

## Impact of Oil Viscosity on Carbon Stabilization Using Polyisobutylene Succinimide (PIBSI) Dispersant

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Polymeric-based dispersant plays a crucial role in stabilizing carbon particles in oil system, where their concentration and the viscosity of the bulk oil play an important role in carbon interactions. In this work, carbon black particles were dispersed in hexane and decane separately as the model system. The model system used is able to produce neat fundamental understanding of the surface charging behavior of carbon particles in oil, influenced by the concentration of polyisobutylene succinimide (PIBSI) dispersant. The novelty of this work emphasizes on the carbon particles surface charging behavior in oil, influenced by the PIBSI dispersant. The particles' surface charge and aggregation behavior were investigated in two different oil viscosities. The surface charges of the carbon particles were calculated from the electrophoretic mobility measurement, and the aggregation behavior was evaluated using the hydrodynamic particle size measurement *via* the Dynamic Light Scattering (DLS) instrument and UV-Vis absorption. Comparatively, the magnitude of carbon effective surface charge in decane system was higher than in hexane without any PIBSI. The trend was the same at high PIBSI concentrations, with decane systems having larger charge magnitude, and particles in hexane showed nearly zero charges. The DLS measurements revealed that large carbon aggregates formed at low PIBSI concentration for hexane system; for decane system the result was inverse. The UV-Vis absorption spectra of the carbon particles in the decane system showed that the system with 5 wt.% of PIBSI had the highest absorption, where the peak of absorption spectra in hexane increased with an increase in the PIBSI concentration.

**Keywords:** Dispersant, Surface charge, Carbon, Aggregation, Interaction

### INTRODUCTION

The interaction between carbon particles in oil is vital in the development of its aggregation and stabilization behaviors. The interplay between carbon surface interaction and the destabilization phenomena is predominantly dependent on the carbon surface reactivity, oil viscosity, and amount/type of the added surfactant. The importance of carbon-derived colloids in oil surface interaction is still poorly understood, despite of its importance in many advanced applications including engine oil formulations and oil recovery systems [1,2].

Dispersants are surfactant-like amphiphilic molecules

that are commonly used to disintegrate carbon particles aggregation or inhibit carbon from forming large aggregates. Dispersants molecule is composed of a non-polar long-chain hydrocarbon tail and a polar moiety at the chain end [3]. In carbon particles laden oil-based systems, dispersants are extensively used to disperse solid particles in oil, occupying the solid-liquid interface, and solubilizing the polar products to avoid the formation of large aggregations. Therefore, dispersants are essential in engine oil formulation to breakdown carbon aggregation caused by the incomplete combustion of fuel in engine and eventually limit the soot production to the environment [4,5].

A polymeric-based dispersant is a type of extensively used dispersant in automotive engine oil formulations. It can be practicality engineered to have a long polymeric chain and

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a head with specific functional groups. Polymeric dispersants can be categorized into succinimide, ester, oxazoline thiophosphonate, and Mannich dispersants, based on the polar head functional group [6]. Succinimide and Mannich dispersants contain primary or secondary amine functional groups [7], whereas ester, oxazoline, and thiophosphonate dispersants contain hydroxyl functional groups [6]. The oil additive industry uses different classes of dispersant, for examples, polyalphaolens, polyisobutylene (PIB), polypropylene, and acrylate-based polymers [8,9]. The selection of appropriate dispersant types and concentrations are two crucial factors to ensure the dispersant can effectively inhibit the carbon aggregations and eventually soot release.

