

# Influence of nanometric silicon carbide on phenolic resin composites properties

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**Abstract.** This paper presents a preliminary study on obtaining and characterization of phenolic resin-based composites modified with nanometric silicon carbide. The nanocomposites were prepared by incorporating nanometric silicon carbide (nSiC) into phenolic resin at 0.5, 1 and 2 wt% contents using ultrasonication to ensure uniform dispersion of the nanopowder, followed by heat curing of the phenolic-based materials at controlled temperature profile up to 120°C. The obtained nanocomposites were characterized by FTIR spectroscopy and scanning electron microscopy analysis and evaluated in terms of mechanical, tribological and thermal stability under load. The results highlight the positive effect of the nanometric silicon carbide addition in phenolic resin on mechanical, thermo-mechanical and tribological performance, improving their strength, stiffness and abrasive properties. The best results were obtained for 1 wt% nSiC, proving that this value is the optimum nanometric silicon carbide content. The results indicate that these materials could be effectively used to obtain ablative or carbon-carbon composites in future studies.

**Keywords.** Phenolic resin; nanometric silicon carbide; nanocomposites; friction coefficient.

## 1. Introduction

Phenolic resin composites have their applications in a wide range of fields from wood and adhesive industry to automotive, aeronautics and aerospace industries. Phenolic resins represent the first industrially produced synthetic polymer, first processed by Bayer in 1872 [1] and patented in 1907 by Baekeland [2]. This class of resins is used in a wide range of applications in fields from construction and automotive materials to high-tech industries such as aeronautics and aerospace [3]. This is sustained by their versatile properties, such as superior mechanical strength, heat resistance, chemical and dimensional stability, high resistance to water, acids and solvents [3], supplemented by medium price and relatively simple processing techniques via heat curing [2]. In the field of advanced composite materials, phenolic resin-based composites are known for their excellent flame resistance and the fact that they evolve low smoke upon incineration [3], being intensely used in the rocket industry because of their ablative characteristics [4]. Other important applications of phenolic resins are thermal insulation materials, molding compounds, foundry, coating materials, wood products industry, adhesives, polymeric blends with other polymers and other composite materials [5–7].

Even though the processing method involves facile routes, the synthesis conditions and parameters are essential to achieve resins with particular properties [8]. Curing time and temperature as well as mold materials influence the resulting homogeneity, glass transition temperature and mechanical properties.

Although phenolic resins have good thermo-oxidative resistance, research studies have been conducted to improve their thermal properties through modification of their structure with introduction of inorganic additives, including boron [8], phosphorous [9,10], zirconium [11,12] and silicon [13] based compounds. Moreover, studies presented the enhancement of ablative properties by the addition of carbon nanotubes [14–16] and nanofibers [17], graphite [18], montmorillonite clays and POSS compounds [19] and nanographene [20].

Owing to exceptional properties such as high temperature oxidative and thermal shock resistance, as well as good friction characteristics, silicon carbide is a promising nanoadditive for obtaining high temperature composites [21] as well as wear-resistant materials [22,23]. Silicon carbide in nanometric form has some advantages over its micrometric form, as being a nanofiller it can be added into much smaller contents relative to the polymeric matrix (down to less than 1 wt%) compared with classical fillers (that require even up to 20 wt% [24]), fact that contributes to achieve minimum weight

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increase. Besides this, lower contents required by nanometric SiC helps in an easier control of matrix viscosity increase issues, consequently helping to obtain a better homogenization with the aim of ultrasonication techniques.

There are several studies that show modified phenolic resin-based composites processed using advanced thermal treatments, used as final application ablative [25–27] or carbon–carbon composites [11,28,29]. Few studies [20,30] present the improvement and characterization of simple cured phenolic resin without supplementary thermal treatments to obtain further advanced composites. This study focusses on presenting the basic characteristics of the ‘precursor’ material used for advanced composites, highlighting the effect of nanometric SiC on the properties of the cured phenolic resin. Therefore, the aim of this study represents the confirmation that once these nanofilled composites are fully characterized and the optimization of concentration is performed, they will be able to serve as matrix for advanced materials (ablative or carbon–carbon composites) that will be achieved in future studies.

