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Research paper

Assessment of particle size distribution and tensile properties of hybrid epoxy composite reinforced with functionalized graphene and CNT nanofillers

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Abstract

The synergetic effect of amine functionalized multiple graphene layers (AMGL) and multi-walled carbon nanotube (AMWCNT) nano-fillers mixed with epoxy resin is investigated on the particle size distribution (PSD) and tensile properties of hybrid composites. The hybrid composites with an equal ratio of nano-fillers at a weight percent of 0.25, 0.50, 1, and 2 wt % are fabricated. The particle size analysis (PSA) is performed by the dynamic light scattering (DLS) technique and image analysis (IA) method; both verify PSD for composites. This is further verified by the analyses of scanning electron microscopy (SEM) images using Image J software. The optimum composite particle size of 6.8 µm and homogeneous mixture with a poly-dispersity index (PI) of 0.74 is investigated for a sample having filler content of 0.5 wt %. Tensile stress and elastic modulus is also found to be maximum at 0.5 wt %, which is 49.91 MPa of 2302 Mpa, respectively. The chemical composition of composite affecting its PSD is characterized by energy dispersive X-ray (EDX) process. Dimensional analysis of particle size in the domain of epoxy matrix provides deep insights to the researchers and may also provide them a direction for selecting an appropriate material for a particular application.

1. Introduction

Graphene and carbon nanotube (CNT) have drawn great attention for fabricating epoxy-based hybrid composites [1]. Graphene has a 2D structure with one atom thick planar sheet of a hexagonal array of sp^2 hybridized carbon atoms, while CNT has a tubal structure with a specific length to diameter ratio [2-3]. Though these

nano-fillers possess extraordinary mechanical, thermal, and interfacial properties, the problem of dispersion and agglomeration still needs more attention [3-5]. This problem of fillers distribution may be addressed by performing their surface treatment, such as functionalization [6-7]. Furthermore, researchers observed that the functionalization of graphene and CNT can enhance its dispersion within epoxy [8-10]. Characterization of hybrid epoxy composites reinforced with surface treated fillers has been performed, confirming the improvement in mechanical properties of composite [9, 11-14]. Tensile properties are improved with amine functionalized multiple graphene layers (AMGL) and amine functionalized multi-walled carbon nanotube (AMWCNT) reinforced in epoxy resin [10, 15-16]. This reveals the synergetic effect of AMGL and AMWCNT on the overall properties of hybrid composites.

Particle size distribution (PSD) using particle size analyzer (PSA) may be helpful in redacting the problem of agglomeration and also give the direction towards pinpoint applications of these hybrid composites. Hence, the relation between PSD and the weight percentage of nano-fillers requires to be investigated. Determining the optimal processing method for obtaining a homogeneous hybrid composite comprising uniformly dispersed nanoparticles is crucial in material science which can be addressed by PSD analysis [17]. The importance of size characterization is an advent with the fact that it can help in selecting proper processing methods, and reduce time in size determination of nanoparticles. This provides strength for carrying out PSD analysis.

Particle size affects the mechanical performance of composite material [18-21]. The composite fabrication process affects the particle size of hybrid composite [22]. As the particle size decreases both tensile strength and ductility increase due to the reduced ligament size and particle fracturing, accelerated wear is observed in the hybrid composite samples reinforced with coarse particles, showing a fairly uniform distribution of particles. A decrease in the size of reinforcement increases the strength by 50 % [23]. However, increasing particle size after a certain critical limit does not affect the hybrid composite properties [24].

As far as the present literature survey, there is an acute shortage of studies made for PSD of graphene-MWCNT epoxy hybrid composite [24]. Though the effect of particle size on mechanical properties of epoxy hybrid composite is investigated and results indicate that mechanical properties depend upon the individual particle size of constituents of composite [25].

There are different methods for performing PSD analysis, such as dynamic light scattering [26-29], image analysis, centrifugal sedimentation analysis [30], and static light scattering [31]. Depending upon the specific parameter of particle size (like equivalent spherical diameter, length, intensity, width) to be analyzed, the appropriate method of PSD analysis is considered. In the present research work, the dynamic light scattering (DLS) method is selected for analyzing PSD since the hybrid composite structure consists of constituents, which are in nano-size and DLS is well established standard method used for the measurement of the nano-size particle [32]. Further to investigate the length, width, and equivalent spherical diameter, PSD is analyzed by image analyzer (IA) using Image-J software. Results obtained from PSD and IA are compared with each other.

