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Preparation and Properties of Epoxy Nanocomposites. I. The Effect of Premixing on Dispersion of Organoclay

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The objective of this work was to systematically study the effect of the conditions of shear and temperature used to premix nanoclay with epoxy resin on the dispersion and intercalation/exfoliation of the nanoclay in the resulting epoxy nanocomposites. The different mixing approaches used were hand stirring, conventional mechanical stirring, a high-speed homogenizer, and a microfluidizer. The quality of dispersion and intercalation/exfoliation of the organoclay in the epoxy resin was analyzed after premixing (before adding hardener) by means of X-ray diffraction (XRD) and rheological measurements. Nanocomposites obtained after adding hardener and curing were characterized by means of XRD, field emission gun scanning electron microscopy, transmission electron microscopy, and image analysis. Both the premixing and curing steps were found to play a determining role in the dispersion and intercalation/exfoliation of organoclay. Increased intercalation and exfoliation can take place during curing, with the extent depending on the curing rate. Although full exfoliation of clay cannot be achieved at the premixing stage, this step appears to be very important in controlling the micro-dispersion and thus affecting the further intercalation and exfoliation that take place during the curing step. POLYM. ENG. SCI., 49:666–672, 2009. © 2009 Society of Plastics Engineers

INTRODUCTION

The dispersion of organoclay in epoxy resin is a complex process, which takes place during the premixing step and the curing step. Both pristine and commercially treated clays tend to form stacks rather than individual platelets owing to their layer structure and the strong ionic and hydrogen bonding forces resulting from the cations and (in some cases) water molecules located between the layers. In most cases, the stacks combine and form large aggregates as a result of such strong secondary interactions. Consequently, it is very difficult to overcome these interactions to disperse the clay layers individually in the epoxy matrix, especially when the two phases are incompatible. During the fabrication of an epoxy nanocomposite, normally, the clay is first premixed with the liquid epoxy resin. In the absence of the curing agent, the system can have an extended shelf life at certain elevated temperatures. Therefore, different means such as mechanical shear and elevated temperature can be used to facilitate the dispersion. In the end, the system is cured by the use of hardener. At this curing stage, external shear stress may not be applied, but further dispersion, more specifically, intercalation and exfoliation, can continue to take place at the beginning of the curing as the hardener and/or intermediate molecules are still mobile enough to diffuse into the clay galleries; the driving force behind this diffusion is chemical thermodynamics. In general, it is easier to achieve good clay dispersion in the premixing step than in the curing step. Dispersion process parameters mainly include premixing temperature, mixing speed and time, power of ultrasonic tooling, shearing forces, etc. Direct mixing of organoclay and epoxy with mechanical mixing and sonication is widely used to disperse nanoclay in epoxy [1–5]. However, it is not enough to produce a high degree of dispersion. Yasmin et al. [6] used a three-roll mill to disperse the clay nanoparticles in an epoxy matrix and improved the distribution of the particles. Chen and Tolle [7] achieved fully exfoliated layered silicate epoxies by high-shear mixing in the presence of acetone. Liu et al. [8–10] used a high-pressure mixing method with assistance of acetone solvent to improve the dispersion of clay in epoxy nanocomposites and observed significant improvement in fracture toughness. However, the large amount of solvent required meant that considerable time was required for removing it. There is still work

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TABLE 1. Notation used to describe the different mixing conditions.

Mixing method	Temperature/time	Shear
Rm	R = Room temperature/1 h	m = Hand mixing for first few minutes only
Tm	T = High temperature (120°C)/1 h	m = Hand mixing for first few minutes only
TM	T = High temperature (120°C)/1 h	M = Mechanical mixing, 1000 rpm
RS	R = Room temperature/1 h	S = High-speed homogenizer, 24,000 rpm
TS	T = High temperature (120°C)/1 h	S = High-speed homogenizer, 24,000 rpm
HP	Special high-pressure process using acetone and microfluidizer	

to be done to develop nanocomposites with fine dispersion and well-exfoliated morphologies. Achieving such morphologies with epoxy-based nanocomposites is a challenge, as high shear is always required but epoxy resin typically has rather low viscosity. There have been many attempts to prepare epoxy nanocomposites, with varying degrees of success and involving various preparation conditions. However, there are practically no reports of systematic studies to compare the effect of different mixing techniques for a given system. The objective of the work described in this article is to overcome this lack by systematically exploring different ways to generate the shear needed to improve the quality of dispersion and intercalation/exfoliation of nanoclay in epoxy nanocomposites.

