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Effect of Micro Glass Flake on Morphological and Rheological Behavior of Epoxy Vinyl Ester Composite Coatings

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ABSTRACT

In the present work, attempts were made to investigate the reinforcement and treatment effect of glass flake (GF) on morphological and rheological behavior of GF/epoxy vinyl ester composites. GF was incorporated into epoxy vinyl ester resin by sonication and mechanical agitation. Rheological and morphological properties were studied as a function of flakes treatment and size distributions. The dispersion morphology, agglomeration degree and homogeneity of additives were analyzed by scanning electron microscopy (SEM) and optical microscopy. The effects of mixing method, surface treatment, and particle size distribution on rheological properties of composites were investigated with a modular compact rheometer. The results demonstrated that the mechanical agitation decreases GF aspect ratio more than sonication. SEM fractography confirms that finer particles improve toughness properties of the composites in comparison with larger ones. GF with larger circular diameter and length is more sensitive to shear than the smaller one. Rheological investigations showed that surface treatment improves the interaction between the polymer matrix and GF. Based on the results, it is indicated that the finer the GF, the better rheological properties will be displayed. Prog. Color Colorants Coat. 10 (2017), 31-41© Institute for Color Science and Technology.

1. Introduction

Over the years, polymeric materials have developed due to their good barrier properties [1]. Epoxy vinyl ester resins are the most important thermosets, widely used as adhesives, coatings, and composite materials [2-4]. In order to enhance the barrier protection, magnetic recording and physical properties such as modulus, thermal expansion, thermal and electrical conductivity of these resins, reinforced fillers have been incorporated [5, 6].

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In comparison with many other flake-like fillers, glass flakes (GF) gained popularity because they have a large aspect ratio and unlike other similar planar fillers, can be made in a variety of thickness to diameter ratios and are not stepped, they are totally impervious to moisture vapor and consistent in composition [7]. Moreover GF has appropriate mechanical properties and is generally considered a simple dust hazard or non-hazardous, particularly when compared with fibers and some other pigments. Other barrier pigments commonly used are opaque, often strongly colored and have poor aspect ratios. Therefore, GF can offer significant advantages in engineering thermo-polymeric coatings due to its unique and special properties [8]. GF containing coatings are being investigated in relation to metal protection in highly corrosive media because they are expected to greatly hinder the water transport through the polymeric matrix [9-12]. GF reinforced products also display good resistance against weathering and chemical attack. The reinforcement effect of glass flake strongly depends on the interfacial adhesion between the glass flake and the surrounding polymer matrix. Besides, the adhesionproperties of the GF containing coatings can beenhanced by incorporating silane based adhesion promotor inthe coating [13]. The bonding of fillers into the resin is a highly important feature in obtaining performance both from a permeation resistance view and mechanical performance. Silanes have been used for many years in the glass fiber industry to improve bonding and in consequence performance having dual functionality being capable of interacting with the GFs and crosslinking into the matrix to form continuous bonding [1, 14-16]. Generally, the performance of thermoset resins is improved by adding a silane coupling agent just before flake loading. Furthermore, size and thickness of flakes are both remarkablefactors on obtaining high performance composites. The quantity and particle size distribution of GF are important, too. A further consideration is whether or not coupling agents are used to provide better adhesion of the GF to the binder and in the case of coatings, the substrate. Interfacial properties play an essential role in determining the mechanical behavior of polymer composites. The degree of interfacial bonding between the reinforcement phase and the surrounding matrix can also alter the rheological and mixing properties of the final composites [13]. Both the filler concentration and interactions alter the rheological and processing characteristics of the resin [17]. The incorporation state of glass flake into the resin can introduce significant performance advantages. The problems in compounding approach are the reduction of aspect ratio of the fibers and flakes. This can be partially solved by surface coating of particulates in order to minimize the required mixing time, incorporating the particulates into polymeric liquids at the highest possible temperature to reduce μ, impinging mixers or other types of equipment where mechanical or high shear breaking action is limited [18].