Mean size of dispersed particles in hybrid composite, poly-dispersity index (PI), and pattern of the PSD can be obtained by DLS method (ISO 22412: 2008, ISO 22412-2008a, and ISO 13321: 1996) [33]. Analysis of particle size by DLS method is done by assuming that particles are spherical in shape, however in actual the particles are of irregular shape and size. Along with PSA, it is also a powerful tool to analyze the quality of dispersion, and particle agglomerates present in the hybrid composite [17]. Morphological analysis of particle size and chemical analysis can be studied by SEM [17], DLS [34], and EDS [35] methods, respectively. The particle size of the composite can be computed using PSD and PSA methods [35]. Particle sizes affect the tensile properties of hybrid composite material [23, 36-37].

The irregular shape and size of particles can be analyzed by image analyzer [38]. Particle size stability was investigated and calculated from the DLS, CLS, and ELS data [33]. DLS is based on the methodology for determination of hydrodynamic mean size and nature of the dispersion of the particles [26-27, 39]. Results obtained from different PSD analyses can be compared basised on different parameters like DLS is used to find out spherically of particles in the liquid matrix, whereas SEM gives a qualitative impression of the final particle size in cured hybrid composite [30, 40] The results of SEM image analysis depends on certain major factors such as the quality of samples, SEM image, operating parameters set in image analysis software and investigation of obtained results [17]. PSA using SEM images has been done by using Image J software [41]. Variation in PSD is further due to various elements which can be analyzed by chemical composition analysis, through which both qualitative and quantitative analysis is done to determine the concentration of elements present [35]. Therefore, it is advent from the above literature review that the reinforcement of nanofillers [42-43] and variation of filler content [44] along with its particle size affects the properties of epoxy composite materials.

Though a number of studies have already been done on the calculation of PSD of nano-fillers, synergetic effects of both functionalized nano-fillers are still to be discussed in deep. In general particle size of nano-fillers after applying all processing techniques can be approximated by measuring the equivalent diameter of AFML and AMWCNT by treating it as a sphere. Variation in PSD and tensile properties of hybrid composites due to the synergetic effect of AFML and AMWCNT fillers is explored using SEM images and the DLS method. Thus, size determination of composite particle and dispersion of filler in epoxy is analyzed. Chemical characterization using EDX for different hybrid composites is also done, which certainly helps in investigating the effect of different chemical group evolutes during the chemical functionalization and processing of hybrid composites. Hence, this study provides a direction for finding out the appropriate application of such hybrid composites, which obviously depends on the PSD of nano-fillers used.

2. Experimental setup

2.1. Materials

Two nanosized particles amine functionalized multi-graphene layer (AMGL) and amine functionalized multi-walled carbon nanotube (AMWCNT) is supplied by United Nanotech Innovations, Bangalore (India). The resin used in this study is epoxy (Diglycidyl Ether Bisphenol A (DGEBA)) commercially available LY556, and the hardener used is tri ethylene tetra amine (TETA, HY-951) obtained from Sakshi Dying and Chemicals, Delhi (India).

In the present experimental research work, graphene and MWCNT filler content are maintained at 1:1. The weight percentage of filler used is 0.25, 0.50, 1.0, and 2 %. The effect of filler quantity variation on the resulting particle size of hybrid composite is evaluated. PSD analysis of the hybrid composite obtained is performed. Elemental constituents of both the fillers are given in Table 1.

2.2. Material processing and specimen fabrication

Sample processing is performed by the sonication method. The amount of weighted epoxy resin is put in a suitable beaker. To reduce viscosity, it is mixed with ethanol and ultrasonicated. To avoid temperature rise and temperature variation during the sonication, the beaker containing mixture is submerged in water. After this, the mixture is mechanically stirred for 10 to 15 min. The obtained solution is mixed with pre-weighted filler.

Table 1. Chemical composition of AMGL and AMWCNT (Ref: United nanotech Innovations, Bangalore, India).

Element / Filler	Carbon %	Chlorine %	Oxygen %	Hydrogen %	Nitrogen %	Traces %	Ash %
AMGL	72	0.8	0.2	17.6	8.8	0.5	0.1
AMWCNT	72	1	0.2	16.2	8	1.5	0.1

The rigorous mixing of these ingredients produces highly reactive volatile vapor bubbles. For removing these vapor bubbles, high vacuum degassing is applied for 30 min. After the bubbles are removed, the mixture is transferred to molds which are put cured for 24 h in the oven. The obtained samples are machined and trimmed for employing mechanical testing and characterization. Samples are further cured for 4 h at 100ºC. Configuration of the sample depending on weight percentage of different constituents is given in Table 2.