EXPERIMENTAL

The epoxy resin used was the standard DGEBA (diglycidyl ether of bisphenol A) resin EPON™ Resin 828 from Resolution Performance Products (Houston, TX). This was cured with the polyoxypropylene diamine hardener Jeffamine® D-230 from Huntsman LLC (The Woodlands, TX) at a level of 32 phr. The organoclay used was Cloisite® 30B from Southern Clay Products (Gonzalez, TX). It was dispersed in the epoxy resin at a level of 2.69 phr, which leads to a loading of 2% by weight after addition of hardener and curing. This nanoclay consists of montmorillonite treated with methyl tallow bis-2-hydroxyethyl quaternary ammonium intercalant. It is widely used in epoxy resins because the 2-hydroxyethyl groups provide improved chemical compatibility with the relatively polar epoxy resin. The epoxy system and its nanocomposites were cured either at room temperature for 2 days or at 120°C for 2 h, with subsequent postcure at 140°C for 2 h in both cases.

Epoxy resin and nanoclay were premixed together under different conditions of mechanical shear and temperature. For future reference, these are summarized in Table 1. The first premixing condition (Rm) was a room temperature process performed without mechanical shear, in which the clay and epoxy were stirred at room temperature by hand (effective speed $<$ 100 rpm) for a few minutes, then left to stand at room temperature for 1 h. The second (Tm) was a "high temperature without shear" process, in which the clay and epoxy were stirred at 120°C by hand for a few minutes, then placed in an oven at 120°C for 1 h. The third method (TM) was a "high temperature with low-speed mixing" process, in which the clay and epoxy were stirred at 120°C and 1000 rpm for 1 h by means of a mechanical stirrer (Lightnin mixer from Cole-Parmer with high-efficiency axial flow impeller of diameter 6.35 cm). The fourth method (RS) was a "room temperature and high-speed mixing" process, in which the clay and epoxy were stirred at room temperature and high speed (24,000 rpm) for 1 h by means of a Model EW-04719-00 homogenizer from IKA Innovative Mixing and Processing Technology, equipped with a cylindrical rotor-stator mixing head of diameter 20 mm. The fifth method (TS) was a "high temperature and high-speed mixing" process, in which the clay and epoxy were stirred at 120°C by means of the homogenizer at high speed (24,000 rpm) for 1 h. The sixth one (HP) was a high-pressure process, in which organoclay was first dispersed in acetone (at a level of about 8%) with a Model M-110EH microfluidizer from Microfluidics ($\approx$15,000 psi or 103 MPa) to form a suspension, and then, the suspension was added into the epoxy resin according to the approach used by Liu et al. [8–10]. The desired amount of paste of organoclay and acetone was added to epoxy resin and then, the mixture was stirred by hand at room temperature. When the epoxy was visibly dispersed, the mixture was mechanically stirred at 1000 rpm in a fume hood at room temperature for 30 min, followed by slow heating to 80°C for 1 h. Finally, the mixture was degassed under vacuum at 95°C for 30 min. The typical batch size for all six mixing conditions involved 200 g of epoxy resin.

For curing, the required amount of amine hardener (32 phr) was mixed with neat epoxy resin or epoxy-clay mixture at room temperature for 5 min, and the mixture was then subjected to vacuum. Samples were cured either at room temperature for 2 days or at 120°C for 2 h, with subsequent postcure at 140°C for 2 h in both cases. Cured specimens were all prepared in the form of disks (2.5-mm thick and 30-mm in diameter) or plates (170 mm × 280 mm × 3 mm) to ensure conformity for comparison of results among the different samples.

