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COMPRESSIVE BEHAVIOUR OF ULTRA-SONICATED STARCH / CARBON BLACK / EPOXY COMPOSITES

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ABSTRACT

Fillers are generally used in order to change some basic properties of polymer matrix but the filler particles often tend to aggregate with consequences in terms of the mechanical properties of the final composite. In order to avoid aggregation, many solutions have been proposed and one of them is to use ultra-sounds in various stages of composites formation. In this study, the compressive behaviour of filled epoxy was analysed. As filler, at a 10% volume ratio, a mixture of three parts of starch and one part carbon black was used. The aim of the analysis was to identify the effect of ultra-sonication at different frequencies and for different periods of time on the compressive properties of composites.

KEYWORDS: compressive behaviour, polymer, ultra-sonic treatment, epoxy resin

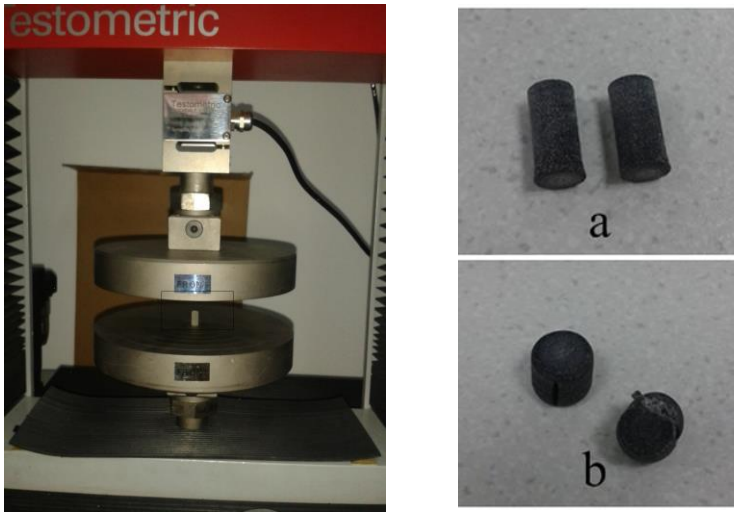
1. Introduction

Among the thermoset materials, epoxy resins show special chemical characteristics such as: absence of secondary products or volatiles during curing reactions, low shrinkage up on curing, curing over a wide temperature range and the control of degree of cross-linking. Depending on the chemical structure of the curing agents and curing conditions, the properties of cured epoxy resins will vary [1]. Ultra-sonication during polymerization is a very efficient method to increase the dispersion of filler particles in the pre-polymer mixture, in the case of thermoset polymers. The effect of ultra-sonication is also observable through some changes which occur in homogeneity, viscosity, relaxation time and hardening of pre-polymer gels [2]. To get superior properties of a polymer, one usual way is to modify it by placing another phase, for instance a nano-sized one, into the polymer volume [3]. Such materials should provide unique mechanical and thermal properties combined with low specific weight and high wear resistance in order to ensure safety and economic efficiency [4]. Carbon nanotubes, carbon black [5, 6], nano-metals, talc, clays, starch [7, 8] and nano-ceramics are among the most commonly used fillers. Carbon black [9] and starch are used to enhance the properties of epoxy composites compared to the unfilled ones – especially regarding tribological [10] and electrical behaviour [11]. The technique of modifying the polymer by adding certain powders is not new at all but is challenged by some difficulties when the used particles are getting smaller and smaller [12]. The properties of such a modified polymer do not depend only on powders properties, but also on the dispersion of particles into the polymer volume. The main goal is to obtain uniform dispersion avoiding clusters formation as clusters represent structural defects from the polymer network point of view [13]. The use of ultra-sounds, exposure of pre-polymer mixture might solve the uniform distribution problem. Under the action of ultra-sounds there is a change in homogenization, viscosity, relaxation time of pre-polymer mixture and it is noticed a strengthening effect on the formed polymer [14]. Ultra-sound has found numerous uses in widely different fields of application. Recently, attention has turned to multiphase systems where attempts have been made to relate the ultra-sonic velocity with the concentration and the degree of dispersion of two-phase systems [15]. In this paper the effect of ultra-sonication on the compressive properties of carbon black and starch filled epoxy resin composites was

investigated, as starch is meant to avoid the aggregation of carbon-black particles.