The key importance of this work predominantly relies on the colloidal carbon interparticle interactions in oil, and the development of the interactions when the polymeric dispersant was added. The stabilization of the colloidal system could be achieved by controlling the interparticle interactions, either via electrostatic surface charge or steric stabilization [5]. Recent works reported on the adsorption of multiple polymeric dispersants architecture on the carbon particles, leading to the carbon stabilization in oil originating from steric interactions between the extended dispersants molecules [10,11]. Little discoveries were made on the carbon surface charge behavior induced by the polymeric dispersant, mainly due to limited work performed on the exploration of colloidal particle surface charging in oil. The low dielectric constant of non-polar solvents causes high energy barriers of charge separation between particles, shown in Eq. (1). The range of electrostatic interaction of two or more charged carbon surfaces can be quantified using the Bjerrum length  $\lambda_B$  equation, where  $e$  is the elementary charge,  $\epsilon_r$  is the relative permittivity,  $\epsilon_0$  is the permittivity in a vacuum,  $k_B$  ( $1.38 \times 10^{-23} \text{ m}^2 \text{ kg s}^{-2} \text{ K}^{-1}$ ) is the Boltzmann constant and  $T$  is absolute temperature [12].

$$\lambda_B = \frac{e^2}{4\pi\epsilon_0\epsilon_r k_B T} \quad (1)$$

Therefore, surface charging was considered to be an improbable mechanism for colloidal stability in non-polar media. However, with the addition of charge-bearing substances such as surfactants and electrolytes, surface charging and electrostatic stabilization was observed in non-

polar solvents [13,14]. Moreover, the change in oil viscosity alters the interparticle interactions between carbon; hence, compensating for the carbon stabilization process in oil. The electrokinetic behavior is affected by the transport effect caused by the changes in the fluid viscosity [15,16]. This phenomenon eventually determines the interparticle interactions and carbon particle stabilization in oil, leading to the ineffective addition of polymeric dispersants.

Nevertheless, the exploration of the mechanisms of carbon stabilization in oil is still limited, especially in electrostatic interactions. The effect of the surface charge on the stabilization of carbon particles is essentially disregarded, especially when ions dissociation in non-polar solvents is insignificant and hardly explored. The limited discovery on the effect of polymeric dispersant on the electrostatic interaction-induced carbon particles in oil could reverse the interaction, causing larger carbon aggregates to form. This work focuses on the charge stabilization of carbon particles in the oil, using carbon black influenced by the addition of polymeric dispersants. Carbon black was chosen as the soot surrogate due to the high carbon content in carbon black, which provides neat findings on their surface interactions in oil. The measurements performed are based on the dilute colloidal system, where the suspended particles are mobile in the continuum. The effect of solvent viscosities on the carbon interparticle interactions will be also evaluated, given that the oil viscosity is a crucial criterion in providing efficient carbon particle stabilization.

## METHODS AND MATERIALS

Carbon black (C) grade N330 was acquired from Sid Richardson Carbon Co. to simulate soot as a substitute for carbonaceous deposits. The average particle size and density of C N330 are shown in Table 1. Decane and hexane of 99% purity were purchased from Merck and used as carrying solvents for carbon particle suspension. The viscosity of decane and hexane used at  $T = 24 \text{ }^\circ\text{C}$  were 0.859 and 0.297 mPa s, respectively. Decane and hexane were used as the model carrying hydrocarbon solvents that are commonly found in engine oil. Mono-polyisobutylene succinimide (m-PIBSI or simply PIBSI) of molecular weight  $2187 \text{ g mol}^{-1}$  was used as an emulsifying dispersant in the oil phase and was acquired from Shenzhen Trass Lubrication

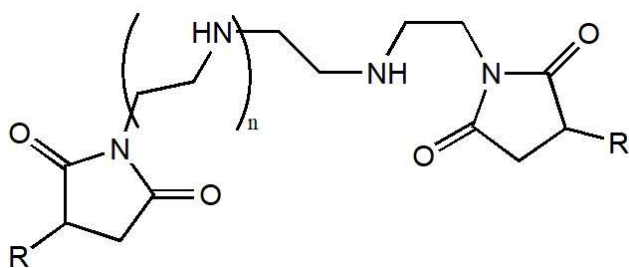


Fig. 1. Molecular Structure of PIBSI.

Table 1. Physical Properties of Carbon Black N330

Average particle size	$126 \pm 3.5$ nm
Density at 25 °C	$1.56 \pm 0.3$ g cm <sup>-3</sup>

New Material Co. Ltd. The molecular structure of PIBSI is presented in Fig. 1.