2.3. Characterization of hybrid composite

PSA of different samples is done using a DLS analyzer. PSD and PSA are obtained through DLS Zeta potential instrument (Beckman Coulter Delso Nano C instrument) at room temperature. The scattered light is collected by the probe to determine particle size. During DLS testing operating parameters like temperature (25^0C), diluents (ethyl alcohol), refractive index (1.3611), and viscosity (1.1015 cP) are being prevailed, which are being kept constant, while scattering intensity, which provides the information about uneven particle shape and size, is being varied with volume of the particles. Cumulant method is adopted for data analysis. DLS instrument can determine PSD and particle zeta potential (electrical charge of the particle).
For surface morphology of tested (tensile) samples, SEM images are captured using a high-resolution scanning electron microscopy (HRSEM) in Zeiss-ULTRA plus SEM instrument for investigation of fractured surfaces. In this study, the accelerating voltage is set at 0.9 to 1.5 kV (for providing a conductive environment in and around the sample), and the surface charge of SEM image is 20 kV.

Table 2. Sample code of hybrid composites.

Sample code	Epoxy resin (wt%)	Carbon nano-filler (wt%) (AMGL+ AMWCNT)	Carbon nano-filler composition (wt%)	
			AMGL	AMWCNT
A	100	0	0	0
B	99.75	0.25	0.125	0.125
C	99.50	0.50	0.25	0.25
D	99	1	0.50	0.50
E	98	2	1	1

An optimal combination of brightness and contrast is adjusted such that clear microscopic observation of samples containing nanoparticles can be obtained.
PSA is performed on specimens of each sample code using Image J, and particles are investigated for shape detection and calculation of mean area equivalent diameter. The original image size has 1024 × 768 resolutions in terms of pixels, the magnification scale of the image is 10 µm and other operating parameters prevailing during image analysis are given in Table 3.
Computation of elastic properties of hybrid composites is done through the tensile test. A series of tensile tests have been performed on Instron-H25KS machine as per ASTM D 638 standard. Dimensions of samples for the tensile test are 57× 14 × 5 mm. Furthermore, the samples are tested by keeping operating parameters such as a clamp length of 220 mm with a displacement rate of 5 mm/min. These aforementioned tests are conducted on every five specimens of specific composite code. Finally, the averages of these values obtained are being considered, and also the standard deviation for tensile tests has been calculated. Operating parameters prevailing during the experiment were: clamp length: 220 mm, Displacement rate of 5 mm/min. at room temperature of 30ºC. Tensile test are performed on five specimens and the result is calculated for the average of five measurements. Energy dispersive X-ray spectroscopy (EDX) is performed for analyzing elements present in a hybrid composite sample. EDX is an analytical characterization technique performed to find out the distribution of elements in a hybrid composite sample.

Table 3. Operating parameters for image analysis

Sample code	Equivalent distance (pixels)	Scale, (pixels/ µm)	Calibrated image size (pixels)	Lower / Upper threshold
A	34.25	3.4	303564 × 205247	122/ 255
B	17	1.7	602352 × 451764	91/ 168
C	50.5	5.0	202772 × 152079	145/ 255
D	68	6.8	150588 × 112941	101/ 255
E	58.75	5.9	174297 × 130723	127/ 255

It helps in finding the elements present in the hybrid composite. An energy dispersive spectrum is obtained comprising X-ray beam energy (0 to 10 keV) on the x-axis and the number of counts per second (cps) on y-axis. Usually, the spectrum is set at a point in the SEM image, at this particular point, chemical composition is being analytically obtained.