To evaluate the intercalation/exfoliation of the nanoclay in the polymer matrix, X-ray diffraction (XRD) patterns were obtained from the surface of the samples with a Bruker Discover 8 powder X-ray diffractometer using CuKα radiation ($\lambda = 1.54250$ Å) and a 2θ scan range from 0.8° to 10°. The scanning speed was 0.6° per minute. For quantitative comparison of the degree of exfoliation among samples in the same series, a simple approach described in the literature [11] was followed. From the XRD results, an approximation of the degree of exfoliation X_E can be calculated according to the following equation:

$$X_E = 100\left(1 - \frac{A}{A_0}\right) \tag{1}$$

where A is the area under the XRD peak for the partially exfoliated nanocomposite and A_0 is the area for nonexfoliated but 100% intercalated nanocomposite. TEM observations at 10 different locations for the sample prepared by the Rm method, then cured at room temperature, indicated only intercalation, while SEM observation showed large clay aggregates. This sample was used as a reference for the exfoliation calculation (0% exfoliation and 100% intercalation). In other words, the area under the XRD peak of this sample was assigned as A_0. XRD peak intensity can be sensitive to the specimen surface topology. To minimize such effects, specimens for XRD measurement were cast in the form of disks as described above on the same selected Teflon molds and each specimen was measured at least twice. Each sample comprised two specimens.

A Hitachi-S4700 field emission gun scanning electron microscope (FEGSEM) was used to observe the dispersion of clay in the epoxy matrix at the micro-level with different magnifications of 1 k, 3 k, 10 k, and 40 k. Prior to observation, samples were prepared with a Leica EMFCS cryo-ultramicrotome. Samples also need to be made conductive by covering the sample with a thin layer of conductive platinum. This was done by using an EMITECH K575X high resolution sputter coater in which an electric field and argon gas were applied. For clay dispersion at the nano-level, ultra-thin (50 to 80 nm) sections of nanocomposite samples were prepared with a cryo-ultramicrotome and supported on a copper 200 mesh grid for observation with a Hitachi H9000 transmission electron microscope (TEM). The TEM was operated at a voltage of 200 kV and at a current of 110 to 120 μA. Rheological measurements for epoxy resin and epoxy-clay suspensions after mixing were performed at room temperature on a Brookfield CAP2000+ viscometer.

RESULTS

The X-ray diffraction curves of uncured EPON828-C30B mixtures after premixing by means of the different methods are shown in Fig. 1. The d-spacing values d_{001} corresponding to the first peak in the curves as well as the difference Δd between these values and that of the neat C30B clay are summarized in columns 2 and 3 of Table 2.

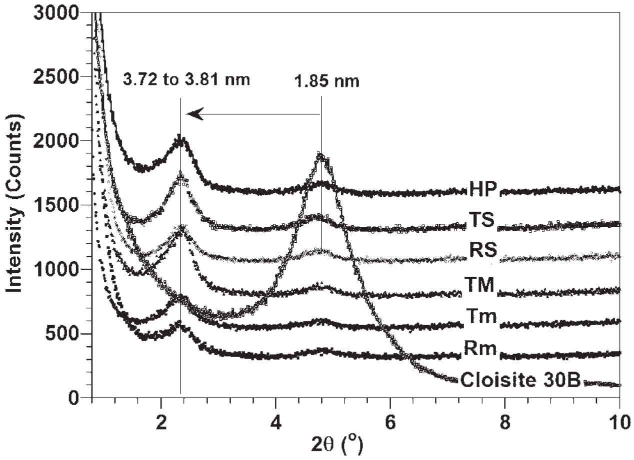

FIG. 1. X-ray diffraction curves of uncured EPON828-C30B mixtures prepared by means of different methods.

First of all, it is very interesting to observe that the clay in the mixtures prepared by hand mixing, both at room temperature and high temperature, has been easily further intercalated by the epoxy. The clay gallery distances are about double that of the pristine Cloisite 30B. Because the shear generated by hand mixing is very modest, the driving force for the intercalation process in this case is likely chemical thermodynamics (the compatibility between the epoxy and the clay surface). The presence of hydroxyl groups in the intercalant of the Cloisite 30B is believed to lead to increased interaction with the epoxy, possibly via hydrogen bonds. Although higher temperature and shear rate tend to give slightly higher d-spacings, the differences among the various mixtures are very small. All the mixtures give a value in the range of 3.72 to 3.81 nm. It is surprising that the basal spacing d of organoclay in epoxy did not increase significantly even with high temperature and high-speed aids. Moreover, the presence of the identical peaks in the XRD curves for the epoxy-clay mixtures clearly indicates that the clay was not fully exfoliated at the premixing step. It would appear that the main driving force is thermodynamic and that a stable state is reached well short of full exfoliation, regardless of the mixing technique applied. At this point, one may wonder if high temperature and/or high shear are really necessary to improve the intercalation. However, the SEM and TEM results for cured samples that will be presented later confirm that though XRD shows that shear and temperature have little effect on the level of intercalation, these parameters can reduce the clay aggregate sizes significantly, which facilitates further intercalation and then exfoliation of clay in the curing stage.