The ultimate properties of composites are strongly dependent on the particle size, degree of dispersion, and the interactionsoccurred between the interface of particles and polymer matrix. Homogeneous dispersion of inorganic parts in the organicmatrix dramatically becomesfeasible, owing to the lack ofaffinity between the interfacial contact area of inorganic fillersand organic polymers. Therefore, a proper remedial measure forthis flaw is crucial to develop new nanocomposites. Rheologicalmeasurement is one of the most effective methods to analyzecomposites. Up to now, most of the reportsfocus on the study of thermoplastics and a few studies reported the properties of thermosetting composite [13-16].

To the best of our knowledge, few papers have been published on the influence of concentration, particle size distribution, adhesion promoter, incorporation, and aspect ratios of GF on the performance and rheological properties of composites which is so important factors on enhancement of adhesion to substrate and longer barrier spots of GF-filled coatings result in a great improvement of corrosion resistance performance compared to pristine epoxy coatings [13-16].

It is possible to alter the incorporation of GF by employing various mixing methods. Two types of GFs (micro and milled) with different aspect ratios were selected in this study. Effect of mixing method on morphological behavior of GF reinforced epoxy vinyl ester composites was investigated. The effect of surface treatment, mixing method and particle size distribution on rheological properties is also consideredin this research.

2. Experimental

2.1. Material

Two types of GF ingredients were used for this research namely GF003 and GF300M, provided by GF Co. (England). General properties of GFsare shown in Table 1. Bisphenol-A epoxy vinyl ester resin with viscosity of 450 cps at 25 °C was supplied by Swancor Company (Taiwan). The curing agents for this resin were 1.5% cobalt naphthenate, 0.1% dimethyl aniline, and 2% methyl ethyl ketone peroxide in diisobutyl phthalate (LPT) provided by AkzoNoble. Around 0.25% benzoin was used as a degassing agent. The composite samples with different compositions were prepared using three forms of GF; untreated GF003 (GF-003un), treated GF300M (GF-300M), and untreated GF300M (GF-300M un). GF-300M was treated with 3-aminopropyl triethoxysilane by the company and was used without further treatment.

2.2. Sample preparation

A specified amount of GF (15 wt %) was dispersed in epoxy vinyl ester resin using two combining methods: mechanical mixer with 500 rpm and sonication at room temperature. Each method was applied at different times referred to as M1 to M4. The mixing mechanisms arelisted in Table 2. Subsequent to mixing, 2.0% of the curing agent (LPT) was added followed by a thorough mixing fora bout 5 minute. All four methods have been used for each types of GF. Notation of samples is selected based on mixing method. For instance, GF003unm2 correspond to dispersion of untreated GF003 by using M2 mixing method.

2.3. Particle size measurement

The optical homogeneity of filler dispersions in epoxy resin was examined by an Olympus BHZZ-UMA optical microscope.

In order to determine the GF aspect ratios, a semi automated SA-CP3 image analyzer was used. With the purpose of suspend GF in acetone, GFs were extracted from resin via the solution extraction method.

Table 1: Physical properties of three types of micro GFs**.**

<table>
<tr><th rowspan="2">Material</th><th colspan="5">Properties</th></tr>
<tr><th>Treatment</th><th colspan="2">Particle size distribution (µm)</th><th>Thickness (nm)</th><th>Density (g/cm3)</th></tr>
<tr><td>GF-300M</td><td>3-aminopropyl triethoxy silane</td><td>1000-300µm
300-50µm
<50µm</td><td>10% or less
65% or more
25% or less</td><td>2.3 - 3.3<m</td><td>2.60</td></tr>
<tr><td>GF-300M un</td><td>untreated</td><td>1000-300µm
300-50µm
<50µm</td><td>10% or less
65% or more
25% or less</td><td>2.3 - 3.3<m</td><td>2.60</td></tr>
<tr><td>GF-003 un</td><td>untreated</td><td>>150µm
150-50µm
<50µm</td><td>2% or less 10% or less
88% or more</td><td>2.3 - 3.3<m</td><td>2.60</td></tr>
</table>

Table 2: Different dispersion procedures.

Mixing method code	Time of mixing (min)	
	Mechanical agitation (500 rpm)	Sonication
M1	10	15
M2	10	30
M3	20	15
M4	30	15

2.4. Scanning electron fractography analysis

In order to examinethe surface characteristics of modified coatings, all the experimentally fractured specimens were metallized with gold using a sputter coater to provide surface conduction and observed with a scanning electron microscope (SEM; Cambridge Stereoscan, model S-360, Germany).