This paper presents the obtaining of phenolic resin-based nanocomposites, comprising a preliminary study regarding resin properties modification by the addition of nSiC in different contents. The processing technique involves heat curing of the simple and nanomodified matrix, the latter being developed via ultrasonication procedure prior to the curing stage. The obtained nanocomposites were tested in terms of mechanical, tribological and thermal stability under load properties and characterized by FTIR spectroscopy and SEM analyses. The results determine the optimum nanofiller content selection for future studies involving the use of this kind of materials as matrix in advanced composites (carbon fibre reinforced nanofilled phenolic resin composites and ablative materials).

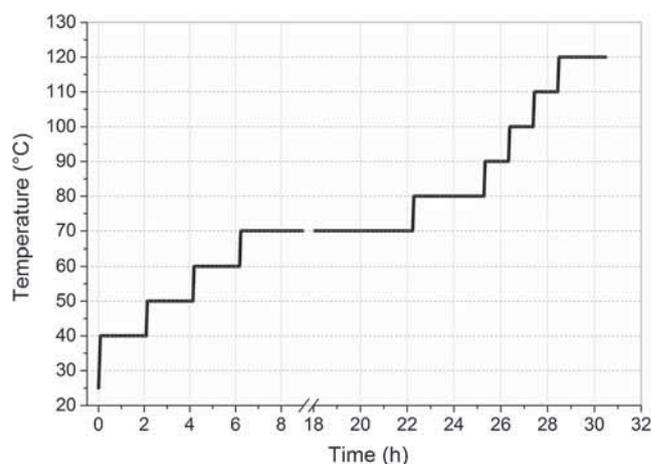
## 2. Experimental

### 2.1 Materials

Matrix used was resole type phenolic resin ISOPHEN 215 SM 57% provided by ISOVOLTA S.A. Bucharest, with  $1.135 \text{ g cm}^{-3}$  density. The nanofiller used was  $\beta$  type nanometric silicon carbide purchased from Nanostructured & Amorphous Materials Inc., USA, with the following characteristics: 97.5% purity,  $34\text{--}40 \text{ m}^2 \text{ g}^{-1}$  specific surface area and  $3.22 \text{ g cm}^{-3}$  true density.

### 2.2 Obtaining method

The nanocomposites obtaining method involved two stages: in the first stage dispersion of the nanometric silicon carbide (nSiC) powder into the phenolic resin (PR) occurred, and the second stage was heat-curing process. The first stage involved the addition of different contents (0.5, 1 and 2 wt%) of nSiC into the resin, mechanical stirring for approximately 5 min for bulk homogenization followed by ultrasonication technique for nanofiller optimum dispersion, each sample



**Figure 1.** Temperature treatment programme for obtaining phenolic resin-based composites.

being sonicated for a total of 12 min in 30 s sonication per step, using Bandelin Sonopuls sonde equipment.

The second stage consists curing process that took place in an oven up to  $120^\circ\text{C}$ . Both simple and nanomodified phenolic resin samples were cured in Teflon moulds with geometry and dimensions specific for the mechanical tests. The inner surface of the moulds was cleaned and coated with a silicone oil [31] as release agent. The silicone oil was observed to be effective in decreasing void formation at the bottom surface of the sample during curing process. The mould was dried in an air oven at  $75^\circ\text{C}$  for 10 min to eliminate the existing water. The moulds were filled with simple and nanomodified resin samples and the curing process was performed following a controlled temperature schedule, according to the scheme in figure 1. Methanol solvent in the resin evaporates around  $70^\circ\text{C}$  generating gas bubbles in the composite mass. The several trial sessions revealed that extended dwell time at  $70^\circ\text{C}$  supports gas release from the resin solution. After the curing cycle was completed, the samples were cooled in air down to room temperature. The following nomenclature for the final nanocomposites was used: PR, PR+0.5% nSiC, PR+1% nSiC and PR+2% nSiC where 0.5, 1 and 2% represent the nSiC weight content relative to the phenolic resin (PR).