X-ray diffraction curves of the cured epoxy and its nanocomposites based on C30B made by the different premixing methods are illustrated in Figs. 2 and 3. A summary of the d_{001} values for the first peak in the XRD

TABLE 2. Summary of XRD results for uncured EPON828-C30B mixtures and their ENCs prepared by means of different methods.

	Before curing		Cured at room temperature		Cured at 120°C		
Mixing method	d_{001}	Δd	d_{001}	Δd	d_{001}	Δd	X_E
Cloisite 30B				1.85			
Rm	3.72	1.87	3.73	1.88	3.76	1.91	~ 54
Tm	3.74	1.89	3.83	1.98	3.84	1.99	~ 70
TM	3.76	1.91	3.86	2.01	3.86	2.01	~ 72
RS	3.80	1.95	3.91	2.06	3.95	2.10	~ 74
TS	3.81	1.96	4.01	2.16	4.03	2.18	~ 90
					6.69	4.84	
HP	3.73	1.88	3.83	1.98	3.94	2.09	

d_{001}, gallery spacing (nm); Δd, increase in d_{001} with respect to pristine C30B; X_E, degree of exfoliation (defined in the text).

curves of the ENCs, as well as the difference Δd between these values and that of neat C30B clay, is also given in columns 4 to 7 of Table 2. The degree of exfoliation values calculated from the XRD peak intensities for the samples that were cured at 120°C are given in column 8 of Table 2. In all the nanocomposite samples, the clay layer separation (degree of intercalation) is considerably higher than in the original C30B. Furthermore, when the nanocomposite values are compared with those of the corresponding uncured mixtures, it is seen that the nanoclay has been further intercalated somewhat by the epoxy matrix for curing at both room temperature and high temperature.

When samples were cured at room temperature (columns 4 and 5 of Table 2), the premixing method used was found to have some influence on the *d*-spacing in the ultimate product. In addition, the intensity (area under the peak) also changed depending on the premixing method. At the same loading level of 2 wt% C30B, the ENC made by the high temperature method (Tm) shows lower peak intensity than the ENC made by the room temperature method (Rm), indicating that premixing at high temperature leads to better delamination of clay than premixing at room temperature. On comparing "high speed at high temperature" (TS) with "high speed at room temperature" (RS), the same phenomenon is observed. "High speed at high temperature" premixing (TS) also shows better intercalation/exfoliation than mechanical premixing (1000 rpm) at high temperature (TM) and just high temperature (Tm). This indicates the effect of shear on the level of intercalation/exfoliation of clay in epoxy nanocomposites.

Curing at the higher temperature of 120°C (columns 6 to 8 of Table 2) leads to better intercalation/exfoliation than curing at room temperature. With the same premixing method, the intensity of the XRD peak is lower for curing at 120°C than for curing at room temperature. This can be explained by the fact that when the temperature increases, the mobility of epoxy and hardener molecules increases and because of this, they can diffuse more easily into the clay galleries and further intercalate or exfoliate the clay [4, 12]. A similar effect can be seen here on the intercalation/exfoliation of clay in the epoxy matrix when high temperature and high speed are used in the premixing step. As can be seen from the degree of exfoliation

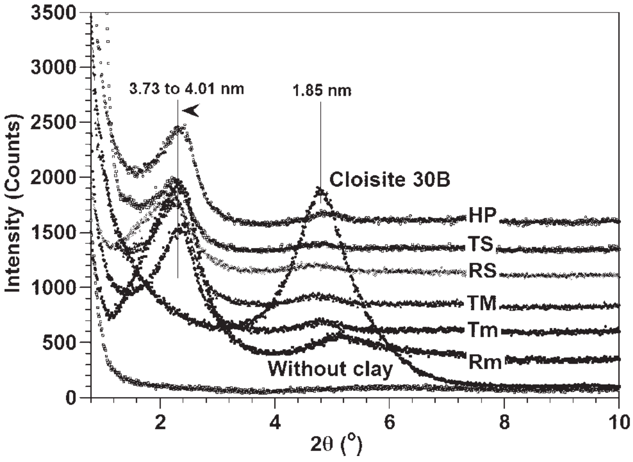

FIG. 2. X-ray diffraction curves of neat EPON828-D230, C30B clay, and their nanocomposites at 2 wt% C30B made by different methods and cured at RT.