2.5. Rheological properties

Rheological properties such as dynamic viscoelastic and steady state viscosity measurements were performed on samples with a modular compact rheometer (MCR300). Due to the low viscosity of prepared systems, experiments were carried out with a co-axial cylindrical geometry having a gap of 1 mm according to the standard concentric cylinder geometry of CC27 presented by PaarPhysica. Angular frequencies under oscillatory shear deformations were set at 0.5 (1/s) and stress amplitude between 1 Pa and 1000 Pa was applied. A humidity chamber was designed and placed to minimize water evaporation during the measurements.

3. Results and discussion

3.1. Effect of mixing method on morphological behavior of composites

Although previous researches, which only had used optical microscopy as an assessing tool, had indicated that dispersing of GF in vinyl ester matrix was not complicated, further investigations revealed that the dispersion was not enough, and GF platelets stuck together [19]. The reinforcement effect of polymer composites strongly depends on the filler’s structural parameters, such as shape, aspect ratio, modulus, volume fraction, interfacial adhesion, surface characteristics and orientation [2–6]. The improved reinforcement efficiency is observed usingfillerswith higher aspect ratio.

On the other hand, GF particles are very sensitive to shear. Mixing can break GF platelets and then decrease aspect ratio distribution or anisotropy. Both of these events diminish the barrier properties of composite which plays the most important role for this filler for using in the coatings.

The better interaction of treated GF and surrounding matrix can also reduce damaging effect of mixing method and provide superior dispersion. Particle size analysis is used for evaluating the reduction of flake's aspect ratio during dispersion in the resin and suffering different tension. Figure 1(a) to 1(c) show cumulative percent (%) of GF aspect ratio vs. aspect ratio for three types of GFs after dispersion in resin via four different mixing methods, and separation of them by the solution extraction approach. Particle size distribution of the extracted particles was analyzed by image analyzer software. An example of analyzed images is illustrated in Figure 2.

As it can be seen in Figure 1, the applied mixing methods have little destructive effect on all types of GFs. Although the aspect ratio of different GFs more or less is similar, it seems that the damages are more evident for the untreated and larger ones. On the other hand, the surface treatment of GF particles can reduce destructions of flakes due to better dispersion and reduce stickiness which was attributed to the interfacial interaction of hydroxyl and esteric groups between the fillers and the matrix. Figure 1 shows that aspect ratio of GF is more damaged with the mechanical agitation (M4)in comparison with the ultrasonic mixing method.

Table 3 indicates the percentage of variation in aspect ratio of mixtures stirred with four different dispersion procedures. As it can be observed in Table 3, aspect ratio of GF does not show a remarkable change during the sonication process while mechanical agitation has a stronger effect on smashing all GF types whichrepresent more damaging consequence of mechanical mixing in comparison with sonication for dispersion of these flakes. On the other hand, the untreated GF with a larger circular diameter and length (GF300) was more sensitive to shear than GF with smaller particle size distribution (GF003). This was attributed to the larger breakable surface area in GF300M which make it more fragile against energetic waves and shear tensions.

Therefore, surface treatment of flakes can confine the detrimental effect of mechanical or high shear mixing methods. Generally, two mechanisms have been found in breaking of fragile fillers: mechanical forces and abrasion of particles together. However, it is not possible to determine which of these mechanisms can be more predominant in breaking the particles. Obviously, surface treatment due to increment of filler and matrix interaction can moderates harmful effect of both mechanisms.