3. Results and discussion

3.1. DLS method for particle size distribution analysis

The results of the DLS test, performed on AMLG/ AMWCNT hybrid epoxy samples are shown in Table 4. Composite with a filler content of 1 wt% shows the maximum size of its constituent particles, whereas the minimum size of its constituent particles is observed for composite for filler content of 0.5 wt%. The reason for this observation can be attributed to the extraction of graphene layers and tubular structure of MWCNT. This leads to improvement in filler dispersion in the composite. Moreover, the particle diameter is inversely proportional to the diffusion coefficient, as indicated in Eq. 1, which is justified by the highest value of diffusion coefficient in the case of sample C. Lowest value of PI is obtained for sample C with 0.714, indicating the best dispersed filler in composite mixture due to reduction in agglomeration.

$$\text{Diffusion constant:} \quad \mathbf{D} = \frac{\boldsymbol{\kappa T}}{\mathbf{6\pi\eta R}} \qquad (1)$$

where, D = Diffusion constant, m^2/s
R = radius of particle, m
$\boldsymbol{\kappa}$ = Boltzmann constant, m^2kg/Ks^2
T = Temperature, K
η = shear viscosity of solvent or medium, Pa.s

Table 4. Cumulated results obtained using DLS.

Sample code	Scattering intensity, (cps)	Cumulant diameter, (nm)	PDI	Diffusion constant, ($\times 10^{-9}$ cm^2/s)
A	11117	1072.3	0.523	3.697
B	8720	3277.4	0.878	1.210
C	4153	1577.6	0.714	2.513
D	7972	3439.4	0.918	1.153
E	2851	2098.2	0.955	1.890

Table 5. Particle size contributing 10%, 50%, and 90% of the PSD.

	Diameter, d µm		
Volume %	10	50	90
Sample code			
A	0.18	0.23	23.85
B	1.17	24.12	38.16
C	0.14	6.81	13.83
D	1.18	28.83	44.96
E	0.21	29.99	49.45

Fig. 1 shows the PSD of different composite samples measured by DLS considering 10, 50, and 90% volumes. Sample C is preformed optimally amongst all hybrid composites with a particle diameter of 6.80 µm, which physically shows the least cluster formation. However, the highest mean diameter of particles is experienced in sample E with 29.98 µm due to agglomeration of the particles, which decreases the tensile properties of hybrid composites. Volume contributions other than 50%, illustrated in Table 5, are being considered only for comparison purposes because highly disruptive values have been observed on above-mentioned volume contributions, which may also be justified from Fig. 1.

3.2. PSD analysis of SEM image of tested samples

SEM images of hybrid composite samples are shown in Fig. 2. Same images of tested samples are further processed for PSA. SEM images for fractured surfaces with filler contents of 0.25, 0.5, and 1 wt% are shown in Fig. 2.

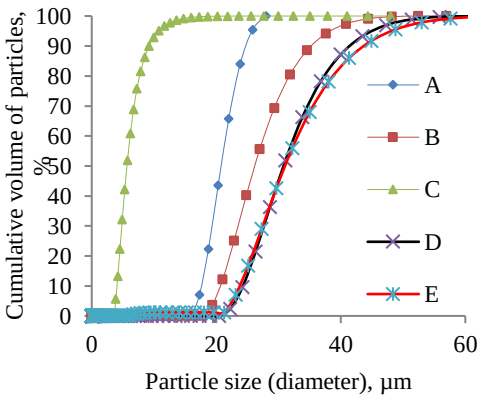

Fig. 1. PSD of hybrid epoxy composite.

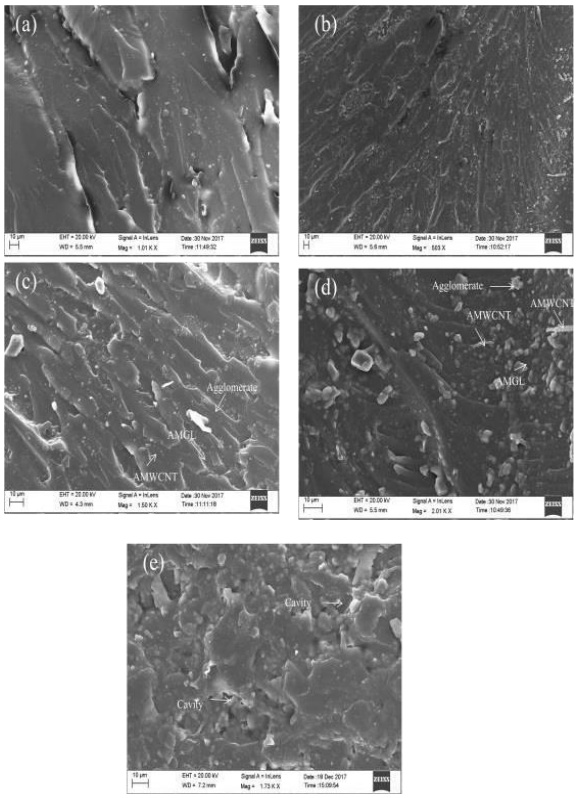

Fig. 2. SEM images of AMGL/AMWCNT hybrid epoxy composite with nano-filler; (a) 0, (b) 0.25, (c) 0.50, (d) 1, and (e) 2 (wt%).