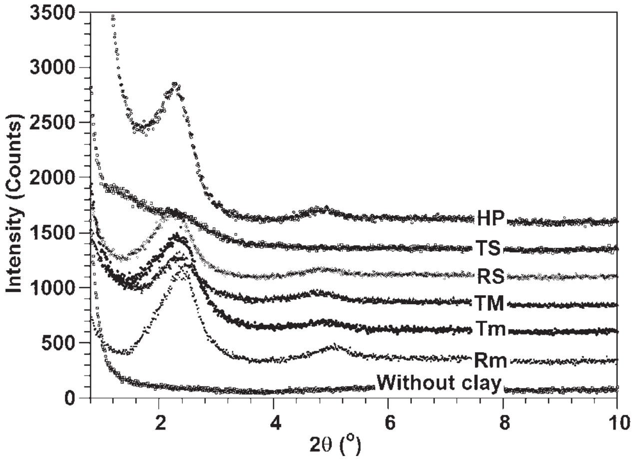

FIG. 3. X-ray diffraction curves of neat EPON828-D230, C30B clay, and their nanocomposites at 2 wt% C30B made by different methods and cured at 120°C for 2 h.

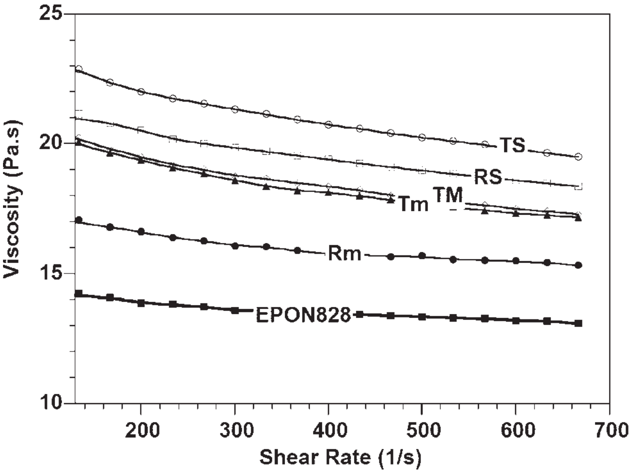

FIG. 4. Viscosity versus shear rate curves of EPON828 and its mixtures with C30B prepared by means of different methods.

values reported in the last column of Table 2, the intensity of the peak for the ENCs decreased when high temperature and speed were used in the premixing step. The orders of intercalation/exfoliation are now TS > RS, TM > Tm > Rm, TS > TM > Tm, and RS > Rm. It should be noted that there is little difference in peak intensity between Tm and TM. It can also be seen that there are two peaks in all the XRD curves for ENCs prepared by the Rm, Tm, TM, RS, HP, and TS methods. For ENCs prepared by Rm, Tm, TM, and RS, the two peaks are at around 2.3° and 4.8°. However, the peaks shifted to lower angles for the TS method. They are located at 1.3° and 2.2°, corresponding to *d*-spacings of 6.69 nm and 4.03 nm, respectively. The TS method shows better intercalation/exfoliation than all the other methods. The intensity of XRD peaks of ENC made by the HP method is lower than for the RS method. In addition, the XRD peak is more asymmetric for the ENC made by the HP method than for the one made by the RS method. It is believed that the HP method gives better intercalation/exfoliation than RS, although the *d*-spacing is a little lower for HP than for RS. In general, the level of intercalation and exfoliation of clay in the epoxy nancomposites is in the following order: TS > HP > RS > TM > Tm > Rm. The explanation for the better intercalation/exfoliation at this step for the ENC prepared with the "high speed and high temperature" premixing method is that the high speed and temperature helped to break down the clay aggregates to smaller sizes and thus produce a more homogeneous dispersion of the clay in the epoxy resin during the premixing step. This has an indirect subsequent effect on the intercalation/exfoliation of clay in epoxy matrix at the curing step. With smaller aggregates and a more uniform distribution, the epoxy resin and hardener have more chance to diffuse into the clay galleries and further expand the distance between clay platelets than for the case of large aggregates and nonuniform distribution, especially with high temperature curing where the mobility of the molecules increases.

