Nanometric SiC influence on tribological properties of phenolic composite materials

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Abstract: *This paper presents an experimental study of the influence of nano metric silicon carbide in the composition of phenolic composites on the coefficient of friction. The paper is divided into three distinct parts investigating from a tribological point of view three different types of composite materials based on phenolic resin with three concentrations of nSiC (0.5; 1 and 2% by mass). In the first part, a comparative study of the behavior of phenolic resin was performed, representing the basis for the development of composite materials. In the second part, a study was performed on laminated materials reinforced with two-dimensional fabrics (glass fiber and carbon fiber, respectively). The last part studied two types of ablative phenolic materials based on micronic cork, on one hand, and on carbon felt on the other hand.*

Key Words: *Composite materials, phenolic matrix, friction coefficient, nSiC filler powder*

1. INTRODUCTION

As advanced research in conjunction with industry leads humanity further and further into outer space, technological needs become more complex and interdependent. In recent years, the interest shown by aerospace researchers in nanocomposite materials has increased more and more, due to the excellent properties (mechanical, thermal, electrical, tribological, etc.) that nanofillers can induce in classical materials.

The special conditions imposed by the aerospace environment lead to the need to develop systems of complex materials based on a wide range of materials, in order to meet the highest requirements in this field. The combination of materials from various classes is necessary to cover the multitude of requirements specific to the conditions, so in order to be able to have high performances from many points of view (mechanical, thermal, tribological, mass, etc.) the range of materials extends from polymers of thermo-reactive or thermos-plastic resins type, cork lignin-cellulose granules, carbon fiber reinforcements in various forms, up to inorganic compounds with mechanical, thermal and tribological resistance, at extreme temperatures.

An important idea of the study was the intention to introduce inorganic compounds into such systems. The main problem of ceramic matrix composites (CMC) is the high specific gravity, which is an eliminatory requirement in aeronautical and aerospace applications. However, the thermal and tribological performances of this type of compounds are extremely high, which makes them suitable for applications in extreme environments. As a solution, attention has been directed to nanometric particles, which can be used as nanofillers in polymer-based materials and various preforms (which have medium thermal properties, but high mechanical and tribological properties), and due to their size can be added in particularly small percentages (less than 5% by mass), which do not lead to significant weight gain, but allow them to exercise their special advantages [1]. Due to the high potential shown in structural applications in the aerospace industry, offered by excellent thermal properties, high mechanical strength and modulus of elasticity at high temperatures and in extreme environments, low density in the series of ceramic compounds, high hardness, abrasive properties and good oxidation resistance [2], nanometric silicon carbide (nSiC) was the optimal candidate proposed as a nanofiller in the systems developed in the study. Most oxidation protection systems are based on SiC deposition on fibers, modification of the matrix with SiC or B_4C, modified layers with SiC, SiC coatings deposited by CVD and silica or borosilica [3].

Studies presented in literature show that the improvement of the mechanical and especially tribological properties was also obtained by adding antioxidant fillers such as MoSi2 [4], ZrC by vacuum infiltration [5], ceramic whiskers [6], or even the synthesis of Ti3SiC2-SiC in the carbon matrix by infiltration of a TiC suspension followed by liquid silicone infiltration [7].

In high-tech and aerospace applications, there are a multitude of properties in specific conditions that need to be evaluated in order to build a realistic image in laboratory scale of the mechanisms that are involved in real life events. Tribological properties are only one of the many properties that a material needs to be evaluated for. The determination of the friction coefficient of materials is useful for evaluating their tribological potential used for applications in friction or anti-friction systems [1]. The present paper presents the evaluation of tribological properties in terms of friction coefficient of nanofilled phenolic resin matrix based materials. The paper presents three classes of composites with phenolic matrix, each of them with three different nSiC content added: simple polymerized phenolic resin, carbon and glass fiber reinforced phenolic resin, cork and carbon felt phenolic ablative materials.

2. EXPERIMENTAL

2.1 Materials

The studied composite materials were based on phenolic resin of ISOPHEN 215 SM 57% type supplied by ISOVOLTA S.A. Bucharest, with a density of 1,135 g/cm^{-3}. The nanofiller used was β spherical nanometric silicon carbide purchased from Nanostructured & Amorphous Materials Inc., USA, with the following characteristics: purity 97.5%, average particle size of 45-55 nm, specific surface area of 34-40 m^2/g and 3.22 g/cm^3 true density. As reinforcement materials, two-dimensional fabrics were used (glass fibers type E and carbon fibers (CARP / T 193, produced by Chemie Craft, France) for laminar composites, and for ablative composites were used micron-sized cork granules and carbon fiber felt preforms, Sigratherm GFA10 purchased from SGL Group - The Carbon Company, GmBH, respectively, with a thickness of 11.5 mm, a surface weight of 1000 g/m^2 and a temperature maximum application of 2000°C in vacuum or inert gas.

