Thus prepared nanofluid samples are named as given in the Table 3.3 and Table 3.4. The prepared samples were kept in a glass tube and periodically observed for their long term stability.
Table 3.3 Composition of samples prepared with various concentration of SDBS

| Sl.No | Sample          | Diesel (%) | SME (%) | Graphene (ppm) | Graphene to SDBS ratio |
|-------|-----------------|------------|---------|----------------|------------------------|
| 1     | SME20/1:1SDBS   | 80         | 20      | 20             | 1:1                    |
| 2     | SME20/1:2SDBS   | 80         | 20      | 20             | 1:2                    |
| 3     | SME20/1:3SDBS   | 80         | 20      | 20             | 1:3                    |
| 4     | SME20/1:4SDBS   | 80         | 20      | 20             | 1:4                    |
| 5     | SME20/1:5SDBS   | 80         | 20      | 20             | 1:5                    |

Table 3.4 Composition of samples prepared with various concentration of SDS

| Sl.No | Sample          | Diesel (%) | SME (%) | Graphene (ppm) | Graphene to SDS ratio |
|-------|-----------------|------------|---------|----------------|-----------------------|
| 1     | SME20/1:1SDS    | 80         | 20      | 20             | 1:1                   |
| 2     | SME20/1:2SDS    | 80         | 20      | 20             | 1:2                   |
| 3     | SME20/1:3SDS    | 80         | 20      | 20             | 1:3                   |
| 4     | SME20/1:4SDS    | 80         | 20      | 20             | 1:4                   |
| 5     | SME20/1:5SDS    | 80         | 20      | 20             | 1:5                   |

4. Characterization of dispersion:

UV-Vis spectral analysis: ultra violet spectral absorbance analysis is an effective and efficient way of evaluation of dispersion stability of nanofluids. If the nano particles dispersed in fluids have characteristic absorption bands in the wavelength 190nm to 1100 nm, this method is an easy and reliable method [2]. The variation of nanoparticle concentration in nanofluids with sediment time can be obtained by the measurement of absorption of nanofluids, as there is a linear relation between the nanoparticle concentration and the absorbance. UV–Vis spectroscopy gives quantitative results corresponding to concentration of nanofluids [2]. It is one of the most reliable methods to evaluate the stability of nanofluids. For UV-Vis spectral analysis, initially, baseline is fixed considering the SME20 as the reference sample as well as the specimen sample. After fixing the baseline, SME20 is taken as the reference sample and graphene nanofluids as the specimen samples. To characterize the dispersion, absorbance values were recorded at a range of 250nm to 750nm. For the stability study, data are obtained for a regular time interval of one week.

5. Result and Discussion:

According to Derjaguin, Verway, Landau and Overbeek (DLVO) theory [5], dispersion stability of nano particles in fluids is determined by the sum of van der Waals attractive forces & electrical double
layer repulsive forces between nanoparticles when they come close to each other during the movement (Brownian motion) inside the fluid. If the attractive force is larger than the repulsive force, the two particles that collide may adhere together and form aggregates with increased size and may settle down due to gravity, and making the suspension unstable. In the absence of the surfactant, due to the lack of electrostatic repulsion, van der Waals attraction dominates and causes graphene sheets to form highly ramified aggregates. As the surfactant concentration is increases, growing repulsion from adsorbed surfactant causes the graphene sheets aggregates to develop a more compact structure due to an increase in sheet–sheet overlap area. Over the time, ramified graphene sheet aggregates restructure into more compact structures due to an increase in overlap area. Above the critical surfactant concentration, it s found that the dispersed state becomes increasingly stabilized, and the growing kinetic barrier prevents the aggregation for a longer period.