### 2.3 Testing and characterization

Nanocomposites were characterized in terms of mechanical, thermo-mechanical, tribological and morphological properties. The phenolic-based nanocomposites were subjected to spectroscopy analysis using a Spectrometer Nicolet iS50 (operated in ATR mode) and scanning electron microscopy (SEM) using QUANTA INSPECT F microscope with field emission gun and 1.2 nm resolution and energy dispersive X-ray spectrometer (EDS). The materials were subjected to mechanical tests by 3-point bending with INSTRON 5982 mechanical testing machine according to SR EN ISO 178, using  $2 \text{ mm min}^{-1}$  testing speed and nominal span length ( $16 \times$  specimen thickness), on rectangular specimens

(80 × 10 × 4 mm), using 5 specimens per sample. Tribological tests were performed using CETR UMT 3 (Universal Macro Materials Tester) block-on ring module, on a 35 mm diameter steel role, under 10 N load, for 60 s at two different speeds: 1.75 and 2.62 m s<sup>-1</sup>, testing 3 specimens for each sample. HDT thermal stability was evaluated using Qualitest HDT1-heat deflection system, according to SR EN ISO 75, testing 3 specimens for each sample using 2°C min<sup>-1</sup> heating rate and 1.8 MPa flexural stress, in silicone oil immersion environment.

### 3. Results and discussion

#### 3.1 FTIR spectroscopy

FTIR spectroscopy analyses were performed on the liquid uncured phenolic resin sample and on the grinded cured

phenolic resin-based composites samples. The control cured sample was compared with the uncured sample (figure 2) to evaluate if the curing process was completed, while the nanofilled samples were compared with the cured unfilled sample (figure 3) to evaluate the existence of interactions between nSiC and phenolic resin resulting in vibrations that generate peak intensity modification or peak position shifting. The used resin is a resole type that contains ether bridges (–CH<sub>2</sub>–O–CH<sub>2</sub>–) that appear at approximately 1015 cm<sup>-1</sup> and methylene bridges (–CH<sub>2</sub>–) that appear at 2900 and 1470 cm<sup>-1</sup> as shown in figure 2. Curing takes place by heating the resin and temperature processing generates formaldehyde elimination from etheric bonds, so that the final heat-treated cured resin will be based only on methylene transversal bonds. This is demonstrated by the disappearance of the peak at 1015 cm<sup>-1</sup> in the cured resin, proving that the curing was completed.

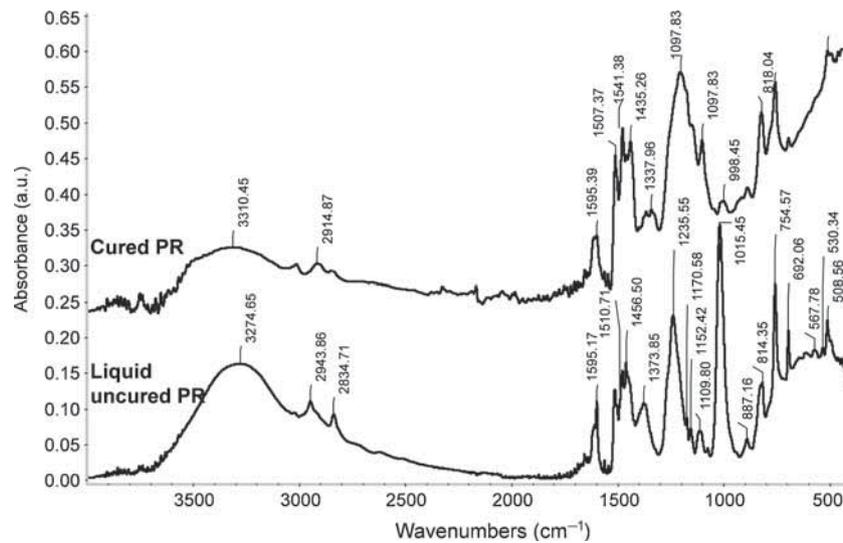


Figure 2. FTIR spectra of the uncured and cured phenolic resin.