Figure 4 shows curves of viscosity at 25°C versus shear rate for the epoxy EPON 828 and its mixtures with organoclay C30B made by different premixing methods. The viscosity values corresponding to a fixed shear rate of 167 s^{-1} are included in Table 3. It can be seen here that the viscosity of the suspensions changes according to the premixing condition, and it corresponds to the following order: $\eta_{TS} > \eta_{RS} > \eta_{TM} \approx \eta_{Tm} > \eta_{Rm}$. High temperature and high shear both show a positive effect on the viscosity of the suspension. For example, $\eta_{Tm} > \eta_{Rm}$, $\eta_{TS} > \eta_{RS}$ (temperature effect) and $\eta_{RS} > \eta_{Rm}$, $\eta_{TS} > \eta_{Tm}$ (speed effect). Again, the result here confirms that the clay has a better dispersion in epoxy resin when increased temperature and high speed are used in the mixing process.

The microstructures of ENCs based on C30B and made by different premixing methods were examined by SEM and are illustrated in Fig. 5. The bright spots on the backscattered images correspond to clay aggregates. Apparently, a portion of the clay remains at the micro-scale level with different size populations depending on the premixing conditions. However, given the resolution limitations of the SEM, one should not rule out the possibility that some exfoliation does take place. The size of aggregates is reduced significantly with high speed (Fig. 5d) when compared with mixing by hand at room temperature (Fig. 5a) or even mechanical mixing at high temperature (Fig. 5c). The size of aggregates became much smaller when both high speed and temperature were introduced at the premixing step (Fig. 5e). The result also confirms that the micro dispersion of ENC made by different premixing methods follows the order TS > HP > RS > TM > Tm > Rm.

From the SEM images, the distribution of agglomerates of clay in the epoxy as shown in Fig. 5a–e was analyzed by means of Image Pro Analysis Software. A summary of the average diameter, the maximum size, and the distribution of clay particles in epoxy nanocomposites made by different methods is given in Table 3 and Fig. 6. The TS method leads to much smaller aggregates (average size <1 μm) and a narrow size distribution. The result is much

TABLE 3. Properties of E828-C30B mixtures: viscosity of uncured mixture; maximum clay particle size; and average clay particle size in corresponding cured samples.

Mixing method	Viscosity (Pa·s)	Maximum diameter (μm)	Average diameter (μm)
None: E828 alone	14.10	–	–
Rm	16.80	24.71	4.15
Tm	19.65	22.65	3.39
TM	19.80	15.20	2.80
RS	20.53	5.81	0.56
TS	22.35	2.35	0.36