In this study, the prepared nanofluid samples were periodically tested by UV-Vis Spectroscopy using the standard size of cuvette. The nanofluids are kept in static condition, for a period of 8 weeks and the UV–Vis absorption spectra at different concentrations of graphene – surfactant ratio are taken. A standard absorption band for graphene in the UV region with maximum wavelength of around 250nm was found that confirms the presence of graphene in the dispersion [6]. The absorbance was measured at the end of each week. It was found that as the time is prolonged, absorbance value was reduced because the particles begin to agglomerate and settle down. Table 5.1 & Figure 5.1 show absorbance values at the end of each week when kept in static condition for the nanofluids dispersed with graphene using SDS as surfactant. The highest absorbance value of 3.187 immediately after the dispersion for graphene dispersed nanofluid using SDS surfactant with a graphene – surfactant ratio of 1:4. The absorbance value was minimum 2.051for the graphene - surfactant concentration ratio of 1:1. Whereas the absorbance value was found to decrease as the time was prolonged. Absorbance was 2.449 for graphene – surfactant ratio of 1:4 and 1.579 for graphene – surfactant ratio of 1:1 at the end of 8th week.

Table 5.1, UV–Vis absorption values over time for SDS surfactant

| Weak No. | GRAPHENE : SDS RATIO |
|----------|----------------------|
|          | 1:1 | 1:2 | 1:3 | 1:4 | 1:5 |
| Weak 0   | 2.051 | 2.166 | 2.493 | 3.187 | 2.773 |
| Weak 1   | 1.948 | 2.052 | 2.366 | 3.021 | 2.632 |
| Weak 2   | 1.896 | 1.998 | 2.303 | 2.942 | 2.562 |
| Weak 3   | 1.845 | 1.944 | 2.241 | 2.862 | 2.493 |
| Weak 4   | 1.804 | 1.901 | 2.191 | 2.798 | 2.438 |
| Weak 5   | 1.743 | 1.836 | 2.117 | 2.703 | 2.355 |
| Weak 6   | 1.681 | 1.771 | 2.042 | 2.608 | 2.271 |
| Weak 7   | 1.643 | 1.728 | 1.992 | 2.544 | 2.216 |
| Weak 8   | 1.579 | 1.663 | 1.917 | 2.449 | 2.133 |
Figure 5.1, UV–Vis absorption over time 8 weeks for SDS surfactant

Table 5.2 & Figure 5.2 show absorbance values at the end of each week when kept in static condition for the nanofluids dispersed with graphene using SDBS as surfactant. The highest absorbance value of 3.84787 immediately after the dispersion for graphene dispersed nanofluid using SDBS surfactant with a graphene – surfactant ratio of 1:4. The absorbance value was minimum 2.48052 for the graphene - surfactant concentration ratio of 1:1. Whereas the absorbance value was found to decrease as the time was prolonged. Absorbance was 2.96280 for graphene –surfactant ratio of 1:4 and 1.90995 for graphene – surfactant ratio of 1:1 at the end of 8th week. It was also observed that with increase in concentration of surfactants, the value of absorbance also increases [3]. This follows the Beer–Lambert’s statement; the absorbance is directly proportional to the concentration of the nanoparticle in the solution. But absorbance reduces after a certain concentration level of surfactant. The absorbance value was minimum 1.81 immediately after the dispersion for graphene dispersed nanofluid using SDS surfactant for the graphene - surfactant concentration ratio of 1:1. A highest absorbance value of 3.84787 for a graphene – SDBS ratio of 1:4, but the absorbance value was reduced to 3.35174 when the graphene – SDBS ratio was increased to 1:5.

Table 5.2, UV–Vis absorption values over time for SDBS surfactant

| Weak No. | GRAPHENE : SDBS RATIO |
|----------|-----------------------|
|          | 1:1                   | 1:2                   | 1:3                   | 1:4                   | 1:5                   |
| Weak 0   | 2.48052               | 2.61363               | 3.01296               | 3.84787               | 3.5174                |
| Weak 1   | 2.35647               | 2.48292               | 2.86225               | 3.65541               | 3.18417               |
| Weak 2   | 2.29446               | 2.41758               | 2.78693               | 3.55921               | 3.10032               |
| Weak 3   | 2.23245               | 2.35224               | 2.71161               | 3.46302               | 3.01653               |
| Weak 4   | 2.18284               | 2.29996               | 2.65135               | 3.38606               | 2.94949               |
| Weak 5   | 2.10842               | 2.22156               | 2.56096               | 3.27063               | 2.84894               |
| Weak 6   | 2.03401               | 2.14315               | 2.47057               | 3.15519               | 2.74839               |
| Weak 7   | 1.98443               | 2.09088               | 2.41032               | 3.07824               | 2.68136               |
| Weak 8   | 1.90995               | 2.01247               | 2.31993               | 2.96280               | 2.58080               |
Figure 5.2, UV–Vis absorption over time of 8 weeks for SDBS surfactant