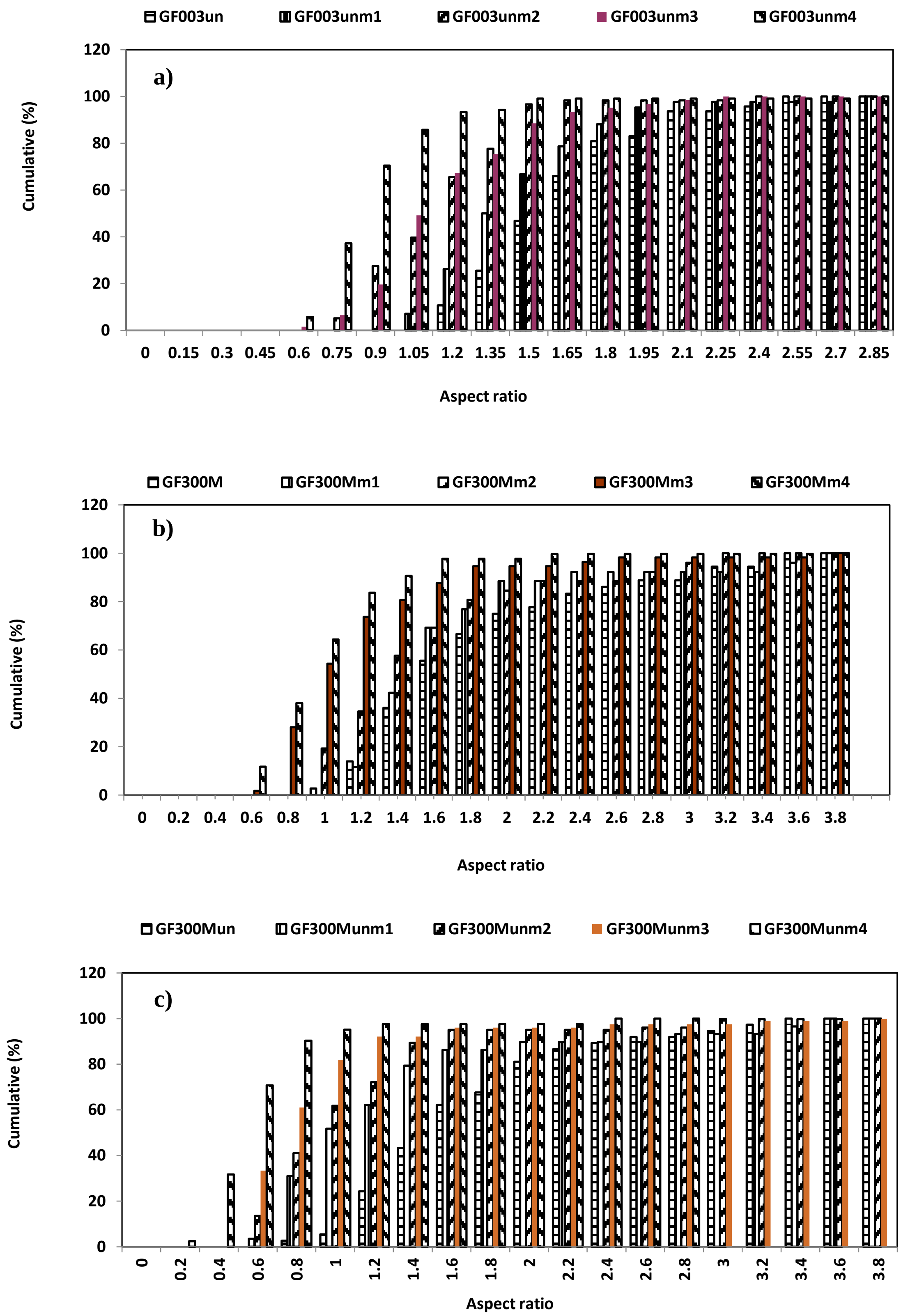

Figure1: Particle size analysis of GF particles after stirrings via four mixing methods. Cumulative percent aspect ratio via aspect ratio for (a) untreated finer GFs (GF003), (b) treated larger glass fakes (GF300M) and (c) untreated larger GFs (GF300Mun).