### Sample Preparation and Testing

The molecular weight of PIBSI was confirmed using the Gel Permeation Chromatography (GPC) Agilent 1260 in tetrahydrofuran at 35 °C at a flow rate of 0.6 ml min<sup>-1</sup>. The bound surface moisture was initially eliminated from the carbon black particles by drying in a conventional oven at 100 °C for 1 h. Then the dried carbon black particles were suspended in each decane and hexane separately at  $3 \times 10^{-3}$  wt.%. The fixed concentration of carbon particles was chosen to qualify the study of the interaction mechanisms within the dilute system regime. Prior to measurements, the solvents were filtered using 0.1 μm Advantec PTFE syringe filters to eliminate impurities and dust. The suspensions were then sonicated in an ultrasonic bath at 60 °C for 30 min. Next, PIBSI dispersant was added into the C-decane and C-hexane suspensions at varied concentrations of 0, 1, 3, 5, 7, 9, 11, 13 and 15 wt.%.

The electrophoretic mobility  $\mu_e$  measurement of the suspensions was performed using the Malvern Zetasizer Nano Z. The suspensions were transferred into a polished four-window glass cuvette before inserting the Malvern dip cell prior to measurements. The instrument was operated at a fixed voltage of 40 V using a 4 mW HeNe laser source of 633 nm wavelength. The surface charge  $Z$  of the carbon particles in decane and hexane was calculated using Eq. (2),

where  $\mu_e$  is the electrophoretic mobility of the carbon particles of a radius  $a$  in a solvent with a viscosity  $\eta$ . The effective surface charge  $Z_{eff}$  is calculated using Eq. (3) with  $\lambda_B$  is the Bjerrum length,

$$\mu_e = \frac{Ze}{6\pi\eta a} \quad (2)$$

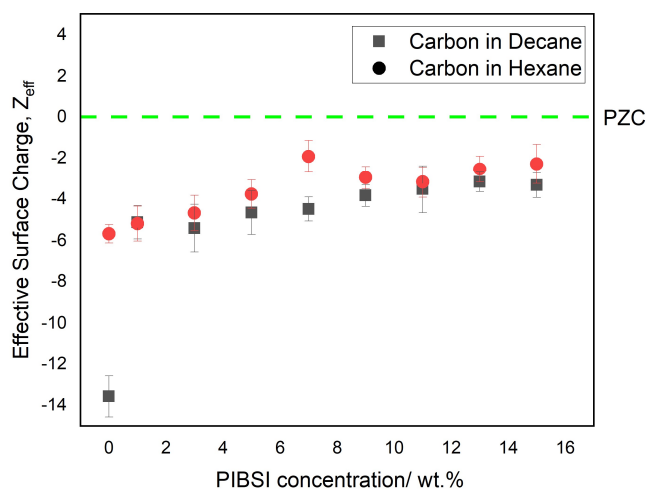
$$Z_{eff} = \frac{Z\lambda_B}{a} \quad (3)$$

The same instrument was utilized to measure the hydrodynamic diameter  $D_H$  of the carbon particles in decane and hexane. The fundamental of this measurement is based on the dynamic light scattering (DLS) technique, where the light source used operated at a wavelength of 532 nm and the incident light source was set up at 90°. The suspensions were diluted with respective solvents before the measurements. The aggregation behavior of the carbon particles in decane and hexane under the influence of PIBSI was evaluated using a UV Spectrophotometer Varian Cary 50 using a wavelength range from 190 nm to 600 nm.