Figure 5.3 shows the schematic of four-stage adsorption model of surfactant on to the graphene layer. Figure 5.3.a the adsorption of isolated surfactant monomers with the alkyl chain is oriented parallel to the surface, 5.3.b the formation of surfactant monolayer in which the alkyl chains are oriented parallel to the surface. 5.3.c The formation of hemi-cylindrical surface micelle prior to micelle formation in bulk solution. Figure 5.3.d shows the formation of micelle in bulk solution. Concentration of surfactant at which a dense monolayer forms on the graphene sheets was found. Surfactant concentration at the onset of surface micelle formation on graphene sheets, known as the critical surface aggregation concentration (csac), and formation of micelle in the bulk solution, known as the critical micelle concentration (cmc).

Both the dispersants used in this study are excellent electrostatic stabilizers with high affinity to absorb onto particle surface leading to high surface charges [7]. Surface electric charges are the major source of kinetic stability of dispersion. This electrostatic stabilization occurs by adsorption of ions on to the electrophilic surface of the graphene sheets. Adsorption creates an electrical double layer which results in a Columbic repulsion force between the nanoclusters.
Figure 5.3 Four stages of surfactant adsorption onto graphene:
(a) Adsorption of isolated surfactant monomers, (b) Adsorption of a surfactant monolayer. (c) Formation of hemi-cylindrical surface micelles (d) Formation of micelles in bulk solution.

Charged surfactant adsorbs with negatively charged part onto the graphene surface and forms an inner Helmholtz layer. Adsorption takes place according to the theory of adsorption isotherms. The surface coverage increases with an increasing surfactant concentration. Consequently the potential of the inner Helmholtz layer increases with the increasing surface coverage, leading to an increase in the charge, mutual repulsion and hence, an increase in the physical stability of the suspensions. But with increase in surfactant concentration, the concentration of Na+ increases and the Na+ group entering into the absorbed layer reduces the net charge of the graphene surface resulting in a weak dispersion. At high surfactant concentrations, electrolyte stabilizers can cause a decrease in the diffuse layer leading to a reduced surface charge, and a decreased physical stability. It is observed that excess surfactant did not affect significantly the stability of dispersion, but higher concentration (Critical Micelle concentration), lead to crowding effect of micelles which increase the viscosity [8]. Therefore it is important to find the optimal concentration for a stabilizer. The optimum ratio of graphene to surfactant is thus can be taken as 1:4, which gives most stable dispersion.

6. Conclusion.

The homogeneous and stable graphene nanofluids of different concentrations were successfully prepared by two-step technique with the aid of SDS dispersant. The dispersion stability scales linearly with the repulsive electrostatic potential barrier that stabilizes surfactant-coated sheets against aggregation [9]. The diffusion coefficient of micelles decreases as their concentration increases, because crowding effects can increase the viscosity [10]. A quantitative comparison of graphene dispersions in SME20 with two commonly used surfactants, each at their optimum concentration, was conducted by using the absorbance. UV–Vis absorption spectroscopy confirmed the presence of graphene dispersed in the nanofluid. From UV–Vis spectral method, it was also observed that the absorbance value of nanofluid was reduced with the increase in the static duration for any concentration of both the surfactant. The degree of absorbance is proportional to the amount of the particles per unit volume so that it can denote the dispersion stability of the particles [11]. A graphene to surfactant ratio of 1:4 gives most stable dispersion in simarouba biodiesel – diesel blend. SDBS is a better dispersant than SDS for stable dispersion of graphene in Simarouba biodiesel blend with petro diesel.

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