2.2 Obtaining method

The paper is divided into three distinct parts that investigate from a tribological point of view three different types of composite materials based on phenolic resin with three concentrations of nSiC (0.5; 1 and 2% by mass). The process of obtaining composite materials is different for each part depending on the material that is developed. Thus, in the case of the materials developed in the first part, PR / nSiC, the nanoparticles were dispersed in the liquid phenolic resin by ultrasonication and the crosslinking was performed at temperature and pressure [1, 8]. In the second part, laminated composites were obtained, in which 5 layers of two-dimensional fabric (glass fiber and carbon fiber) were mechanically impregnated and then processed under temperature and pressure using a heated plates press [1, 9]. In the last stage, ablative type materials were developed. The reinforcement materials (based on carbon felt and micronic cork granules, respectively) were impregnated with phenolic resin and kept at a pressure of 2 bar for 24 hours for deep impregnation, followed by crosslinking under temperature and pressure. [1, 10, 11].

2.3 Tribological properties testing

Tribological properties were evaluated in terms of friction coefficient. The friction coefficient was determined using the CETR UMT 3 (Universal Macro Materials Tester) equipment provided with the roller-shoe type test module, being calculated by the equipment software. The tribometer records in real time the value of the friction force, the movement in the direction of force application and offers the user the possibility to view the parameters on the computer monitor interconnected to the equipment, the friction coefficient being calculated using the following equation:

$$CoF = \frac{F_X}{F_Z} \quad (1)$$

CoF= friction coefficient,
F_x= friction force, N
F_z= compression force, N

In Fig. 1 it is presented the simplified diagram of the UMT installation, equipped with the roller-shoe module. The principle of the test is to insert the specimen into a sample holder (with the bottom open so that the specimen comes into contact with the roller below) and apply a set force (for a set period of time). When the preset force is reached, the steel roller at the bottom of the holder begins to rotate at a preset speed. The roller used is made of steel type material “Rul” with a diameter of 35 mm and a width of 10 mm.

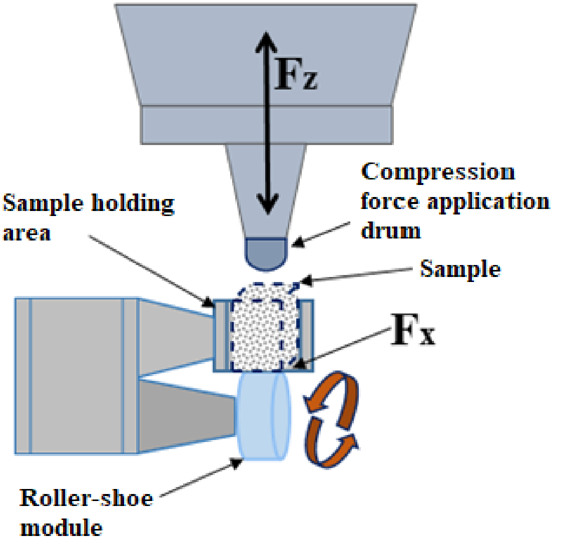
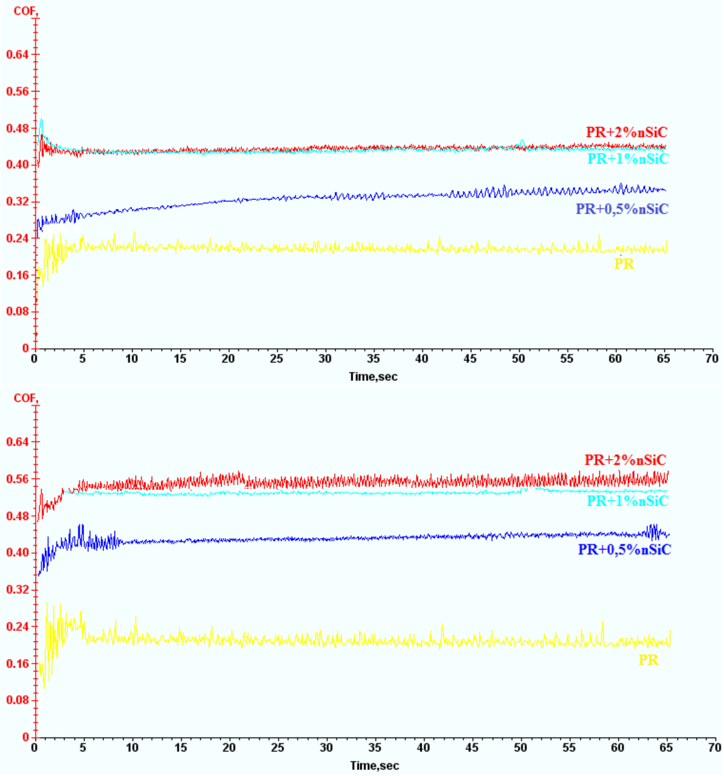