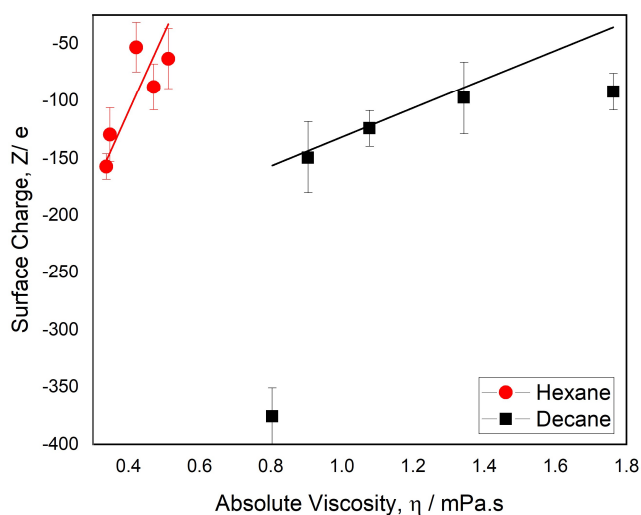
## RESULTS AND DISCUSSION

### Surface Charges Interactions between Carbon Particles with PIBSI in Decane and Hexane

Figure 2 shows the effect of PIBSI dosages on the  $Z_{eff}$  of carbon particles in decane and hexane. Generally, the magnitude of  $Z_{eff}$  of carbon particles in decane was greater compared to the ones in hexane. The absence of PIBSI in the system recorded the highest  $Z_{eff}$  value for both decane and hexane systems. At a high PIBSI concentration regime, the  $Z_{eff}$  values for both systems merged into similar surface charge magnitudes. The surface charge of carbon in decane was two times higher than in hexane without any dispersant added. This phenomenon indicates that the carbon particles formed a more stabilized dispersion in decane as opposed to that in hexane, mainly caused by the high viscosity of the solvent. The enhancement of system internal viscosity could also be achieved by the addition of polymeric additives that could further destabilize the particles, leading to the formation of non-homogenous system [20]. Figure 3 compares the dependency of the carbon surface charge on the viscosity of the dispersant. Both hexane and decane systems



**Fig. 2.** Comparison of effective surface charge of carbon particles in decane and hexane with varied PIBSI concentration.



**Fig. 3.** Comparison of surface charge of carbon particles in decane and hexane with respect to the dispersion viscosity at 25 °C.

showed a monotonic linear trend of surface charge, corresponding to the absolute viscosity of the dispersion. Hexane systems exhibited a steeper and more rapid increase in carbon surface charge, opposed to the decane systems. The high value of surface charge corresponds to an enhanced zeta potential value. The lowest viscosity regime of decane

system tends to produce a high magnitude of surface charge due to the surface ionization of carbon in the solvent. In a low viscosity solvent such as hexane used in this work, the suspended particles can be effortlessly mobile throughout the continuum, giving greater probability for surface interactions, eventually forming aggregates and agglomerations. Highly viscous solvent resists particle movement, thus suppressing interparticle interactions [17], while in low viscosity system, the particle surface charge is affected by the mobility of the particles.

However, looking at the dispersions with PIBSI dispersants, the overall surface charge and zeta potential values are lower in the hexane compared to that in decane, where the surface charges for PIBSI-laden hexane systems approached the point of zero charges (pzc). It implies that for dispersions with PIBSI, the colloidal surface charging is more probable and well-regulated in systems of high viscosities.

