Effects of organic compounds and nickel nitrate on friction coefficient of thermosetting

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Abstract. The principal aim of this study is to point out the effect of organic compounds mixed with nickel nitrate in determined weight ratios on friction coefficient of two distinct types of epoxy systems. The two used organic compounds are starch and agar. Ultrasound exposure is typically used to properly prepare the powder modified composite materials namely to obtain a uniform dispersion of nickel nitrate in to the epoxy. Friction coefficient behaviour of composite materials is essential for successful applications among various modern industries of these challenging materials. Friction coefficient of the modified epoxy matrix was accurately measured using pin-on-disc geometry with TRM 1000 tribometer from Wazau at the Research and Development Center for Thermoset Matrix Composites from Dunărea de Jos University of Galați.

1. Introduction

Epoxys are handled in a variety of applications due to their excellent properties such as thermal stability, mechanical response, electrical, corrosion resistance, and good dimensional stability [1]. It has been established in recent years that polymer based composites reinforced with a small percentages of strong fillers can significantly improve the mechanical, thermal and barrier properties of the pure polymer matrix [2]. The use of organic or inorganic filler has become popular in polymeric systems [3]. In fact, it was verified experimentally by several research groups all over the world, that particles of metallic or inorganic type, prove the ability to reinforce effectively thermoplastic and also thermosetting polymer matrices [4]. Filling the polymer with various substances in different forms (powders, whiskers, and flakes) was, at the beginning, a way to reduce the amount of polymer used to form a structure but this method became a way to modify the basic properties of the polymer leading to improvements regarding thermal, electro-magnetic or tribological properties [5], [6]. An improvement in a property arises when a uniform dispersion of these fillers into the epoxy resin is
obtained. It has been shown, that a considerable improvement of the mechanical and tribological properties can already be achieved at very low filler volume content, somewhere in the range of 1–5 vol.% [7].

The dispersion of starch and agar together with nickel nitrate into epoxy resin is the biggest challenge. The commonly used methods to disperse organic or inorganic filler in a polymer matrix is by ultrasonication. Better mixing between these filler and the polymer matrix can provide higher strength and stiffness whereas poor mixing is seen to decrease those properties. There are a large number of industrial processes that employ sonication technique as an energy source for the generation of the fine emulsions and dispersions [8]. Several studies about the application of ultrasound in composite materials preparation found that ultrasound may assist in better filler dispersion [9].

Friction is a very common phenomenon in daily life and industry, which is governed by the processes occurring in the thin surfaces layers of bodies in moving contact [10]. There are many polymers that have not yet studied in terms of tribology. Due to the importance of these properties which is equal to the mechanical properties, it is highly recommended for comprehensive study for such important materials [11]. The most popular and also practically confirmed as the best tribological combination is steel-on-polymer frictional tribosystem [12] therefore the tests were done with this system. Most of the reported works mentioned that there is film transfer generated during the thermoset sliding against metal surfaces [13]. The transfer film formed on a non-polymer counterface is governed by the counterface material and roughness, and of course the sliding conditions [14]. The transfer of material from polymer to metal surface initiates because of adhesion between the two materials which during sliding contributes to shear in the subsurface region of the contact [15]. Very wide possibilities to modify polymeric materials by fillers, and many other additives give very good perspectives to find polymeric composites that show excellent tribological properties as matched with non-polymer component [16].

The aim of this study is to point out the effect of organic compounds and nickel nitrate used as modifying agents, subjected to ultrasound exposure, for epoxy resins and to explore their role on friction coefficient of the composite materials. Friction coefficient of the composite materials was accurately measured using pin-on-disc geometry with TRM 1000 tribometer from Wazau.