The best composite structure in terms of dispersion of fillers is observed in sample D, which is verified by SEM images and image analysis (IA) software.

Fractography of composite, as shown in Fig. 2, exhibits rough surfaces, which signifies that crack initiation is resisted to propagate; this may be due to the deep rooted nano-fillers in the epoxy matrix and also the enhanced resistance offered by epoxy to the nano-fillers when being pulled out from the surface. This may also be justified through the minimal point and edge dislocation for sample D, when it experiences a series of tensile tests. However, beyond 1 wt% of both the filler contents, cluster formation in the form of irregular patterns is observed, which deficit the efficacy of hybrid composite, this may also be justified through the cavity formation as seen in Fig. 2(e) because of inadequate dispersion, results in poor load transfer capability of nano-fillers to the epoxy matrix.

PSD analysis by SEM image is done to compare results obtained by the DLS method. Processing of hybrid composite SEM images is performed in Image J software. To increase the comparability to SEM, particle sizes are determined according to the maximum ferret diameter. Various steps carried out in Image J software for finally making SEM image ready for PSA are enumerated in Fig. 3.

Image analysis results are shown in Fig. 4. Color threshold, along with the binary method, is enabled in order to increase image accuracy for particle size computation. Results obtained in terms of the processed figure are shown in Fig. 4. Output from Image J delivers an image indicating particle size shown within a red circle, through which the cumulated particle size distribution can be calculated. Locations of agglomeration are indicated by continuous black portions present in Fig. 4 (a-2, b-2, c-2, d-2, and e-2).

A comparative of PSD for hybrid composite obtained after image analysis is shown in Fig.5.

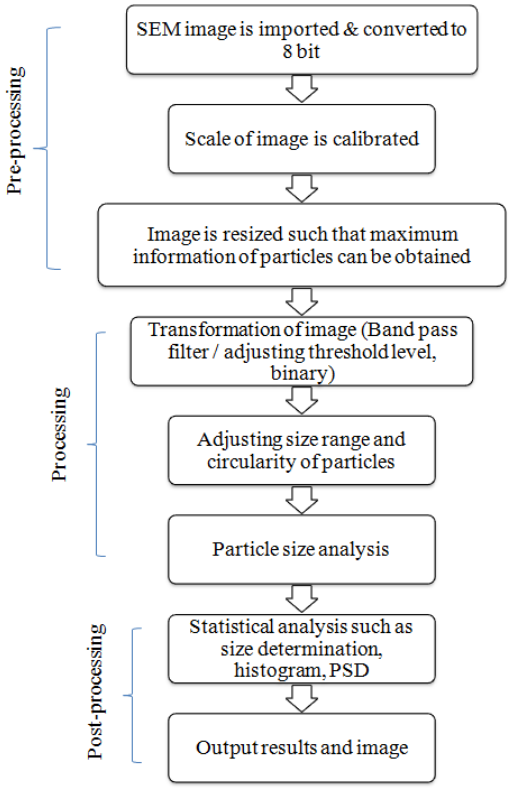

Fig. 3. Flow process for PSD used in Image J software.

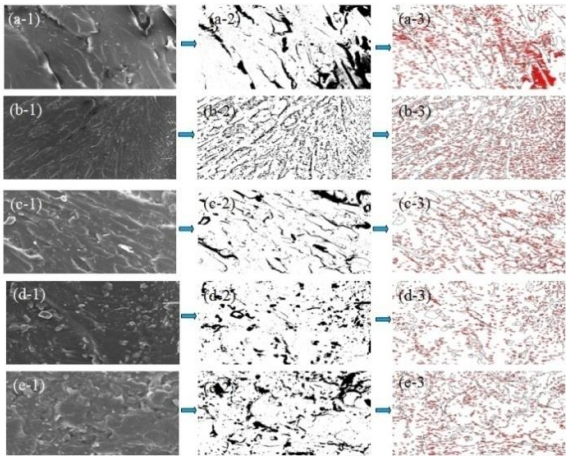

Fig. 4. Image analysis of AMGL/AMWCNT hybrid epoxy composite nano-filler; (a) 0, (b) 0.25, (c) 0.50, (d) 1, and (e) 2 (wt%).

