Effect of Sonication on Mechanical Properties of Polymers and Nanocomposites

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ABSTRACT

This study investigates the influence of sonication on the viscosity and mechanical properties of polymeric nanocomposites. Nanocomposites made with sonication show better mechanical properties than the analogous nanocomposites made with mechanical mixing only.

In this work the comparison of polymer matrix properties with and without sonication as well as nanocomposites produced with and without sonication is performed. Parameters of study are temperature level, time of sonication, concentration of nanoparticles [nanoclay], type of neat polymer (vinyl ester, epoxy), and percentage level of catalyst (for kinetics study). It is shown that the viscosity of polymer matrix is increased after sonication and cooling to the initial temperature (Figure 1). The kinetics of solidification checked with help of viscometry is changed also. Viscometry studies significantly affect the nanoparticles dispersion quality and properties of the nanocomposite. Results indicate that there are shifts in mechanical characteristics of polymer matrix and nanocomposites. Decreases in Young's modulus and strength of polymer matrix are experienced while ultimate strain and energy absorption properties exhibit increases (Table 1). Data tend to confirm the hypothesis that cavitations of the sonication process cause rupture of macromolecules and creation of free radicals which are attached as lateral branches making polymer more grafted and rubber-like.

Keywords: Sonication, Vinyl Ester, Nanoclay, Viscometry

INTRODUCTION

Sonication is the main technology for mixing nanoparticles with polymer matrix. The quality of nanocomposites produced by mixing nanoparticles with liquid resin using mechanical stirring only is significantly less than the quality of nanocomposites, in mixing of which the sonication was used. Sonication provides uniform dispersion of nanoparticles, destroys aggregates of nanoparticles, degassing polymer matrix, etc. However, influence of sonication on polymer matrix itself and its properties during whole technological process is studied insufficiently.

Sonication is a treatment by ultrasonic waves going from a vibrating indenter. In the case of nanoclay particles sonication helps to exfoliate nanoclay particles originally consisting of several platelet shape layers. During sonication, the local temperature and pressure in micro-volumes is increasing by several decimal orders. It can lead to splitting macromolecules, to creation of free radicals, and to change of the structure of macromolecules to more grafted ones.

To achieve good quality and repeatability of properties, sonication during several hours is used in research laboratories. For the industrial applications the decreasing of the time of each operation without significant loss in quality is critical. The aim of this investigation is to estimate how the time of sonication together with other parameters of the process such as temperature and volume concentration of nanoparticles are affected on the mechanical properties of nanocomposites. The method of Design and Analysis of Experiments (DAE) was used for this purpose. Quality of dispersion is stabilized after some time of sonication. However, because some properties of pure matrix are deteriorated during long sonication, it must be some optimum time, which is providing the best mechanical properties of nanocomposites. The search of the optimum time of sonication was one of the goals of the investigation.

EXPERIMENT

Impregnating 1%-6% of nanoclay into the corresponding quantity of vinyl ester, using the sonicator to mix them when the temperature is 45-85◦C, the time is 10-40 min. Add the 0.3% Cobalt Naphthanate 0.04% Dimethylaniline and 1.25% of HI POINT 90 Catalyst into the mixture after finishing the sonication. Stir them then pour into the molds. After cooling down in 30 min, put the molds into oven at 100 °C, 1 hrs for post curing.

Modified classical method of DAE was used. First, the two levels, three factors experiment were used to determine the trends and significance of interactions of two factors. After excluding non-significant factors or interactions, the third level of factors was used. The coding system used was +1 for maximal level and 0 for the minimal one.

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RESULTS

It is shown that the viscosity of polymer matrix is increased after sonication and cooling to the initial temperature (Figure 1). The kinetics of solidification checked with help of viscometry is changed also. NMR and viscometry studies show that ultrasonic mixing results in changes of chemical structure of vinyl ester and epoxy resin of this effort, and significantly affect the nanoparticles dispersion quality and properties of the nanocomposite. Results indicate that there are shifts in mechanical characteristics of polymer matrix and nanocomposites. Decreases in Young's modulus and strength of polymer matrix are experienced while ultimate strain and energy absorption properties exhibit increases (Table 1). Data tend to confirm the hypothesis that cavitations of the sonication process cause rupture of macromolecules and creation of free radicals which are attached as lateral branches making polymer more grafted and rubber-like.

