Journal of Molecular Liquids 221 (2016) 292–297

Contents lists available at ScienceDirect

Journal of Molecular Liquids

journal homepage: www.elsevier.com/locate/molliq

Comparative studies on Graphite and Carbon Black powders, and their dispersions

Alok P.S. Chauhan *, Komal Chawla

Department of Physics and Materials Science and Engineering, Jaypee Institute of Information Technology, A-10, Sector-62, Noida, U.P. 201307, INDIA

ARTICLE INFO

Article history:
Received 6 May 2015
Received in revised form 6 May 2016
Accepted 16 May 2016
Available online xxxx

Keywords:
Suspensions
Spectroscopy
Carbon
Particle size analysis
Scanning Electron Microscope

ABSTRACT

The colloidal dispersions of Carbon Black (CB) and Graphite powders were prepared in dimethylformamide (DMF) and dimethylsulphoxide (DMSO) as solvent using a facile method. These dispersions were characterized using Ultraviolet-Visible (UV–Vis) and Fourier transform infrared (FTIR) spectroscopy. The Tauc's relation in UV–Vis data affirmed high band gap of 3.32 eV for Graphite suspension in DMSO solvent (GSO) and low band gap of 1.96 eV for CB in DMSO solvent (CBSO). This is due to minimum absorbance of GSO in UV–Vis spectra and inverse relationship of particle size with band gap. The Fourier transform infrared (FTIR) spectroscopy facilitated the study of interaction between solvents (DMSO, DMF) and carbon powders (Graphite, CB). The CB powders showed wider particle size distribution than Graphite powders in Scanning Electron Microscope (SEM) micrographs and even confirmed by span factor calculation. And, X-ray diffraction (XRD) results endorsed amorphous nature of CB powder having short range order in contrast to Graphite powder. Thus, particle size and amorphous nature could be linked with the higher stability of dispersions of CB than that of the Graphite.

1. Introduction

Carbon is having one of the unique characteristic to exist in a number of allotropic forms, with remarkably varying chemical and physical properties due to the diverse nature of chemical bonds and arrangement of carbon atoms in 3D (diamond), 2D m(Graphite), and 1D (carbine) [1,2]. CB[1] is elemental carbon in the form of very fine particles possessing an amorphous molecular framework. It is produced by the process of incomplete combustion of hydrocarbons such as feedstock oil, coal tar, etc. CB is extensively used in the form of filler in plastics, paints and elastomers [3]. It enhances the electronic, mechanical, and optical characteristics of the material in which they are used and thus determine their application in different fields [4]. CB enhances UV protection, electronic conductance, and field of darkness (jetness), opacity, and reinforcement when it blends with plastics, paints and elastomers [5,6]. The diverse nature of surface and dispersion properties of different grades of CB has immense impact on their realization in xerographic toners [7]. In addition to this, CB is used in the tire industry as reinforcement filler [8] as well as pigment for printing inks, plastics and coatings [9,10]. CB is known to possess high refractive index ($n = 2.26$) with good chemical and thermal stability [11]. Graphite, in spite of being a non-metal, is another allotropic form of carbon which is an efficient conductor of electricity. It has diverse applications ranging from everyday purpose such as lead pencils to big industrial uses like steel making due to its versatile properties [12].

The powder dispersion of carbon powders in aqueous media is of immense significance for the preparation of good quality coatings and inks [5]. The high specific surface area, hydrophobic character and oil absorption ability of CB powders affect preparation of stable dispersions; facilitated by adsorption of solvent on solid surface assisting colloidal stability [13–16]. This augmentation of suitable polymeric dispersing agent aids in special applications, where the nature and stability of dispersion would be required to obtain homogeneous colloidal dispersions [17]. These augmented stable polymeric dispersions have applications in pigments of ink-jet printers [18,19]. Generally, the surfactants used for preparing stable dispersions of carbon powder particles are poly-dimethylsiloxane [20], Orotan® - a polyacrylate [21], sodium dodecylbenzenesulfonate, DMF[2] [22], sodium dodecyl sulphate [23–25]. Generally, CB as well as Graphite easily agglomerate in hydrophilic solvents and liquid media not having right range of cohesive energies, due to their inherent hydrophobic nature. Therefore,