2. Experimental section

To obtain these composite materials, organic agents such as starch with agar and nickel nitrate were used. The epoxy resins used are those of type HT and C with the hardener for each resin. The determination of the amount of material required was done according to Table 1.

| Material | Resin | Hardener | Nickel nitrate | Starch | Agar | Ultrasonication |
|----------|-------|----------|----------------|--------|------|-----------------|
| H0       | 100g  | 48g      | 5g             | 1g     | 0g   | -               |
| H1       | 100g  | 48g      | 5g             | 0g     | 1g   | -               |
| H2       | 100g  | 48g      | 5g             | 1g     | 0g   | -               |
| H3       | 100g  | 48g      | 5g             | 0g     | 1g   | +               |
| H4       | 100g  | 48g      | 5g             | 1g     | 0g   | +               |
| C0       | 100g  | 60g      | 5g             | 0g     | 1g   | -               |
| C1       | 100g  | 60g      | 5g             | 1g     | 0g   | -               |
| C2       | 100g  | 60g      | 5g             | 0g     | 1g   | -               |
| C3       | 100g  | 60g      | 5g             | 1g     | 0g   | +               |
| C4       | 100g  | 60g      | 5g             | 0g     | 1g   | +               |
Ultrasonication was performed using an ultrasonic generator UP 100H from Hielscher (Fig. 1). The ultrasonic processor frequency is 30kHz. The diameter of the horn is 3mm. The time for ultrasonic treatment was for 2 hours.

![Figure 1. Ultrasonic Processor UP100H.](image)

The friction coefficients behaviour were investigated using a TRM 1000 tribometer from Wazau (Fig. 2). Friction coefficient studies of epoxy systems against steel disc are usually carried out at different load, sliding velocity, and distance conditions. R1, R2 and R3 represents the friction coefficient regimes used for composite materials tests. Tribological tests were done 23°C for 1000 m distance. The tests were performed at three different regimes with three loads and three sliding speeds in order to respect the same product load-speed (15N and 1m/s = R1; 20N and 0.75m/s = R2; 25N and 0.6 m/s = R3).

The cylindrical pin specimens of 8 mm diameter and 30 mm length were tested on pin-on-disk geometry, with the pin made of analyzed material and steel disk. The calculations of friction coefficient were carried out using Tribo Control Wazau software.

![Figure 2. TRM 1000 Wazau tribometer.](image)

3. Results and discussion

Evolution of friction coefficient during friction for composite materials based on epoxy resin HT are shown in Figures 4, 5 and 6. Each graph represents a regimen R1, R2 and R3 in which five curves are represented, corresponding to the five different materials: H0, H1, H2, H3 and H4.
Figure 3. Friction coefficient of HT composites for R1 regime.

Figure 4. Friction coefficient of HT composites for R2 regime.
In Figures 3 and 4, H1, H2 and H4 materials exhibit lower friction coefficients. The materials represented in Figure 5 behave almost the same as the control material H0. The exception is the H3 material which in Figure 3 has higher coefficients of friction and in Figure 5 it behaves almost the same as the control material H0.

Evolution of friction coefficient during friction for composite materials based on epoxy resin C are shown in Figures 6, 7 and 8. Each graph represents one of the R1, R2 and R3 regimes in which five curves corresponding to the five different materials are represented: C0, C1, C2, C3 and C4.

The materials represented in Figures 6, 7 and 8 behave almost the same as the C0 control material except C3 and C4 materials exhibiting lower friction coefficients.
To analyze the mass losses following the tribometric tests, the material was weighed before the test and after the test had been carried out. Analyzing mass losses, the results shown in Figures 9 and 10 were obtained.
From Figures 9 and 10 it can be seen that ultrasound treated materials lead to higher mass losses in tribometric tests. Regarding the test regimes, one cannot establish a rule after which mass loss occurred.

4. Conclusions
In this paper we analyzed the evolution of the friction coefficient and the mass loss to identify the effect of the presence of additives (agar, starch and nickel nitrate) in the epoxy matrix and exposure to ultrasonic irradiation on materials of the same composition. As a conclusion of the behavior of composite materials modified with additives (agar, starch and nickel nitrate) following tribological tests, we can state both the quantity and the type of the modifying agent have led to visible changes for some of the composite materials.

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