Fig. 1 Diagram of the tribological installation with the roller-shoe mode [1]

Tribological testing consisted of measuring the coefficient of friction in a dry environment at two rotational speeds, the results showing the same variation depending on the percentage of nSiC as in the case of mechanical tests [1, 8-11] Fig. 1.
Tribological tests were performed using a pressure load of 10 N, the test period being 60 sec. Two values for the test speed were used, 1000 and 1500 rpm (1.75 and 2.62m/s, respectively). A set of 3 specimens was used for each sample, the final coefficient of friction being calculated as the average of the 3 values obtained for the 3 specimens or the average of two similar values (if one of them left the field).
Qualitatively, materials with friction coefficients with maximum values of 0.2-0.25 are included and can be used as anti-friction materials (sliding bearings). In the case of used materials that have friction coefficients with values higher than 0.25, they are considered friction materials (clutches, braking systems).
The paper is divided into three distinct parts that investigate from a tribological point of view three different types of composite materials based on phenolic resin with three concentrations of nSiC (0.5; 1 and 2% by mass).

2.4 Results and discussions

The paper is divided into three distinct parts that characterize from a tribological point of view three types of distinct classes of materials to determine the coefficient of friction of each class of materials after modification with different mass concentrations of nSiC, to observe the behavior following the addition.
In the first part, a comparative study of the behavior of phenolic resin was performed (fig. 2), representing the basis for the development of composite materials.



Friction coefficient of PR/nSiC measured at 1000 rpm

Friction coefficient of PR/nSiC measured at 1500 rpm

Fig. 2 Friction coefficient of PR/nSiC

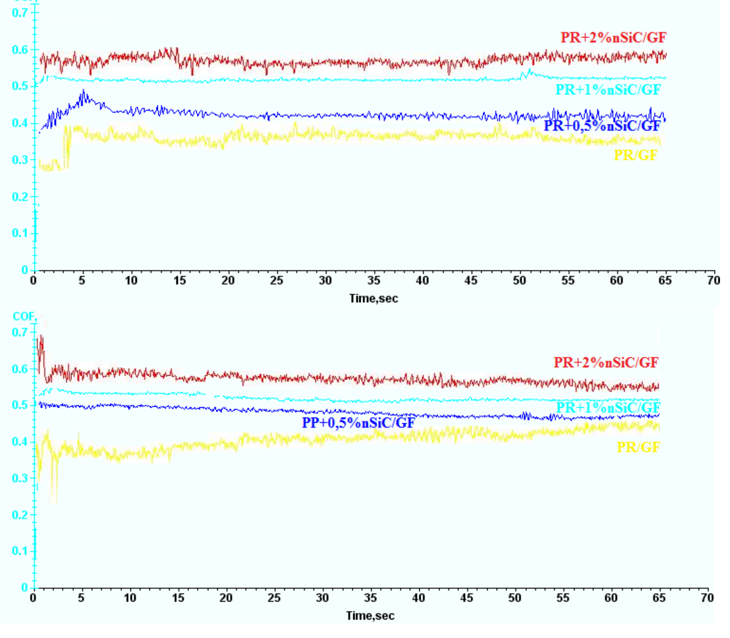
Composite materials were developed based on only simple phenolic resin and added with different mass concentrations of nSiC to illustrate the influence of this filler from a tribological point of view on the polymer matrix that will be used to obtain the composite materials from the next steps.