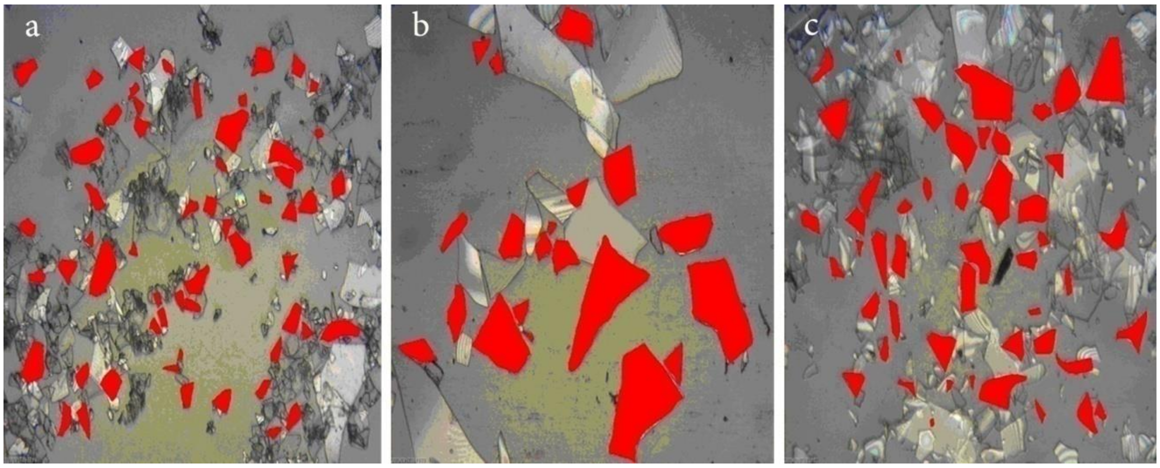

Figure 2: An example of analyzed images (taking image via optical microscopy by magnification of 270X and 135X for finer and bigger platelets, respectively): (a) untreatedfiner particles (GF003), (b) Treated bigger particles (GF300M) and (c) Untreated bigger particles (GF300Mun).

Table 3: Percentage of change in aspect ratio of different GF-filled epoxy vinyl ester composites stirred in four different dispersion procedures.

Mixing method code	Percentage of change in Aspect Ratio		
	GF003un	GF300M	GF300Mun
M1	0.06*	0.04	1
M2	1.2	1	1.5
M3	1.6	1	1.5
M4	2.01	1.01	2.7

Percentage of change ⁎: $\rho(1) - \rho(0)/\rho(0) \times 100$

Where $\rho(1)$ and $\rho(0)$ are aspect ratio of stirred and primary glass flake, respectively.

3.2. Effect of mixing method on rheological properties of composites:

The effect of GF on the rheological properties of prepared compositesby various mixing methods and times were probed using rheometer in shear rate range of 0.01-1000 (1/S).

As mentioned before, level of dispersion and flake size distribution have altered through four different mixing procedures. Change in shear history of mixtures is investigated by rheological experiments. This technique allows investigating the relaxation caused by the Brownian motion of the main chains due to the transitions occurred during the levelling process. It is likely that this motion can be affected by the type of mixing method and also the presence of GF because of the interactions in the interface layer surrounding the flakes [13-16]. The flow curves (apparent viscosity, μ, vs. shear rate, $\gamma^{\cdot}$) in low amplitude were fitted to the power law expression:

$$\mu = A\,\gamma^{\cdot(n)} \qquad (1)$$

Where μ is the apparent viscosity, A is the specific pre-exponential factor, $\gamma^{\cdot}$ is the shear rate and n is the shear thinning exponent. Figure 3 represents the flow curves of GF300-filled samples. Variation of absolute value of viscosity vs. shear rate for all the samples suggests an almost shear thinning behavior at different shear rates. In order to determine A and n, a plot of log (μ) vs. log ($\gamma^{\cdot}$) was prepared and fitted to a straight line. The shear thinning exponent (n) is the proposed semi-quantitative measure of micro-dispersion of the sample.