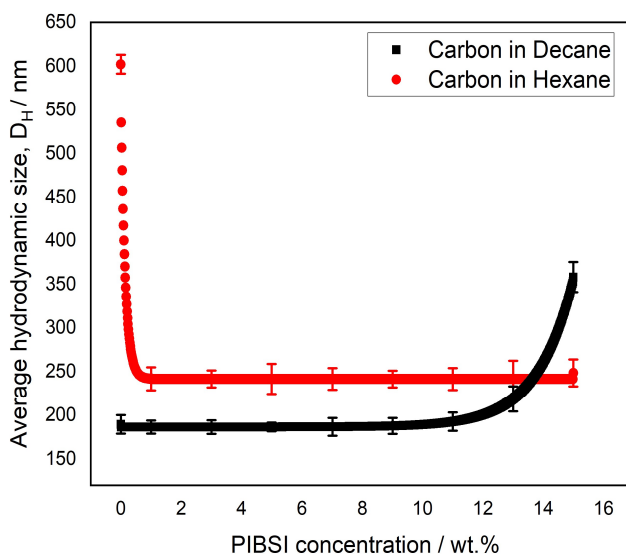
The strength of ionic entities in liquid can be quantified by the Bjerrum length, and this characteristic length scale depends on the dielectric constant of the solvent. Hexane and decane have a dielectric constant of 1.88 and 2, respectively. This indicates that the two-point of charges in decane interact at a slightly longer distance than the ones in hexane. In a non-aqueous system, the ionic distribution on particle surfaces and their surface potential can be described by the linearized Poisson-Boltzmann equation [18], where the ionic concentration across the distances of the surface decay exponentially. PIBSI molecules are amphiphiles, consisting of a polar head and a non-polar tail [19]. This structural arrangement is similar to ionic surfactants, where micelles formation is expected as the critical micelle concentration (CMC) is reached. In a work reported on anionic AOT surfactant in PMMA-laden oil dispersion, the AOT formed inverted micelles due to the absence of polar molecules in oil, resulting in the extension of a long hydrophobic tail in the oil phase and charged heads aggregating in the middle [20]. Roberts and coworkers (2008) reported that AOT formed charged micelles, and were eventually adsorbed on the colloidal particle surfaces, affecting the resulting particle charge [21]; Shafiq and coworkers (2019) also reported the same findings [22]. Similar behavior was obtained in the PIBSI system, where the adsorption of PIBSI molecules and micelles affected the carbon surface net charge.

### Effect of PIBSI Dispersant on Carbon Particle Aggregation Behavior in Oil

Figure 4 shows the average hydrodynamic diameter  $D_H$  of carbon particles at varied PIBSI concentrations in both decane and hexane. For most systems with PIBSI, the carbon  $D_H$  in decane were slightly lower than that in hexane, except at  $[\text{PIBSI}] > 11$  wt.%. Overall trends showed an exponential decay of average carbon  $D_H$  value for hexane systems. Previous work suggested that the primary soot particles size was below 100 nm [23], and well dispersed soot in oil had sizes range of 150 nm to 350 nm [24]. Without any PIBSI, the  $D_H$  of carbon particles in hexane was exceptionally larger (by the factor of 3) than the ones in decane. Solvents with low viscosity tend to promote a high collision rate between the particles through random Brownian motion, increasing the probability of particle surface interactions, eventually promoting carbon particles aggregation. This phenomenon is also known as perikinetic flocculation [25]. However, perikentic collision and agglomeration are widely observed and modelled in aqueous-based systems [26-28], where the particle size is very small and at a smaller length-scale of Kolmogorov length [29]. As opposed to the PIBSI-less decane system, the relatively small carbon  $D_H$  is predominantly related to the enhanced colloidal stability. Therefore, the solvent's viscosity is established to influence the collision efficiency and interparticle interactions between the colloidal particles, which can be further justified from the Stokes-Einstein Eq. (4) shown below [30]:

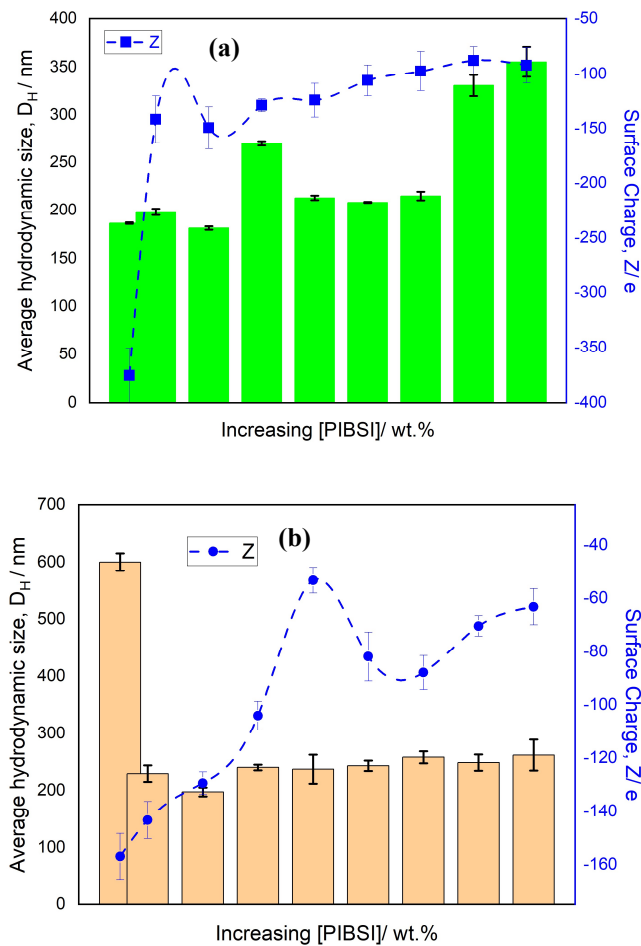
$$D = \frac{k_B T}{6\pi\eta a} \quad (4)$$