Table 1. The friction coefficient of PR/nSiC measured at s 1000 and 1500 rpm speed [1, 8]

Material	Friction coefficient (1000 rpm)	Friction coefficient (1500 rpm)
PR	0.2341	0.2348
PR + 0,5% nSiC	0.3996	0.4484
PR + 1% nSiC	0.4664	0.5206
PR + 2% nSiC	0.4538	0.5137

In both test speed values, due to the known abrasive properties of nSiC, materials with the addition of nanoparticles have higher friction coefficient values than the control sample, which increases with increasing percentage of nSiC in the matrix.

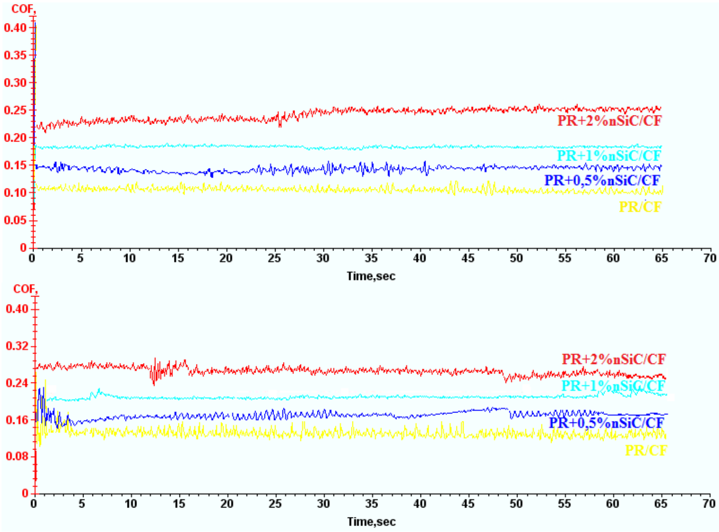
The maximum is also presented in this case by the PR + 1% nSiC samples, the difference being extremely small compared to the PR + 2% nSiC. At 1000 rpm, the coefficient of friction of the samples with 0.5, 1 and 2% nSiC showed an increase of 71, 99 and 94% compared to the control sample. At 1500 rpm, the coefficients of friction for the 3 samples (with 0.5, 1 and 2% nSiC) were 91, 122 and 119% higher than the control sample. The slight decrease in the coefficients of friction in the case of PR + 2% nSiC compared to PR + 1% nSiC may be due to the uneven areas visualized in this sample, which may lead to surfaces with a higher percentage of resin, subject to friction at various speeds, which decreases the resulting final values of the coefficients of friction [8]. In the second part, a study was performed on laminated materials reinforced with various two-dimensional fabrics (glass fiber and carbon fiber respectively). At this stage, two groups of materials were studied that differ in the reinforcement material: on the one hand, glass fiber (fig. 3) on the other hand, carbon fiber (fig. 4). Very different values of the friction coefficient between these two groups of materials were highlighted due to the different nature of the two types of fabrics.



Friction coefficient of PR/nSiC/GF measured at 1000 rpm

Friction coefficient of PR/nSiC/GF measured at 1500 rpm

Fig. 3 Friction coefficient of PR/nSiC/GF



Friction coefficient of PR/nSiC/CF measured at 1000 rpm

Friction coefficient of PR/nSiC/CF measured at 1500 rpm

Fig. 4 Friction coefficient of PR/nSiC/CF

As a framework, carbon fiber is used in anti-friction material systems [12, 13], while fiberglass is used in the production of friction material systems [14, 15].

It is known that by their nature glass fibers are also materials used in applications that involve friction.

Table 2. The friction coefficient of PR/nSiC/GF measured at 1000 and 1500 rpm speed [1, 9]

Material	Friction coefficient (1000 rpm)	Friction coefficient (1500 rpm)
PR/ GF	0.3934	0.4302
PR + 0,5% nSiC/GF	0.4484	0.4896
PR + 1% nSiC/GF	0.5374	0.5531
PR + 2% nSiC/GF	0.5749	0.6037

Therefore, the addition of nSiC in the matrix of PR/ GF composites generates increases in the coefficient of friction of about 15% in the case of 0.5% nSiC, 30-40% in the case of those with 1% nSiC and 40-45% in the case of those with 2% nSiC. It is important to note that the values of the coefficient of friction presented by PR/nSiC/GF materials fall within the range of high coefficients of friction, which recommends these materials for applications where abrasive properties are required (such as brake pads in automotive applications. or aeronautics).