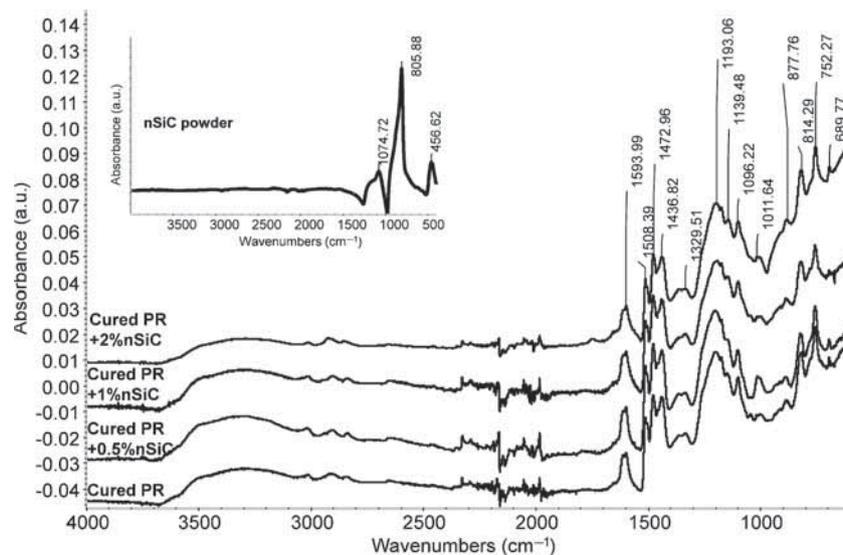


Figure 3. FTIR spectra of simple and nSiC-based phenolic resin samples.

FTIR spectroscopy analysis were performed on the grinded cured samples. Figure 3 shows the spectra of the simple and silicon carbide nanofilled phenolic resin. All samples show the characteristic peaks of the cured resin: OH stretching from resole appears at  $3280\text{ cm}^{-1}$ ,  $\text{CH}_2$  stretching at  $2900\text{ cm}^{-1}$ ,  $\text{C}=\text{C}$  stretching in the aromatic ring results in peaks in  $1600\text{--}1470\text{ cm}^{-1}$  range,  $\text{CH}_2$  bending vibration is visualized at  $1437$  and  $1330\text{ cm}^{-1}$  and  $\text{C}-\text{O}$  stretching at approximately  $1200\text{ cm}^{-1}$ . The three peaks in the  $900\text{--}600\text{ cm}^{-1}$  range are due to the ortho-disubstituted, meta-disubstituted and mono-substituted benzenes [31,32].

Nanofilled samples spectra do not present major visible differences compared to the cured resin. This is due to the fact that the nSiC characteristic peak, assigned to  $\text{Si}-\text{C}$  vibration, appears at approximately  $800\text{--}815\text{ cm}^{-1}$  [33,34], overlapping with meta-disubstituted benzene peak from phenolic resin. But the nSiC interaction with the phenolic resin can be highlighted by the peak shifting from  $805$  (figure 3 inset) to  $\sim 815\text{ cm}^{-1}$ . Evaluating the intensity ratio between the peaks from approximately  $815\text{ cm}^{-1}$  and one of the main peaks of the phenolic resin ( $1595\text{ cm}^{-1}$ ), it can be noticed that its