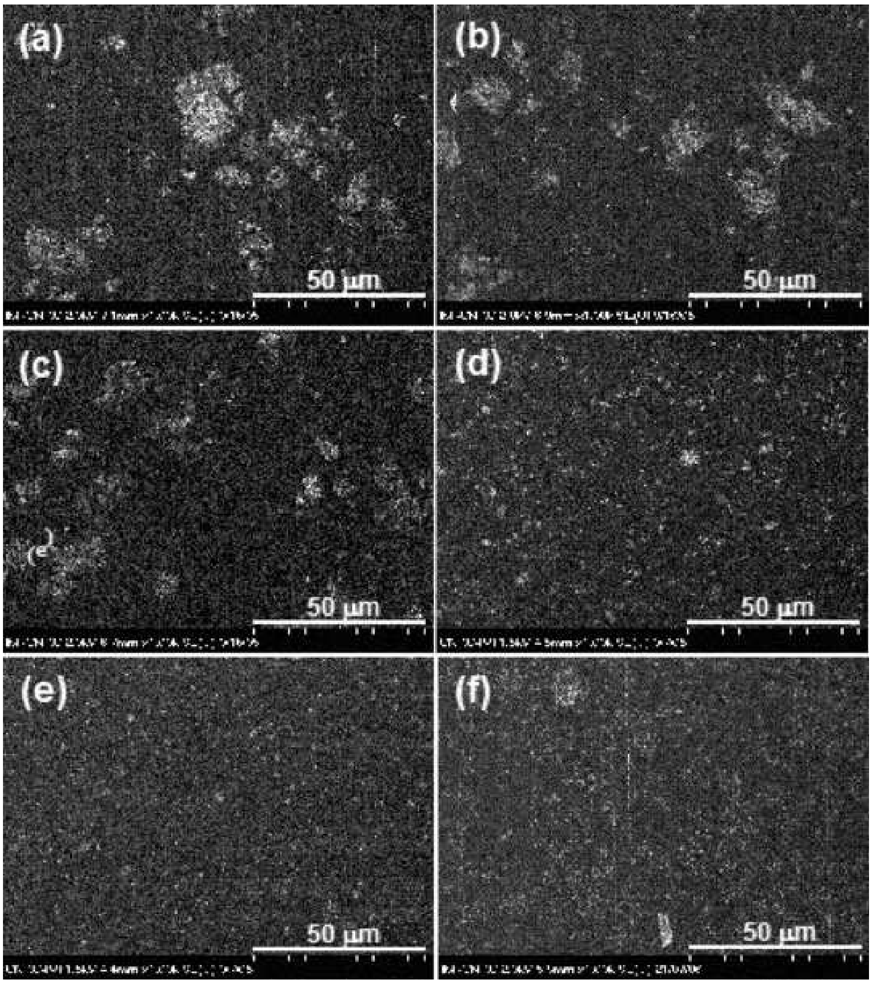

FIG. 5. SEM micrographs of ENCs based on EPON828 and D230 with 2 wt% C30B made by different premixing methods: (a) Rm, (b) Tm, (c) TM, (d) RS, (e) TS, and (f) HP.

better than for the direct mixing method with mechanical stirrer (average size around 3 μm and many large aggregates). During premixing, shear forces broke down the clay aggregates to smaller sizes. This has an indirect beneficial effect on intercalation/exfoliation of clay in epoxy at the curing step, especially at high temperature.

Figure 7 shows TEM photos of nanocomposite prepared by the TS technique (120°C and 24,000 rpm for 1 h) at two different magnifications and of nanocomposite prepared by the HP method. It can be seen in Fig. 7a that the clay has been well dispersed in the epoxy matrix by the TS method. The size of the small aggregates or clay stacks is less than 0.5 μm. At higher magnification (Fig. 7b), the dark lines indicate the individual silicate nanolayers. Although the clay particles were not completely exfoliated into individual platelets, there are many regions showing single, double, and triple clay silicate nanolayers. The HP method also leads to rather good dispersion and intercalation/exfoliation (Fig. 7c), in keeping with the results reported by Liu et al. [10]. However, the quality of dispersion was not as good as that for the sample prepared by the TS method. So far, the results from XRD, SEM, and TEM combine to indicate that with high speed and temperature assistance, one can obtain a fine dispersion at the micro-scale, good distribution of clay, and good intercalation/exfoliation of clay in the epoxy system.

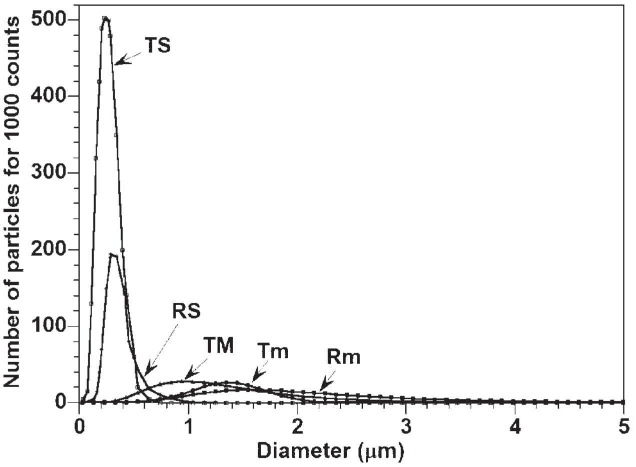

FIG. 6. Clay size distribution for different premixing methods.