As in the case of the group of materials based on glass fibers, the carbon fiber laminated composites were tribologically tested using the two rotational speeds.

Table 3. The friction coefficient of PR/nSiC/CF measured at 1000 and 1500 rpm speed [1, 9]

Material	Friction coefficient (1000 rpm)	Friction coefficient (1500 rpm)
PR/ CF	0.1301	0.1620
PR + 0,5% nSiC/ CF	0.1515	0.1760
PR + 1% nSiC/CF	0.1923	0.2136
PR + 2% nSiC/CF	0.2505	0.2694

It is observed in table 3 that as in the case of layered fibers based on fiber, the values of the friction coefficient increase with the increase of the percentage of nSiC.

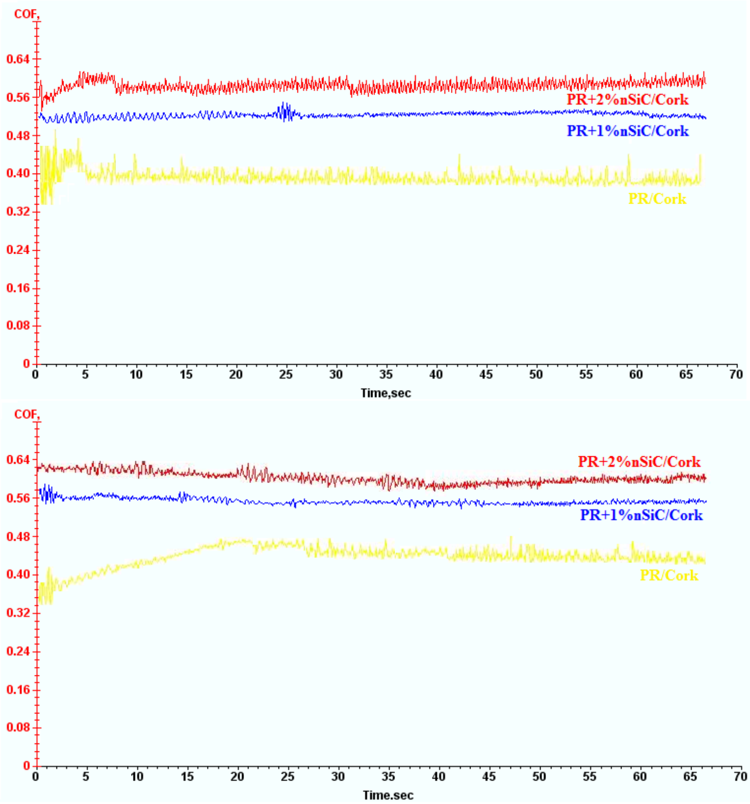
The use of the different contents increases the coefficient of friction by 10-16% for samples with 0.5% nSiC; 30-50% for samples with 1% nSiC and 70-90% for those with 2% nSiC.

In the last part, two groups of ablative phenolic materials were studied that differed through the reinforcement material (based on micronic cork on one hand and carbon felt on the other hand).
As expected, in this case as well, following the tribological testing, the ablative composite materials revealed the same variation as in the case of the composite materials studied in the previous stages, the values of friction coefficients increasing with increasing percentage of nSiC added to the matrix in both cases of the test speed values used.

Table 4. The friction coefficient of PR/nSiC/cork measured at 1000 and 1500 rpm speed [1, 11]

Material	Friction coefficient (1000 rpm)	Friction coefficient (1500 rpm)
PR/cork	0.422	0.45
PR + 1% nSiC/cork	0.528	0.577
PR + 2% nSiC/cork	0.586	0.596

Thus, in the case of the set of composite materials based on cork granules (fig. 5), at a speed of 1000 rpm, the addition of 1 and 2% nSiC, respectively, generated an increase in the friction coefficient by 25 and 39%, respectively and at a speed of 1500 rpm, the increases were 28 and 33%, respectively, compared to the control.