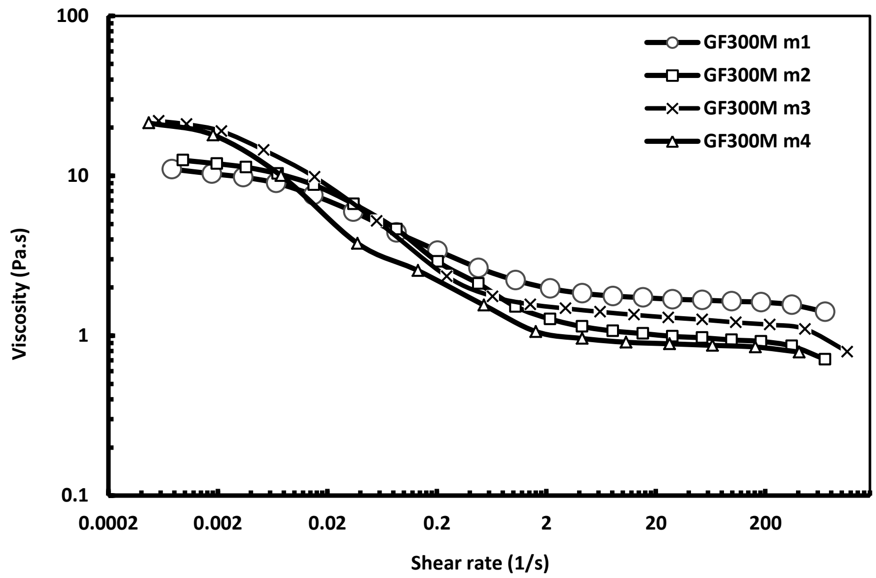

Figure 3: Viscosity of GF300-filled composites as a function of shear rate in different mixing methods.

The respective values of the exponent n for M1 to M4 are -0.29, -0.40, -0.41 and -0.42, respectively. Apparently, the shear thinning exponent allows a quantitative discrimination between the samples. On the other hand, the samples which were stirred by M4 showed the highest amount of shear thinning exponent while the samples with more mixing time showed higher zero shear viscosity, which confirmed the yield phenomena in the GF composites. According to the results, shear thinning behavior is observed in the systemby adding GFs. Since levelling is raised in low shear rates, and rheological properties oscillations are not suitable in this state, higher rheological index leads to better levelling. This issue represents the definite preference of M1, M3 and M4 mixing methods compared to M2 method. Definitely, the increase in n index in M3 and M4 methods is obtained by breakage and defects of most of the flakes which examined in the previous parts. Although increasing of isotropy as a result of particle breakage is desirable, the consequences of this phenomena is the loss of matrix interactions and the anti-corrosion performance of these systems. The M2 method is the best method of mixing in this system. Even though, the degree of dispersion has only remaineda limited effect on the anisotropy, flake breaking decreases anisotropy and then diminishes the interaction between GF and matrix. Thus, mixing method M2 is chosen as the best dispersion procedure.

3.3. The effect of surface treatment on rheological properties of composites

Viscosities of the resin suspensions with different modified and unmodified GFs are comparatively studied at different rates as shown in Figure 4. The results show that the epoxy resin alone is almost Newtonian with the complex viscosity being independent of the angular frequency [13-16]. As the dispersive medium is almost Newtonian fluid, most of the elastic and strain-rate dependent behaviors in the GF composites suspensions are driven from the addition of the GF and/or their degree of dispersion and orientation [18]. According to the viscosity results, both composites showed ashear thinning behavior that confirms the micro-structure formation.

The presence of silane groups on the GFs surfacecould introduce an additional ‘‘soft layer’’ preventing theagglomeration of GFs and subsequently improving thedispersion quality [13-16]. It might be deduced from Figure 4 that the modified GF composite showed higher zero shear viscosity compare to untreated GFs containing composite due to further interfacial bonding between the GFs and the epoxy vinyl ester as a result of surface modification of GF. The reduced filler–filler interaction was manifested by the mono-dispersion of the GFs [13-16].

Clearly, the shear thinning exponent of treated and untreated GF-filled samples are the same. Shear thinning exponent is truly dependent on the meso-scale composite structure formed by the (partially) exfoliated platelets [18]. Therefore, treatment of GF improves the interaction between the polymer matrix and GF.

3.4. Effect of particle size distribution on rheological properties of composites

GF aspect ratio determines to which extend GF sheets interact with each other to build an inter-connecting network known as percolation [13-16]. However, in practice, the dispersion quality plays an important role in corrosion properties of the composite [14].

The results of storage modulus (G') of epoxy/GF suspension are presented in Figure 5. Storage modulus is measured by steady state viscoelastic tests in linear viscoelastic region in which not only deformations do not create any changes in molecule's structures but also release them from primal equilibrium state and entanglements.