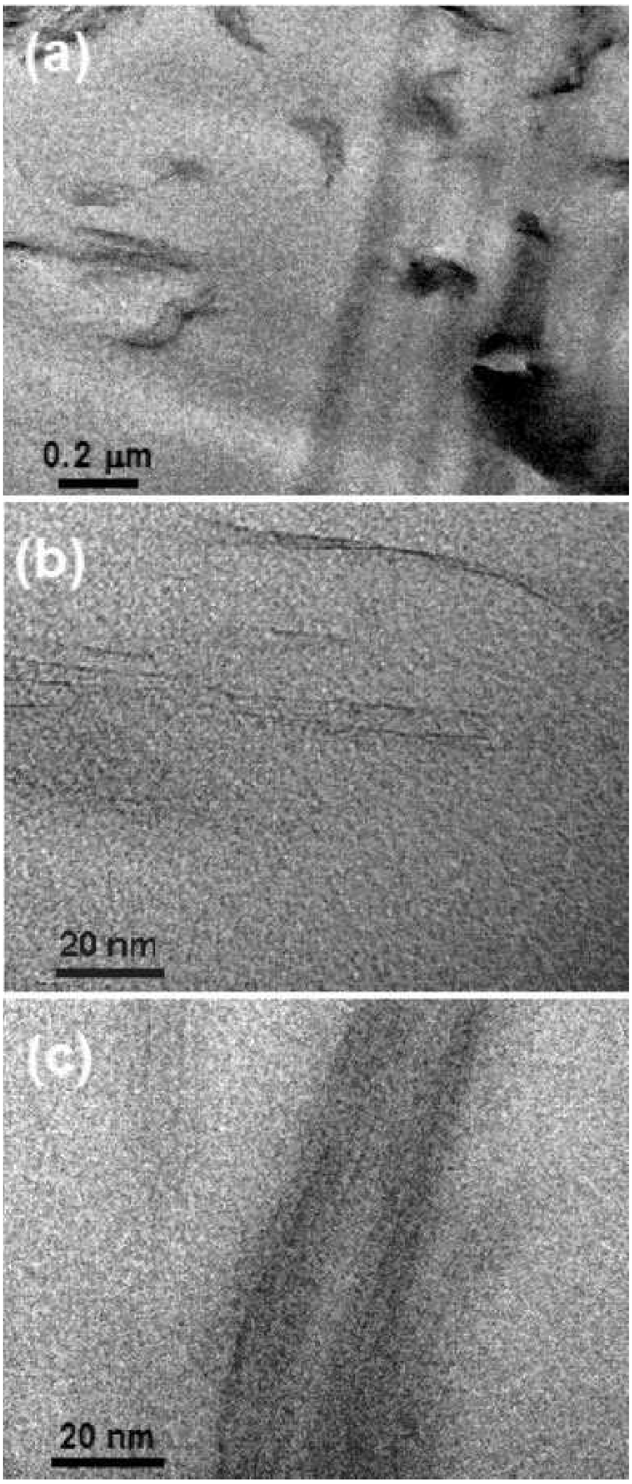

FIG. 7. TEM photos of 2 wt% Cloisite 30B prepared by the TS method (a, b) and by the HP method (c).

Kornmann et al. [3, 4] also obtained an intercalated/exfoliated structure for epoxy nanocomposites, but the clay particles were not uniform and homogeneously distributed in the matrix, and a substantial number of large clay aggregates remained. Good intercalation and good dispersion have also been obtained by using a three-roll mill to disperse the clay nanoparticles, as reported by Yasmin et al. [6]. However, from a practical point of view, the TS method described here has an advantage over the three-roll mill owing to its flexibility, mobility, and low investment cost, while from a technical point of view, it can handle low viscosity systems, which is a limitation of the three-roll mill approach.

CONCLUSIONS

Well dispersed and well intercalated/exfoliated epoxy nanocomposites have been produced with a nonsolvent-assistance method. Although thermodynamic forces play a major role in the intercalation process, our systematic experiments have confirmed that increased shear forces and high temperatures during the premixing of epoxy resin and nanoclay lead to significantly better micro-dispersion of the nanoclay in the form of small well-dispersed tactoids with high surface area. This facilitates the further intercalation/exfoliation that occurs during the subsequent curing step, which is also improved by the use of a higher cure temperature. A well dispersed and well intercalated/exfoliated state of organoclay in ENC can therefore be achieved by using a high-shear high-temperature premixing process (involving, for example, a homogenizer) followed by controlled curing at an elevated temperature. Such a method avoids the use of solvent and is simple and economical to apply in addition to being environmentally friendly.

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