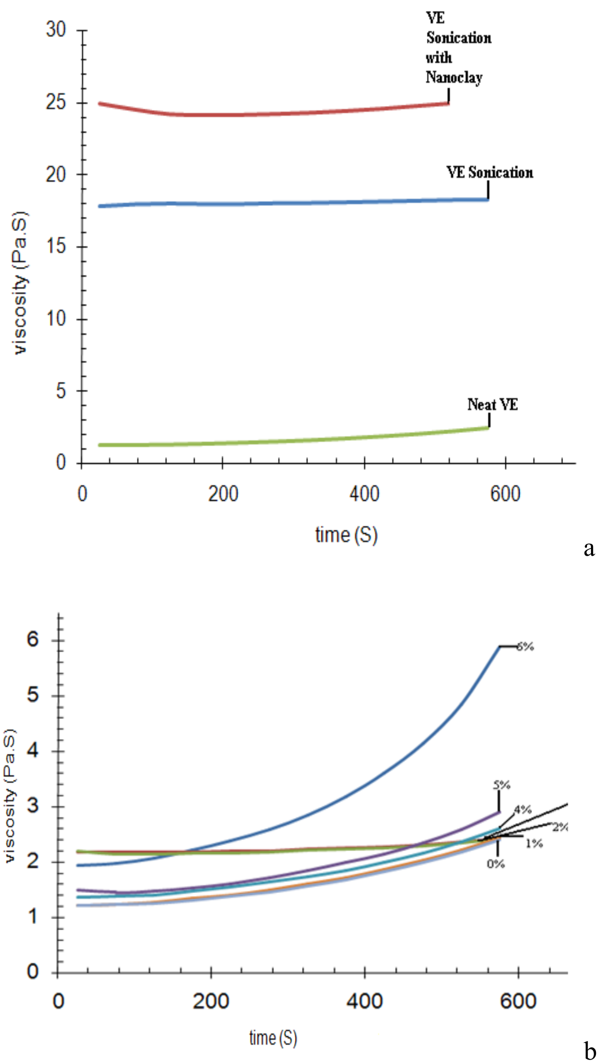

Figure 1. Viscosity change with time without adding the catalist (a) and after adding it (b).

Table I. Flexural Mechanical Properties of Sonicated and Non-sonicated Vinyl Ester Matrix.

Different condition	Young Modulus MPa	Strength MPa	Ultimate Strain %	Ultimate Energy Absorption KJ/m^3
VE Sonicated	2042	86.6	8.75	543
VE Non-sonicated	2122	94.3	7.30	463

DISCUSSION

It is normally set up by building a table using zero (0) signs to show the low levels of the factors and one (1) signs to show the high levels of the factors.

Coded arguments (changed from 0 up to 1):

$$x_1^{(i)} = \frac{t - t_{\min}}{t_{\max} - t_{\min}}$$

Plan of experiments

Table II. Factor level codes

$x_1^{(i)}$	$x_2^{(i)}$	$x_3^{(i)}$	$x_1^{(i)}x_2^{(i)}$	$x_1^{(i)}x_3^{(i)}$	$x_2^{(i)}x_3^{(i)}$	$x_2^{(i)2}$
1	1	1	1	1	1	1
1	1	0	1	0	0	1
1	0	1	0	1	0	1
1	0	0	0	0	0	1
0	1	1	0	0	1	0
0	1	0	0	0	0	0
0	0	1	0	0	0	0
0	0	0	0	0	0	0
0.5	0.5	0.5	0.25	0.25	0.25	0.25

In drawing up experiment, it is common to designate the factor levels by some codes instead of using them written out all the way. Here x_1 corresponds to time, x_2 corresponds to maximal temperature, and x_3 corresponds to nanoclay concentration. The polynomial model with linear terms and double coupled terms was used. Quadratic term with respect to the time was added and the additional experimental study was performed at the center of hyper cube. It allows finding the first approach to the optimal time. Symplex method was used for further optimization

Polynomial coefficients were calculated from the condition of least sum of deviation squares from the experimental results.

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After calculating coefficients by solving the system of 8 linear equations using MathCad, the corresponding matrix was obtained. Example of the solution for energy absorption:

$$\frac{y}{y_0} = 1.579 + 3.276x_1 + 0.195x_2 - 1.509x_3 -$$
$$0.549x_1x_2 + 0.086x_1x_3 + 0.401x_2x_3 - 3.27x_1^2$$

Here $y_0 = 270.2$ KJ/m^3

The coefficient value shows the significance of the corresponding factor.

Coefficient a_{00} shows the overall change of the value studied. If it is about 1, it means that the output value is not changed with change of governing input parameters. If it is below 1 we have increase of value with increase of parameters. If it is over 1 we have decrease of value with increase of parameters. All other coefficients have to be compared with 1. Small coefficients $(a_{ij} << 1)$ mean negligible influence, coefficients comparable with 1 mean significant influence of corresponding factor. It is necessary to mention that the influence of sonication parameters on corresponding properties of postured and non-post cured samples was in many cases opposite. In some cases low influence of a parameter detected in 2-level plan of experiments doesn't mean that it can be neglected but it is result of existing of some minimum or maximum at intermediate level. For example, mechanical properties of nanocomposites are improved with nanoclay concentration increase from 0 to 3-4%, but with further increase of concentration, the mechanical properties dropped down. The results of optimization will be reported on the conference.

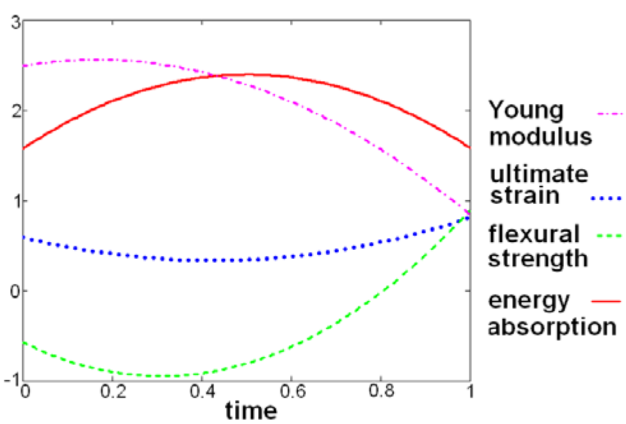

Figure 2: Properties change versus time of sonication.

CONCLUSION

Time of sonication of Nanocomposites used in protective structures designed for maximal energy absorption has to be restricted.

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