In order to represent the effect of size distribution of flakeson rheological behavior, two different unmodified GFs were selected; therefore there is not any further interaction between resin and filler. As it isillustrated in Figure 5, it can be concluded that an addition of finer GF forms a more stable, resistance and smaller structures which dropped off and decomposed in higher stress.

Higher surface area between finer GF and matrix leads to formation of a network-like structure while larger particlesformed unstable network in which poor interaction between untreated GF300 particles and vinyl ester in the composites can be observed. Also, the larger amount of initial modulus of GF300un-filled composites indicates that larger structures are formed by using larger flakes.

3.5. Fractography

SEM was used to observe the dispersionofGF, filler–matrix interface and interaction on the fractured surfaces of the cured composites. The enhancment of distribution by treatment of flakes has positive effect on general dispersion of GF in the matrix [19-21]. SEM micrographs of composites with 15 wt % (of different GFs mixed by M2 methode are depicted in Figure 6.

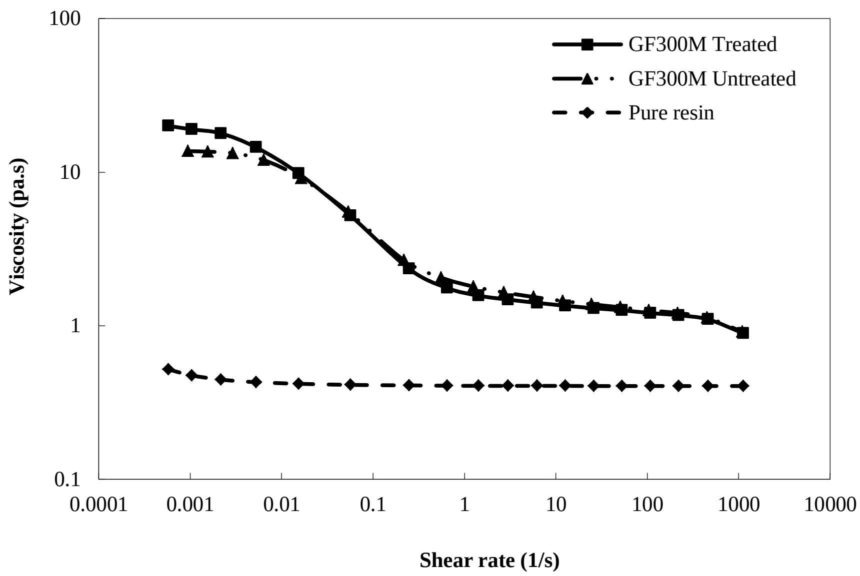

Figure 4: Viscosity of pristine resin and GF filled composites as a function of shear rate in different mixing methods.

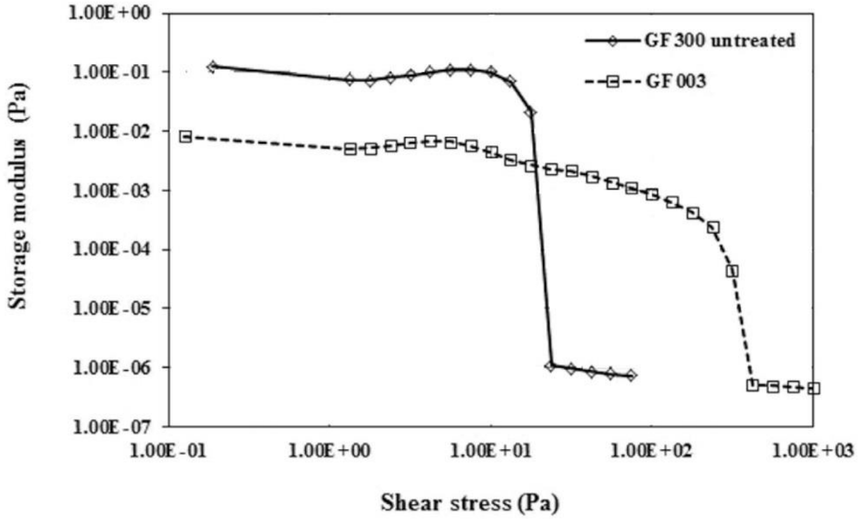

Figure 5: Storage modulus vs. shear stress of GF filled composites.