2. Experimental method

For this study, compressive tests were performed on modified epoxy composites. The matrix consisted of EPIPHEN RE4020 – DE4020 epoxy system that was modified with a mixture of three parts of starch and one part of carbon black. A composite material is generally composed of two or more materials with distinct properties but through their bond they can provide a single composite material with unique properties [16]. This is true, however, if both constituents are present in a reasonable proportion of at least 5% and various properties [17]. The powder mixture was used to obtain a 10% volume ratio of modifying agent in the epoxy matrix - based on other studies [18]. The powder was mechanically dispersed into the required quantity of resin and the mixture was exposed from one to five minutes to the ultra-sonic flux generated by an air-generator at different frequencies. After this step, the required quantity of hardener was added and the mixture was mechanically mixed at 45 ºC for 15 minutes - in order to maintain the low viscosity of the mixture during the gel phase and afterwards poured into moulds. The 15-minute time of mixing was established after trial and error tests that confirmed the fact that during this step the viscosity of the mixture remained low so that it could be easily poured into the moulds and could allow for the elimination of gaseous products. After this step, all the samples were exposed to ultra-sonic influence at a distance of 1 m from the ultra-sound air-generator. Samples extraction was performed after 24 hours and was followed by a recommended thermal treatment. Compressive tests were carried out at room temperature with M350-5AT testing machine from Testometric (Fig. 1). The compression tests were performed at a speed of 5 mm/min. The compressive specimens were 5 mm in height and 10 mm in diameter. Five specimens of each material were tested. The testing method, the sample dimensions and the test parameters were set according to ASTM-D-3410-87 [19, 20].



I. II.

Fig. 1. *I - M350-5AT compressive testing machine from Testometric; II - compressive specimens for testing: a) before the compression test; b) after the compression test*

3. Results

The compression Young Modulus and the Strain at Yield of epoxy resin are plotted as a function of ultra-sonic exposure time (0 to 5 minutes).

Regarding the compression Young modulus, it seems (Fig. 2-5) – but this assumption has to be verified – that the exposure at 24 kHz and 42 kHz leads to an improvement in compression elasticity of the polymer – on the x-axis during the epoxy resin ultra-sound exposure. The results are strongly influenced by the local conditions at the time of forming and by ultra-sonication. However, the results in the case of the 26 kHz frequency point out that ultra-sonic exposure has no effect on the compressive elastic modulus, excepting the use of the four-minute ultra-sound exposure whose effect is destructive.

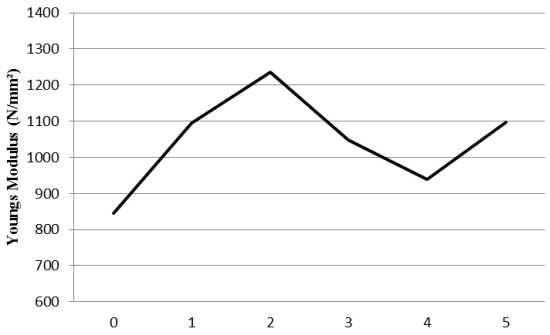


Fig. 2. *Compression Young Modulus of 24 kHz ultra-sonicated epoxy*

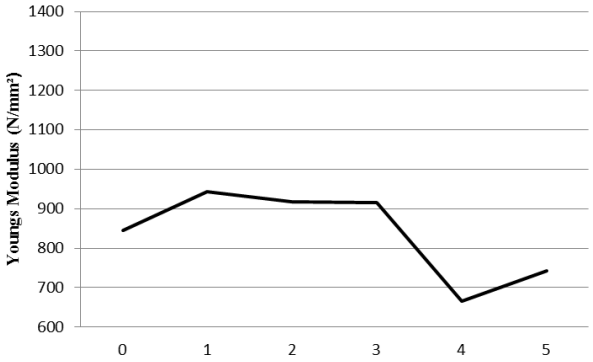


Fig. 3. *Compression Young Modulus of 26 kHz ultra-sonicated epoxy*

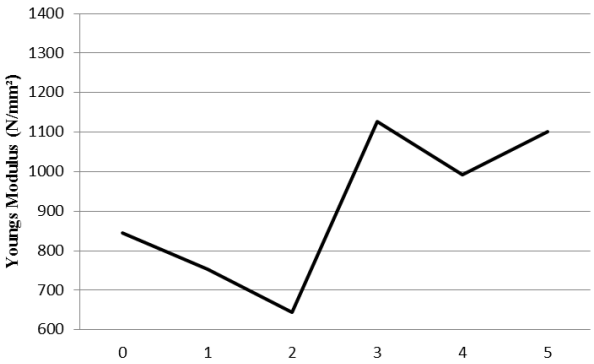


Fig. 4. *Compression Young Modulus of 30 kHz ultra-sonicated epoxy*

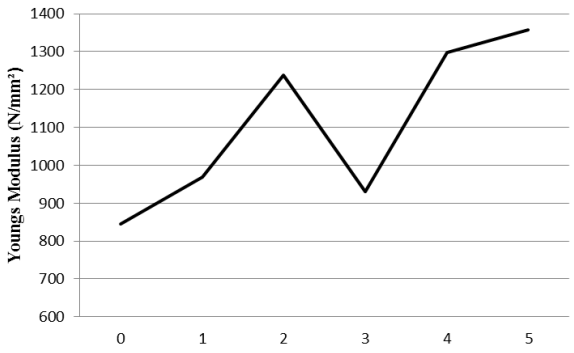


Fig. 5. *Compression Young Modulus of 42 kHz ultra-sonicated epoxy*

At the two higher frequencies, it seems that the ultra-sound exposure time is important since the three-minute ultra-sound exposure time (30 kHz) and, respectively, the two-minute ultra-sound exposure time (42 kHz) lead to better results regarding the materials elasticity.