Abbreviations: CB, Carbon Black; DMF, dimethylformamide; CNT, carbon nanotube; DMSO, dimethylsulphoxide; XRD, X-ray diffraction; UV–Vis, ultraviolet–visible; FTIR, Fourier transform infrared spectroscopy; SEM, Scanning Electron Microscope; CBMF, Carbon Black suspension in DMF solvent; GMF, Graphite suspension in DMF solvent; CBSO, Carbon Black suspension in DMSO solvent; GSO, Graphite suspension in DMSO solvent; PSD, particle size distribution; RSF, Relative Span Factor.

* Corresponding author.
E-mail addresses: chauhan.alok@gmail.com, alok.chauhan@alumni.stonybrook.edu (A.P.S. Chauhan).

[1] CB Carbon Black.

[2] DMF dimethylformamide.

http://dx.doi.org/10.1016/j.molliq.2016.05.043
0167-7322/

production of homogeneous colloidal suspensions of electrically conductive CB and Graphite is achieved by using DMF and DMSO as dispersing agents. These solvents (DMF and DMSO) have high dispersion component due to their high electron pair donicity, high solvochromic parameter and negligible hydrogen bond donation parameter [26,27]. The synthesis and analysis of CNT[3] dispersion in polymer matrix had been reported earlier by numerous techniques. In the case of commonly used stirring, rate of dispersion was controlled by speed of mixing as well as size and shape of the propeller. Therefore, rigorous stirring of CNTs in polymer matrix give fine dispersion of CNT [28–30]. As there has been substantial studies on the degree of dispersion of CNTs and Graphene [25,31–36] but limited investigation on the dispersion behaviour of CB [17,20] and Graphite [37–39] in different solvents, the present research work focuses on the latter using two solvents. Here, carbon (mainly, CB and Graphite) dispersions in two different solvents (DMF and DMSO[4]) were examined for particle size distribution and structural information using Laser Particle Size Analyzer and XRD,[5] respectively. Further, the colloidal dispersions of carbon particles were analyzed by UV–Vis[6] spectroscopy to determine the direct band gap of the powders in the different solvents. The intermolecular interaction between the solvents and the carbon powder was also studied by FTIR[7] analysis.

2. Experimental

2.1. Materials

Laboratory grade fine Graphite powder (99.5% pure) and industrial grade CB powder (N-375) were obtained from Central Drug House CDH, India and Continental Carbon India Limited, respectively. DMF [$(CH_3)_2NC(O)H$] and DMSO [$(CH_3)_2SO$] solvents were sourced from Sisco Research Laboratories SRL, India with purity 99.8%, water content 0.05% and 0.03%, respectively.

2.2. Preparation of Graphite and CB suspensions in DMSO and DMF solvents

DMSO and DMF solvents belong to polar aprotic class of colorless organic solvents and their functions are invariably identical in various applications. DMSO is more nucleophilic than DMF, and it can dissolve most organic and inorganic salts. The boiling point of both the solvents is very high; but freezing point of DMF at −60 °C gives it a preference over DMSO at lower temperatures. The dispersion formulation of Graphite and CB in two dissimilar solvents (DMSO and DMF) was implemented by a simple one step process without any sonication [22,28]. The various suspensions formulated were: Graphite in DMSO and DMF, CB in DMSO and DMF. The concentration of carbon powder additives was taken to be 7% weight by volume to disperse in solvents. The segregation of suspensions of Graphite and CB powders in both solvents was prevented by magnetically stirring for more than 5 h.