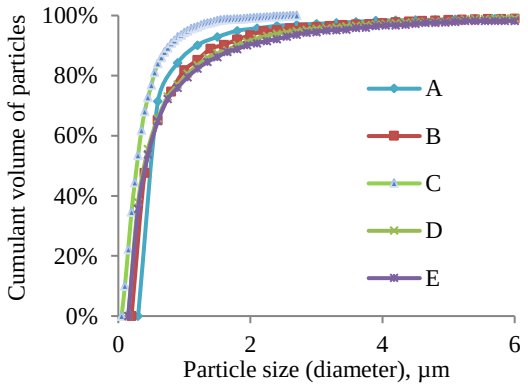

Fig.5. PSD of AMGL/AMWCNT hybrid composite.

The relation between the cumulated volume of particles and particle diameter is shown. Slight variation in PSD is observed for different compositions of hybrid composites when compared with DLS results which verify the analysis. The smallest particle size diameter is observed for sample code C.

Cumulative results of IA are indicated in Table 6. Results obtained depend on image selection, its quality, and processing method. Minimum sizes of particles are present in sample C, which ranges from 0.066 to 2.678 µm. Similarly, the maximum size of particles is present in sample A which ranges from 0.33 to 57.97 µm. The volume-based distribution delivers a minimum mean particle size of 0.29 µm for sample C. Results observed from DLS outcomes and image analysis resemble each other. This validates both methods.

Table 6. Cumulative result of image analysis.

Volume %	Diameter, d µm			PI
	10	50	90	
Sample code				
A	0.11	0.42	0.36	0.3895
B	0.12	0.50	1.18	0.1348
C	0.15	0.29	0.63	0.2667
D	0.16	0.39	1.25	0.1469
E	0.10	0.42	1.32	0.1031

3.3. Tensile properties of hybrid composites

Mechanical characterization of hybrid composites in the form of tensile tests is performed on five specimens for each sample code. Test results of the hybrid composite with standard deviations are illustrated in Fig. 6 and Fig. 7. The brittle failure in the form of the stress-strain curve of different hybrid composites can be seen in Fig. 6. Samples C and D show the best results for brittle failure. Thus composite with filler content below 1 wt% shows better tensile properties. However, tensile properties decrease when filler content is increased up to 2 wt% due to difficulties faced in obtaining uniform dispersion and formation of agglomeration, as shown in SEM images, Fig. 2.

Comparative analysis of tensile stress and elastic modulus for different sample codes of hybrid composite is shown in Fig. 7. An increase of 19.25 and 13.10% in tensile modulus is observed for samples B and C, respectively, in comparison to pure epoxy sample A. This may be due to imperfections involved in sample preparation majorly contributed by an optimal value of sonication, resulting in sedimentation, which further forms small clusters of nano-fillers.

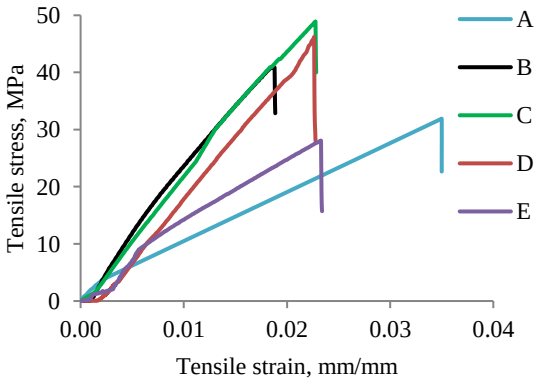

Fig. 6. Tensile test curves of hybrid composites.