Where,  $D$  is the diffusion efficiency of the particle,  $k_B$  is the Boltzmann constant ( $1.38 \times 10^{-23} \text{ m}^2 \text{ kg s}^{-2} \text{ K}^{-1}$ ),  $T$  is the absolute temperature,  $a$  is the diameter of the particle, and  $\eta$  is the viscosity of the medium. As the viscosity of the solvent decreases, the diffusion efficiency increases, promoting the flocculation between the carbon particles. Interestingly, for  $[\text{PIBSI}] > 11$  wt.%, the average carbon particles diameter increases by approximately 40% in decane compared to that in hexane. At this concentration regime, the concentration ratio of PIBSI to carbon is scaled, giving rise to more PIBSI



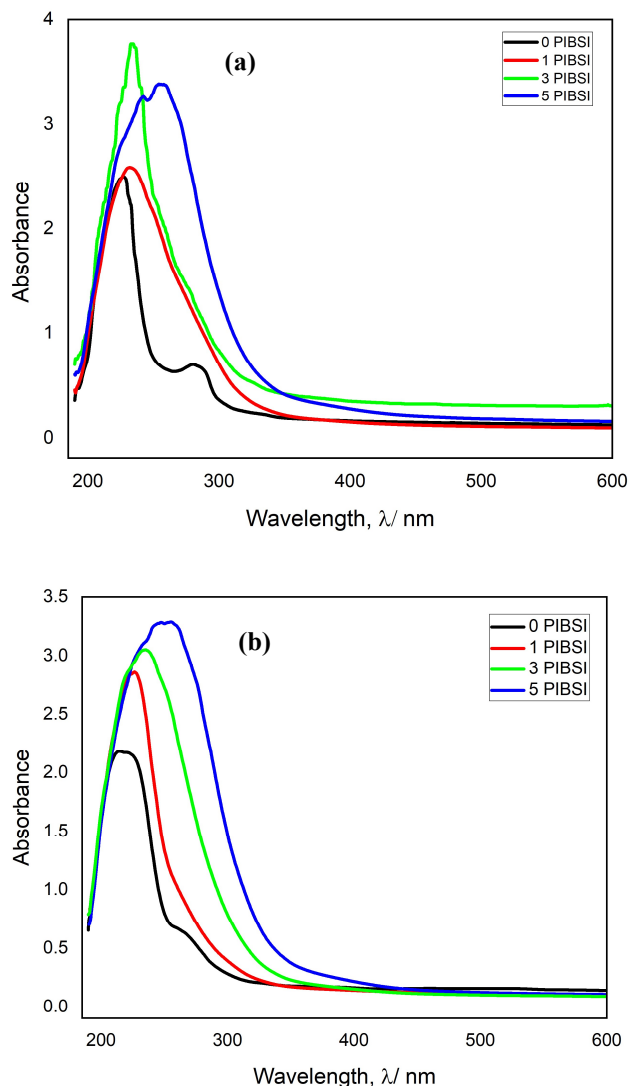
**Fig. 4.** Average hydrodynamic diameter average hydrodynamic diameter  $D_H$  of carbon particles at varied PIBSI concentration in decane and hexane.