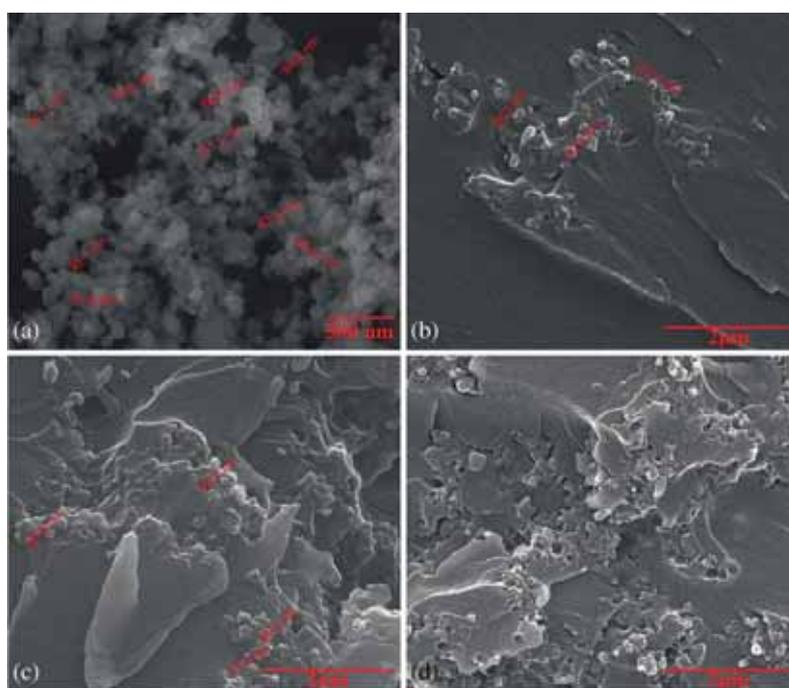
value increases with nSiC content increase, as illustrated in table 1. The position of the peak from  $\sim 815\text{ cm}^{-1}$  is shifting towards lower value once with the nSiC content increase. It is also worth to mention that strong shift occur at 0.5 and 1% nSiC and only marginal shift for the 2% SiC which means that the additional 1% SiC practically does not interact with the matrix. This effect could be due to the interaction between nSiC and phenolic resin, the intensity increase being a consequence of the higher nSiC content that interacts with the matrix. The noise between  $1900$  and  $2300\text{ cm}^{-1}$  observed because of the absorption of the diamond crystal and not due to the absorption of the sample and hence must be ignored.

### 3.2 SEM analysis

SEM microscopy analyses were performed in the fracture cross-section of the mechanically tested sample. Figure 4 presents SEM images of the PR-based materials. Figure 4b–d shows nSiC-based samples illustrating the brittle nature of the fracture. The nSiC presence is more visible as the content increases. Some agglomeration areas can be noticed, but the

**Table 1.** Intensity ratio between nSiC peak and one of the main peaks in PR.

Sample	PR	PR+0.5% nSiC	PR+1% nSiC	PR+2% nSiC
Peak 1 position	1594.76	1594.99	1594.84	1593.99
$I_1$ (peak 1 intensity)	0.0536	0.0145	0.0163	0.0306
Peak 2 position	818.04	817.9	814.77	814.29
$I_2$ (peak 2 intensity)	0.103	0.0292	0.038	0.0833
$I_2/I_1$	1.92	2.01	2.33	2.72



**Figure 4.** SEM images of (a) nSiC nanopowder and PR-based materials cross-section of (b) PR+0.5% nSiC; (c) PR+1% nSiC and (d) PR+2% nSiC.

**Table 2.** Mechanical and thermo-mechanical properties of PR-based materials.

Sample	Flexural strength (MPa)	Flexural modulus (GPa)	Extension (%)	Heat deflection temperature (°C)
PR	111.1 ± 8.76	5.3 ± 0.12	2.91	104.2
PR+0.5% nSiC	125.5 ± 13.8	5.8 ± 0.35	2.34	126.7
PR+1% nSiC	148 ± 5.6	6.9 ± 0.15	2.28	132.1
PR+2% nSiC	126.2 ± 7.8	6.1 ± 0.06	2.21	129.7

nanoparticles dimensions can still be distinguished. As nSiC SEM image (figure 4a) shows the irregular shaped nanoparticles' diameter is in the range of 35–140 nm. The nanoparticles average dimension in PR samples is approximately in the same range showing that the nanoparticles did not form large aggregates, but they are distributed in the whole mass-forming areas with high concentration of nanoparticles.

