

Evolution of dispersion properties during the delamination of nanoclays

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Abstract

Nanoclay dispersions or “nanocomposites” are achieved by the dispersion of intercalated or exfoliated clay platelets in a polymer melt or monomer prior to polymerisation. Polymer-based clay nanocomposites are usually divided into three categories (Fu & Qutubuddin, 2001, Zhu and Xanthos, 2004; Kotsillkova, 2007):

- In *conventional composites*, the clay acts as a filler material to the polymer.
- *Intercalated nanocomposites* are formed when a small amount of polymer moves into the gallery spacing between the clay platelets to promote the swelling of the clay platelets.
- *Exfoliated nanocomposites* are clay platelets fully delaminated and dispersed in a continuous polymer matrix.

Intercalated and exfoliated nanocomposites have shown significant improvements in terms of product properties even at low clay concentrations. These include:

- thermal stability (Zhu and Wilkie, 2000; Wang et al, 2002),
- barrier properties (Messersmith et al, 1995; Bharadwaj, 2001),
- tensile strength (Chen et al, 2000) and
- increased elongation at break (Ma et al, 2001).

This is because when the filler is uniformly distributed throughout the polymer matrix and both components are made chemically compatible through the modification of the surface properties of the clay mineral, a larger surface area contact is achieved.

The study of the intercalation and exfoliation processes has presented challenges in terms of choosing appropriate experimental techniques to monitor these processes. X-Ray diffraction technique has typically been used to determine the inter-gallery spacing and hence has been found useful in monitoring the intercalation process. There has been limited success in obtaining reliable and meaningful particle size data. Dispersion rheology, on the other hand, has proved to be a promising tool as this is a dispersion property that significantly varies throughout the process. Electron microscopy (TEM, SEM) have provided additional information complementing the above.

In this presentation, selected data obtained during the delamination of two types of nanoclay dispersions will be shown:

- Cloisite 30B in a polyol and
- Cloisite Na⁺ in water.

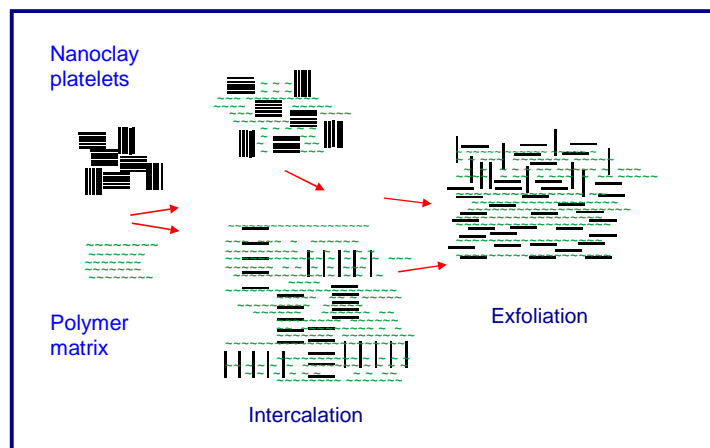
Different mixing protocols have been followed to prepare the nanoclay dispersions:

- stirred tank equipped with a hydrofoil
- stirred tank equipped with a saw tooth impeller and
- ultrasonic disperser.

Some of the rheology results are shown in the Table below for a given concentration of nanoclay dispersion obtained through different mixing protocols. These illustrate how the dispersion rheology changes during the delamination process. Further results will be presented and discussed during the meeting along with the limitations of various techniques used to characterise nanoclay dispersions.

keywords: nanoclay dispersions, delamination, exfoliation, intercalation, dispersion rheology

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Schematic representation of the delamination of nanoclays

Evolution of the rheological properties of 5% Cloisite 30B dispersions during processing obtained with different dispersion methods

Device used	Processing Time (min)	τ_{yHB}	K	n	Viscosity at 300s ⁻¹ (Pa s)	Goodness of fit (%)
Ultrasound 75%	0	17.7	1.3	0.91	0.82	100.0
	30	38.3	2.0	0.87	1.07	100.0
	60	46.1	2.3	0.85	1.15	100.0
	120	47.6	2.4	0.85	1.18	100.0
	180	49.3	2.5	0.84	1.19	100.0
Ultrasound 100%	0	19.3	1.4	0.90	0.90	99.5
	30	24.6	1.7	0.89	0.97	100.0
	60	27.2	1.8	0.88	1.01	100.0
	120	31.2	2.0	0.87	1.08	100.0
	180	31.5	2.0	0.87	1.10	100.0
A310 (single) 200rpm	0	13.5	1.06	0.89	0.63	100.0
	30	25.2	1.32	0.89	0.78	100.0
	60	18.5	1.14	0.90	0.71	100.0
	120	18.9	1.23	0.90	0.76	100.0
	180	17.8	1.25	0.90	0.77	100.0
Sawtooth impeller 1500rpm	0	23.1	1.5	0.88	0.80	99.9
	30	40.3	2.0	0.86	1.01	99.8
	60	47.6	2.3	0.85	1.09	99.8
	120	52.9	2.8	0.82	1.17	100.0
	180	61.4	2.8	0.82	1.21	100.0

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