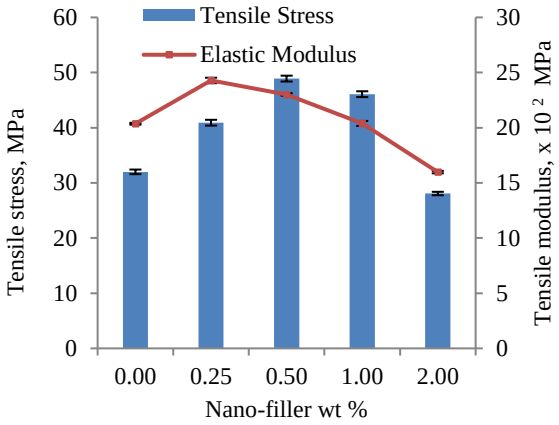

Fig. 7. Tensile stress and elastic modulus of hybrid composites

The maximum value of tensile strength (49.91 MPa) and minimum particle size observed for composite with filler content is 0.5 wt%. For composite with a filler content of more than 0.5 wt%, the size of particles becomes coarser. The maximum value of tensile properties observed for hybrid composite with a filler content is 0.5 wt%. DLS results indicate mean particle size of the composite varied from 2.31 to 29.98 µm, whereas the image indicates variation from 0.25 to 5 µm. Variations in terms of particle size due to different methods followed for analysis of particle size. However, a similar pattern of PSD for different composite samples is shown by both DLS and IA methods.

Variations in particle size dimensions from IA and DLS are due to the number of particles considered for particle size evaluation from both methods. In SEM the numbers of particles considered are lower in comparison to DLS techniques [30]. IA shows mirror processing of certain sections of SEM images which may be the reason for variation in statistical results. Also, the DLS method is based on 3-D analysis, whereas IA specifies a 2-D image. Another reason for variation in IA and DLS results is the sphericity of the aggregates. This becomes the reason for similarity in the pattern of PSD by both methods though variation in numerical values exists. Current research work may be helpful in the selection of filler particle size to achieve improved tensile properties of hybrid composite.

3.4. EDX analysis

EDX analysis, as shown in Fig. 8(a), justifies the chemical composition of carbon and oxygen in the pure epoxy composite. EDX analysis confirms the presence of carbon and oxygen as 97.16 % of the total elemental composition. However, Fig. 8(b) illustrated the presence of other elements, such as silicon and chlorine. Similarly, EDX analysis of hybrid composite shown in Fig. 8(b) confirms the presence of carbon and oxygen as main elements and found 99.65 % of the total elemental composition. Elements present in the substrate, due to chemical reaction and impurities, constitute for remaining 0.35% weight percentage, accumulating other elements such as magnesium, aluminum, silicon, chlorine, potassium, calcium, and iron.

The dark areas in SEM micrographs of Fig. 2 are due to the presence of carbon, whereas white areas indicate the presence of oxygen. The diffused grey regions constitute remaining element.

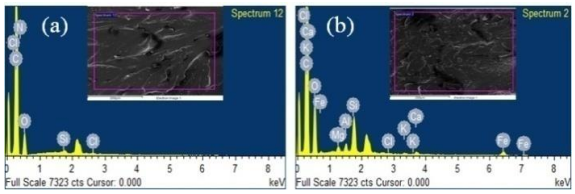

Fig. 8. Elemental composition of hybrid composite obtained from EDX analysis; (a) pure epoxy and (b) hybrid composite**.**

4. Conclusions

A hybrid composite comprising different filler weight percentages of AMGL and AMWCNT in epoxy resin is successfully fabricated. Investigation of particle size along with PSD analysis, mechanical properties, and chemical composition is performed. Characterization of hybrid composite is done using SEM, DLS, IA, and EDX, while mechanical testing is being done through a tensile testing machine. Conclusions of research work are enumerated below:

- The synergetic effect of both AFML and AMWCNT nano-fillers with their different

wt% on PSD and tensile properties of hybrid composites is analyzed. The best results for tensile tests and optimum particle size are produced for a 0.5 wt% (each AFML and AMWCNT) hybrid composite. The optimum composite particle size for 0.5 wt% sample comes out to 6.85 µm. This information may be very much helpful in procuring the nano-fillers for the initial fabrication process of hybrid composites. Thus results are helpful in optimizing the processing of hybrid composites, the particle size of constituents of composite and the properties of composites.

- The comparisons of above mentioned composite particle size and SEM images with different wt% tested samples through the DLS method using image J software are also performed in this work. The composite particle size obtained by this method comes out to 0.29 µm, which can be considered as nano-dimension but very much differed from the value obtained through PSA. This may be justified through the number of particles evaluated in SEM image analysis being lower in comparison to those considered in DLS method. This may be further justified that for the same volume the cluster formation in DLS contained more particles as compared to image J software, therefore, this study certainly helps the researcher in performing the analysis of microstructures at a lower level of particle dimensions.
- Due to chemical reactions during composite processing different elements in the composite sample are present. Further investigation of composite by chemical characterization using EDX analysis reveals that the composition of oxygen and other heavy metals occurs more in the hybrid composite as compared to pure epoxy composite, as a result particle size increases.

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