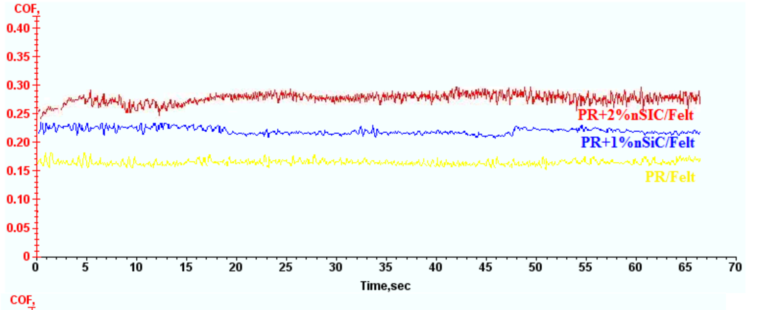


Friction coefficient of PR/nSiC/Cork measured at 1000 rpm

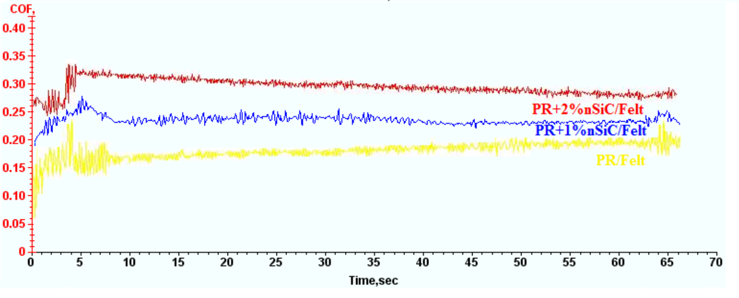
Friction coefficient of PR/nSiC/Cork measured at 1500 rpm

Fig. 5 Friction coefficient of PR/nSiC/Cork

The results obtained show that PR/nSiC/cork materials could be suitable as friction materials (abrasive materials).
In the case of the set of materials based on the carbon felt preform (fig. 6), although it is an anti-friction reinforcement, the ablative materials with the addition of nanometric silicon carbide increase their tribological properties.



Friction coefficient of PR/nSiC/Felt measured at 1000 rpm



Friction coefficient of PR/nSiC/Felt measured at 1500 rpm

Fig. 6 Friction coefficient of PR/nSiC/Felt

Table 5. The friction coefficient of PR/nSiC/felt measured at 1000 and 1500 rpm speed [1, 10]

Material	Friction coefficient (1000 rpm)	Friction coefficient (1500 rpm)
PR/felt	0.178	0.187
PR + 1% nSiC/felt	0.23	0.252
PR + 2% nSiC/felt	0.297	0.299

Tribological testing of these materials showed the same variation as in the case of cork-based ablative materials samples, the coefficient of friction increased with the increase of the percentage of nSiC added to the matrix in the case of both test speeds used.
At 1000 rpm, the addition of 1 and 2% nSiC, respectively, generated an increase in the coefficient of friction by 29 and 67%, respectively. At 1500 rpm, the increases were 35% (for samples with 1% nSiC) and 60% (for samples with 2% nSiC) compared to the control.

3. CONCLUSIONS

Determining the coefficient of friction of materials is useful for assessing their tribological potential used as candidates for applications in friction or anti-friction systems. Qualitatively, materials with friction coefficients with maximum values of 0.2-0.25 are included and can be used as anti-friction materials (sliding bearings). In the case of materials that have friction coefficients with values higher than 0.25, they are considered friction materials (clutches, braking systems).
The paper presents the influence of the addition of nanometric silicon carbide in various mass percentages on the tribological properties of phenolic thermoreactive resin.
Overall, the results illustrate that nanometric silicon carbide substantially improves the tribological performance of phenolic resin, the friction coefficient increases with increasing percentage of nSiC, being able to recommend these materials as potential candidates as friction materials in abrasive applications.
It has also been observed that higher test speeds lead to higher friction coefficients.
Following this information, groups of layered composite materials and groups of ablative composites were developed that used the phenolic resin studied as a polymer matrix.

In the case of the group of layered composites based on glass fiber composites, the addition of nSiC generated increases of 30-37% in percentages of 1% and 40-45% for percentages of 2% by mass. In the case of carbon fiber laminated composites, the PR/ CF control sample has a very low coefficient of friction (0.13), due to the nature of the carbon fiber, so the addition of an abrasive compound such as nanometric silicon carbide generates very significant increases in the coefficient of friction namely 30-35 for 1% percent and 70-90% for 2%, respectively.
And in the case of the group of composite materials of ablative type, the abrasive nature of the nanometric silicon carbide determines major increases of the friction coefficients of the control materials (PR / Cork and PR / Felt).
Overall, the results suggest that the optimal percentage to obtain properties improved both mechanically and tribologically, is 1% by mass nSiC in the phenolic matrix of all materials developed in the work.

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