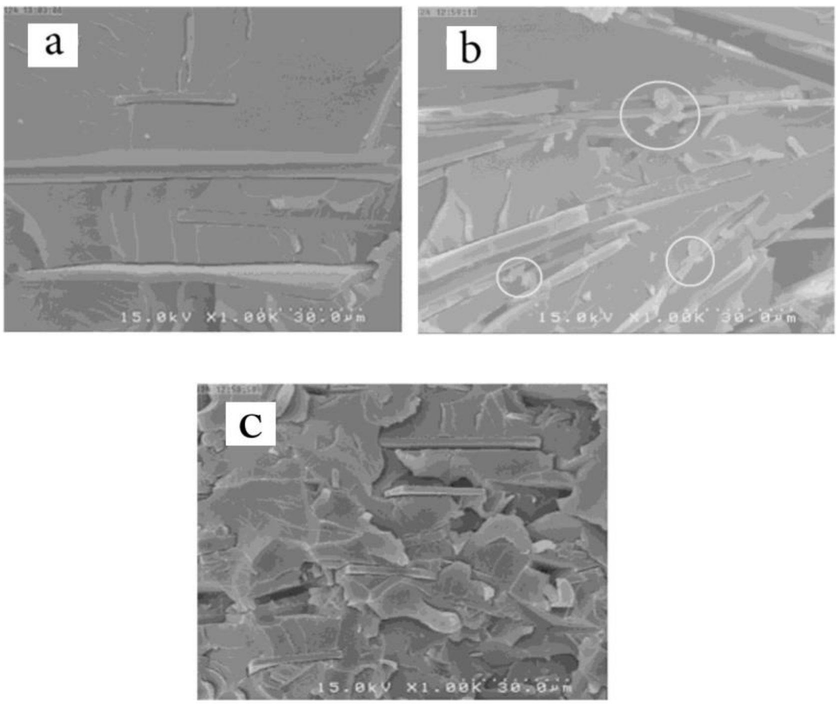

Figure 6: SEM images of fracture surface of epoxy vinyl ester composites prepared by M2 mixing method. (a) GF-300M treated by amino silanes, (b) untreated GF-300M and (c) untreated GF-003.

As it is shown in Figure 6, the state of dispersion was approximately different in the samples and it was almost uniform. In addition, a suitable adhesion between GF and resin matrix was attained using treated GF300M. A considerable aggregation of particles is observed when untreated GF300M particles is used as representative of the presence of large voids around the particle aggregates. Some aggregated flakes depicted in Figure 6b which are assigned by white circles.

The fracture surface of larger GF filled samples

(GF300M) reveals a brittle behavior characterized by large smooth area which corresponds to the low growth of crack-like defects while finer particle indicates a rough fracture surface as a result of increasing the surface area. therefore, finer filled GF samples are tougher than larger ones. This result is confirmed with mechanical tests which will be studied in future work of this team.

4. Conclusions

It was demonstrated that GF mixing can lead to breaking of particles and a decrease in average aspect ratio. The shear thinning exponent (*n*) is a semi-quantitative measure of the degree of anisotropy. Two mechanisms were found in the breaking process: the mechanical agitation and abrasion of particles together. It was found that more failure of particle is caused by using mechanical agitation mixing method.

On the other hand, it is shown that in order to minimize the mixing time, surface treatment of particles can limit mechanical or high shear breaking effect. GF with a larger circular diameter and length was more sensitive to shear than smaller particles.

Rheological properties of resin were altered by incorporating GFs. Studies showed that decreasing of average aspect ratio declines shear thinning of samples. Treatment of GF improves interaction of polymer matrix and GFs. Furthermore, among untreated GF-filled samples, the composites containing finer GF showedimproved rheological properties.

Additionally, the properties strongly depend on the flow-induced morphology and the yield phenomena or orientation. The rheological experiments indicated that the power law index (n) was decreased in the sample with larger mixing time while this sample showed higher zero shear viscosity, which confirmed that filler-matrix network formed as a result of filler breakage in the mixing process andalso became more elastic by using finer flakes in the composites.

SEM fractography showed that the enhancment of distribution by treatment of the flakes has a positive effect on general dispersion of GF in the matrix. Also,the suitable adhesion between GF and resin matrix has been attained when treated GF300M is used.

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