adsorption on carbon surfaces, and eventually inducing carbon aggregation in decane. Figure 5 compares the trend of carbon hydrodynamic radius with the surface charge of carbon particles in hexane and decane. The decrease in the magnitude of surface charge indicates the formation of large average carbon size in decane. A more erratic trend of carbon particles surface charging in hexane, and the rapid reduction of particle size with increasing PIBSI concentration were obtained. Previous work suggested that the switchover is related to the basis of PIBSI-carbon interactions in the solvent [31]. At the excess amount of PIBSI, the free and non-adsorbing macromolecules from polyisobutylene chains portion of PIBSI in the colloidal dispersion induced an effective attraction between the carbon particles; this is in agreement with the depletion interaction model of macromolecules-induced interactions between particles proposed by Asakura and Oosawa (1958). Recent findings showed that the free segments of polyisobutylene chains (tails) of the adsorbed PIBSI on carbon surfaces attached onto the other carbon particles in a solvent, leading to the bridging agglomeration, showing a good agreement with the work done by Seo and Paik (2016) [32].



**Fig. 5.** Comparison between surface charge  $Z$  with the average hydrodynamic diameter  $D_H$  of carbon particles at varied PIBSI concentration in decane (a) and hexane (b).

The UV-Vis spectra of the carbon particles in hexane and decane with increasing PIBSI concentration are shown in Fig. 6. Based on the spectra the carbon particles display characteristic peaks between 190-260 nm. It was reported by Jäger et al. (1999) that the  $\pi$ - $\pi^*$  transitions appeared in the range of 180-260 nm in all the carbonaceous materials, which were related to the electronic transitions between the bonding and anti-bonding  $\pi$  orbital [33]. The UV-Vis absorbance peaks values recorded at around 230 nm and 260 nm were taken into consideration, directly related to the corresponding amount of the carbon particles dispersed in the solvents. Increasing the PIBSI concentration, the absorption spectra increased from 0 to 3 wt.%, then declined at 5 wt.% for both



**Fig. 6.** UV-Vis absorption spectra of carbon particles in decane (a) and hexane (b) at different PIBSI concentrations

solvents. At both 0 and 3 wt.% of PIBSI, the UV-Vis absorption peaks in decane were higher than those in hexane. In contrast, at 5 wt.% of PIBSI, the UV-Vis absorption peak of carbon particles in hexane was slightly higher than in decane within the 190-260 nm range. Both the UV-Vis spectroscopy and DLS measurements indicated that the  $D_H$  for the carbon particles was smaller for the decane system compared to hexane at both 0 and 3 wt.% of PIBSI. For a viscous system with 5 wt.% of PIBSI, the attractive colloidal interaction was promoted by the excess PIBSI dispersants relative to a system with low solvent viscosity. The increase

in absorption could be attributed to the increase of the total surface area of carbon particles exposed to the radiation with increased dispersion stability.

## CONCLUSIONS

The interactions between carbon particles in solvents with different viscosities were evaluated in this work. The novelty of this work lies in the discovery of the spectrum of colloidal interactions, mainly the electrostatic interaction between carbon particles. The latter interaction is predominantly affected by the solvent viscosity and PIBSI dispersant concentration, which in turn gives different types of surface interactions between carbon particles. Previous works performed on the carbon-PIBSI in oil systems were motivated by the polymer-particle interactions such as steric interactions. These fundamental findings are beneficial for the oil and lubricants formulators to improve the dispersion technology. From the electrophoretic mobility measurements and analysis, it was found that the PIBSI dispersant transferred charge more effectively in low viscosity solvent such as hexane, giving rise to high surface charge value for particles in hexane. The surface charge development in a more viscous solvent such as decane was slower. The DLS and UV-Vis absorption measurements also supported that the inter-carbon interactions were more dominant in low viscosity solvent since the overall hydrodynamic diameters were larger than those in decane, due to the high particle diffusivity in lower viscosity solvent. This indicate that the interparticle interactions (electrostatic, in this work) of carbon particles are compensated by the viscosity of the carrying oil. The evaluation of dispersant-induced carbon interactions can be applied as a fundamental knowledge for engine oil formulators to engineer an effective oil system that inhibits the formation of soot.

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