### 3.3 Mechanical testing

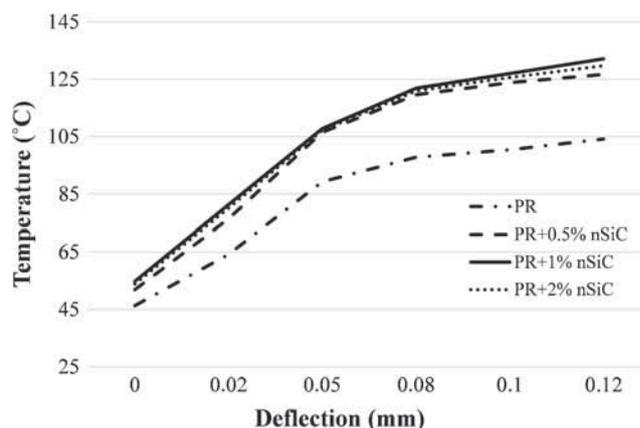
Mechanical testing consisted of 3-point bending tests. Flexural tests were performed both on the simple cured resin and on the nanofilled resin samples. The test was set to be carried out until conventional deflection, but all specimens broke before this value was reached, as phenolic resins are brittle materials. Maximum flexural stress represents flexural strength.

For each tested sample, an average of optimum specimen replicas was calculated. Comparing flexural strength and modulus results (table 2), it is observed the positive effect generated by nSiC addition in the phenolic resin. The most substantial increase in strength and modulus is presented by the samples with 1 wt% nanofiller. In terms of flexural strength, adding 0.5 and 2 wt% nSiC resulted in a 13% increase compared to the simple resin, while the addition of 1 wt% nSiC-generated an increase of flexural strength greater than 33%. Regarding flexural modulus, 0.5 wt% nSiC-based samples showed an average value around 10% higher than the simple PR, followed by the 2 wt% based samples with an increase of 15% and 1 wt% based sample with an increase of approximately 30%. The obtained results could be due to the fact that the nanoparticles are embedded in the phenolic resin and uniformly covered, creating a strong mechanical interface between the phases. A possible reason could be the fact that the embedded nanoparticles could contribute to reduce the crack propagation effect in the resin, acting as a reinforcing agent, increasing the PR strength and stiffness.

Flexural strain average values were in accordance with strength and modulus values, all nanofilled samples showed a decrease of strain compared to the simple resin. Lower strain values generate higher strength and modulus.

### 3.4 Thermal stability under load

Figure 5 illustrates the deflection that the specimens suffered during HDT testing, showing the similar trend of the samples. It can be noticed that nSiC presence enhances the phenolic resin thermal stability under load.

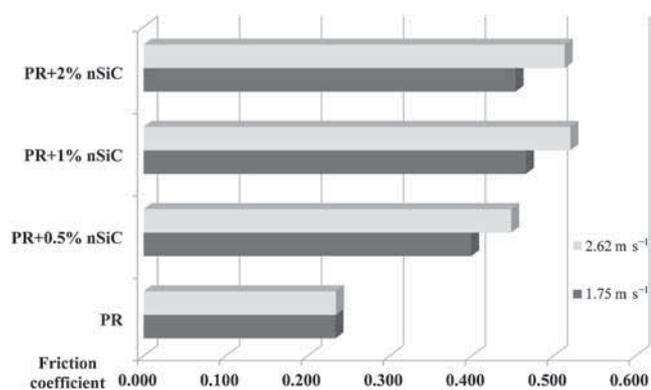
**Figure 5.** Deflection–temperature curves during HDT test.

The most substantial increase in thermal stability under load is presented by the samples with 1 wt% nanofiller as it can be seen in table 2. In terms of thermal stability under load, adding 0.5 and 2 wt% nSiC resulted in a 22% increase compared to the simple resin, while the addition of 1 wt% nSiC generated an increase of thermal stability under load greater than 27%.