2.3. Characterization

The particle size analysis of Graphite and CB powders was done using *Horiba Laser Scattering Particle Size Distribution Analyzer Partica LA-950*; whereas structural information was observed microscopically employing *TESCAN MIRA3 LM Field Emission SEM*.[8] The characteristic XRD patterns of these powders were recorded using an *Xpert' PRO* XRD spectrometer, equipped with Cu anode X-ray tube (with K_α radiation of 1.54 Å), a flat crystal monochromator, and a proportional detector consisting of a cylindrical chamber filled with xenon/methane gas mixture. UV–Vis optical studies of the prepared samples of CBMF,[9] GMF,[10] CBSO[11] and GSO[12] were investigated using *Lambda 35 Double Beam UV–Vis Spectrophotometer*, manufactured by Perkin Elmer, Germany. The FTIR observations were performed using *Spectrum BX-II Spectrophotometer*, made by PerkinElmer, Germany.

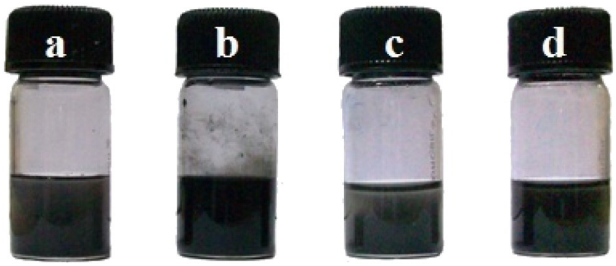

Fig. 1. The image shows colloidal dispersions for (a) GSO, (b) CBSO, (c) GMF and (d) CBMF.

3. Results and discussion

The dispersibility of the CB and Graphite powders is examined in DMF and DMSO solvents. Fig. 1 depicts the optical image of Graphite and CB powder suspensions in DMSO and DMF solvents. As can be noticed from the photograph, CB suspensions have less transmission as compared to Graphite suspensions. Further, it can be easily deduced that CB suspensions in both the DMSO and DMF solvents are stable (without formation of any precipitate/flocculate) and homogeneous as compared to Graphite suspensions. The stability of the dispersions can be easily associated with the particle size of the used powders as larger particle size powders depicted improved stability.

3.1. Particle size analysis

Particle size is one of the vital physical characteristic of powders useful in engineering applications as well as manufacturing of engineering products. Mainly the material properties such as reactivity or dissolvent rate, stability in suspensions, appearance, flowability, viscosity, packing compactness and absorbency are directly affected by the particle size of the ceramic powders [40,41]. The fundamental idea in laser diffraction is that the particle will scatter light of intensity proportional to its size. The scattering of light takes place at wide angles for small particle size compared to small angles for large particle size. A pattern of scattered light is obtained from the assortment of particles defined by the intensity and angle which are plotted into particle size distribution results [42].

Particle size distribution of Graphite and CB is assessed using laser diffraction by applying wet analysis method. In this technique, the powdered particles are suspended in liquid dispersant (deionized water in our case). Here, wetting of the particle surfaces with the dispersant reduces the surface energy which in turn decreases the force of attraction between the neighbouring particles. This allows them to be disjointed and be suspended in the dispersant. The correct dispersion is achieved by providing appropriate external energy to the system. This is accomplished by thorough agitation or stirring; whereas for very fine powders or, strongly bound agglomerates, ultrasonic irradiation is preferred. The powdered samples are first dispersed in deionized water and sonicated using ultrasonic bath for half an hour. After that they are injected in to measuring cylinder. The volumetric PSD[13] plots are drawn from the intensity profile of the scattered light using Laser Scattering Particle Size Distribution Analyzer Partica LA-950 software which is based on Mie

[3] CNT carbon nanotube.
[4] DMSO dimethylsulphoxide.
[5] XRD X-ray diffraction.
[6] UV–Vis ultraviolet–visible.
[7] FTIR Fourier transform infrared spectroscopy.
[8] SEM Scanning Electron Microscope.
[9] CBMF Carbon Black suspension in DMF solvent.
[10] GMF Graphite suspension in DMF solvent.
[11] CBSO Carbon Black suspension in DMSO solvent.
[12] GSO Graphite suspension in DMSO solvent.
[13] PSD particle size distribution.