Marking a change in the polymer mechanical response (from elastic to plastic), the strain at yield is an important parameter used to describe polymers behaviour. The values of this parameter do not show dramatic changes excepting the cases of the one minute ultra-sound exposure time at 30 kHz and the three-minute ultra-sound exposure time at 42 kHz but, once again, these results might be influenced by the environmental conditions.

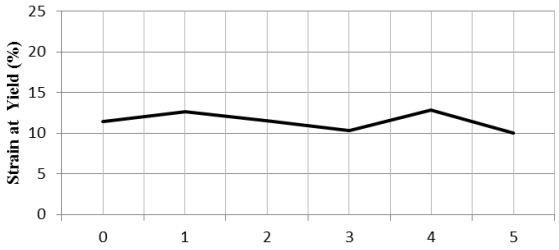


Fig. 6. *Strain at Yield of 24 kHz ultra-sonicated epoxy*

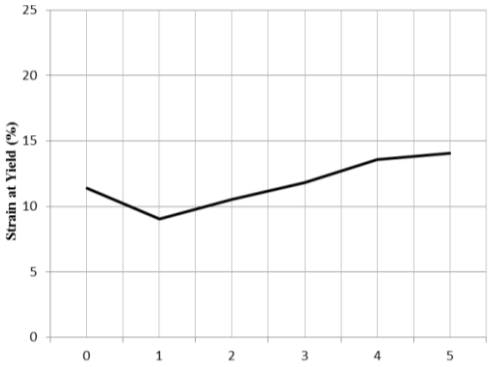


Fig. 7. *Strain at Yield of 26 kHz ultra-sonicated epoxy*

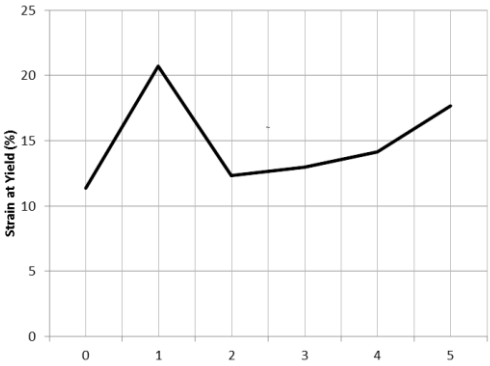


Fig. 8. *Strain at Yield of 30 kHz ultra-sonicated epoxy*

Because of the interaction between ultra-sounds and polymer matrix, it was expected a decrease of compressive parameters values but, analysing the graphs above, the behaviour is far from expectations. The strain at yield (Fig. 6-9) is practically unchanged compared to the standard probe (the sample of epoxy resin without any ultra-sound exposure) taking into account the unavoidable gaseous intrusions inside the samples. The strain at yield is independent both from the ultra-sound frequency and from the ultra-sound energy. It is a fact that the use of ultra-sound exposure leads to better dispersions of powders than the dispersion that had been used to modify the basic properties of the epoxy resin.

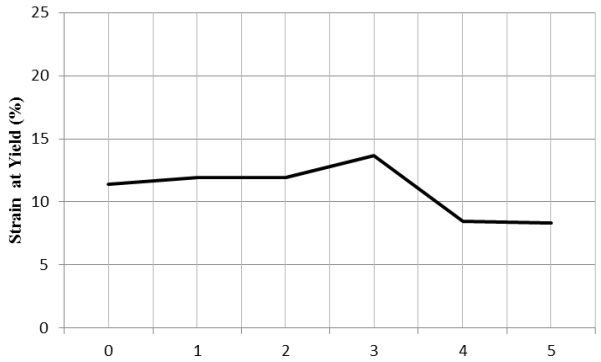


Fig. 9. *Strain at Yield of 42 kHz ultra-sonicated epoxy*

4. Conclusions

These experimental results provided a database on the compressive behaviour of ultra-sonicated starch / carbon black / epoxy composites. A comprehensive and reproducible set of data has been obtained to characterise the mechanical behaviour of the ultra-sonicated starch/carbon black / epoxy composites. These results are particularly significant from the compression point of view, because it seems that ultra-sonication does not induce severe modifications. However tensile and bending tests are necessary in order to complete the picture of the influence of this type of treatment on the mechanical properties of the epoxy resin. One issue that has to be dealt with during further studies is the identification of a method to avoid the gaseous intrusions taking into account the fact that they are responsible for non-linear responses at external loadings.

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