These results are in accordance with flexural properties obtained from the 3-point bending tests that show that nSiC content increase up to 1% leads to an increase of properties, while increasing the content up to 2% generates a decrease of strength and stiffness compared with prior content. This trend can be correlated with FTIR spectroscopy analysis that suggest that the additional nSiC added in 2%-based samples does not interact with the matrix (in comparison with 0.5 and 1%) and also with the presence of higher degree of agglomerations in the 2% nSiC-based phenolic resin. These agglomeration are most likely owing to the higher concentration that generated higher viscosity mixtures, that were more difficult to uniformly disperse and create homogenous materials, but also to the additional nSiC that did not interact with the matrix. The inhomogeneity in PR+2% nSiC act as stress concentration sites that negatively affect mechanical properties at room temperature and higher temperature, as they act as defect points. When a load is applied, in these agglomeration sites, the load distribution and transfer is significantly affected, generating premature failure of the materials and lower strength and stiffness.

### 3.5 Tribological testing

Likewise, tribological tests results highlighted the positive effect of nSiC addition. Figure 6 illustrates the friction



**Figure 6.** Friction coefficient variation function of nSiC content, evaluated using 2 speed rate values ( $1.75$  and  $2.62$   $\text{m s}^{-1}$ ).

coefficient average values obtained for simple and nanofilled resin. For both speed values, friction coefficient increases when nanocarbide was added. For the  $1.75$   $\text{m s}^{-1}$  speed value, friction coefficient increased with the increase of nSiC content, up to 1 wt% nSiC, and at higher content (2 wt%), friction coefficient showed a minor decrease compared to 1 wt% content. For the  $1.75$   $\text{m s}^{-1}$  speed value, the friction coefficient of 0.5, 1 and 2 wt%-based samples increased by 71, 99 and 94% compared to the simple resin.

For  $2.62$   $\text{m s}^{-1}$  speed, friction coefficient increased uniformly with nSiC content increase, showing 91, 122 and 119% higher values than PR.

The 2 wt% nSiC-based samples' friction coefficient slight decrease could be due to small size nonuniformities of the nanopowders dispersion in the matrix, as 2 wt% contents present higher agglomeration tendency compared to lower contents.

Overall, these results show that nanometric silicon carbide can substantially improve tribological performance of phenolic resin, supporting them as potential candidates for high friction materials to use in abrasive applications.

#### 4. Conclusions

The preliminary results presented in this paper highlight the positive effect of nSiC addition in phenolic resin on mechanical, thermo-mechanical and tribological performances, improving their strength, stiffness and abrasive properties. Following a trial established thermal treatment, different PR/nSiC formulations were obtained, characterized by FTIR spectroscopy and SEM and tested in terms of mechanical, thermo-mechanical and tribological properties. Results show that the best performance in all conducted tests are obtained when using 1 wt% nSiC. This content is the optimum as it leads to a solid embedding of the nanoparticles into the resin, as they interact completely with the matrix, as FTIR analysis shows, without generating high agglomeration areas that form at higher nSiC content (2 wt%) as shown by SEM analysis. This allows the nanoparticles to act as a reinforcing agent, probably by reducing crack propagation effect and

leading to higher mechanical and thermo-mechanical (heat deflection temperature) properties. Therefore, the same trend noticed in both flexural and thermal stabilities under load tests is most likely owned to the fact that additional nSiC in 2% samples does not interact with the matrix and also to the significant agglomerations degree in the 2% nSiC-based phenolic resin generated by the higher nSiC content that increased the mixtures viscosity, and consequently hindered the uniform dispersion and homogeneity of the materials.

nSiC being a popular abrasive material, the tribological properties improvement was also confirmed, the best result was obtained for 2% content, but 1 wt% nSiC also determined substantial friction coefficient increase. Therefore, the optimum nSiC content can be considered for 1 wt% as it leads to several properties improvements.

The results represent the preliminary stage experimental study regarding phenolic resin properties modification with the aim of using it as matrix for advanced composite materials, such as carbon preform (fibre, fabric, felt)-based materials for aerospace applications.

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