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Development of CNC prototype for the characterization of the nanoparticle release during physical manipulation of nanocomposites

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Abstract

This work focuses on the release of nanoparticles from commercially used nanocomposites during machining operations. A reliable and repeatable method was developed to assess the intentionally exposure to nanoparticles, in particular during drilling. The paper presents the description and validation of results obtained from a new prototype used for the measurement and monitoring of nanoparticles in a controlled environment. This methodology was compared with the methodologies applied in other studies. Also, some preliminary experiments on drilling nanocomposites are included. Size, shape and chemical composition of the released nanoparticles were investigated in order to understand their hazard potential. No significant differences were found in the amount of nanoparticles released between samples with and without nanoadditives. Also, no chemical alteration was observed between the dust generated and the bulk material. Finally, further developments of the prototype are proposed.

Keywords: Nanosafety, commercial nanocomposites, standardization, machining.

Introduction

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Nanomaterials are one of the most promising technologies of this century. The annual quantity of nanomaterials marketed in 2012 was around 11.5 million tones with a value of roughly 20 billion euros. [1] However, during its life-cycle, a nanotechnology-based product can release nano-sized particles exposing workers (including researchers), consumers and environment to potential risks. [2] The impact of these risks is not well known, [3–5] and specific legislation and regulation around the world in relation to chemicals and environmental protection does not cover this type of materials. [6, 7]

Recently, researchers have investigated the release of nanoparticles in different mechanical stress situations such as shredding, drilling, sanding, and abrasion of nanocomposites. [8–11] These situations are supposed to represent different common machining operations of nanoproducts. For example, Sachse et al. [12] studied the release of nano-size particles during the drilling of different polyamide-6 nanocomposites within NEPHH project. The aim of NEPHH project was to develop a protocol for assessing the unintentionally release of nanoparticles and it is one of the pioneering attempts in this area. [12, 13] They found that the integration of nanofillers into a polymeric matrix influences the material behavior, the quantity of particles released during drilling experiments and the physical properties of the nano-sized particles emitted. However, this study has significant problems and deficiencies like contamination from tools and the environment and a lack of control over the process parameters.

Azarmi et al. [14] conducted a study focused on the exposure to particles from processing concrete. They studied mixing, cutting and drilling of concrete and used water spray during the drilling activities to mimic the practices in the construction sector. The sampling of airborne particles was placed 1m away from the source. They found that the cutting activity produced the highest release of particles, followed by drilling and mixing activities. However, the study did not take into account the possible contamination from the tools.

The current work, which is linked to SIRENA project [15], contributes to a better understanding of potential exposure. [16] Some of the challenges involved in this area were brought to the fore by Gendre et al. [2] , namely

1. a complete analysis of all the possible exposure scenarios is necessary
2. no standardized method exists to measure and characterize nanoparticles released during mechanical stress situations
3. the equipment used in order to estimate the quantity of nanoparticles released in the air can be a source of error for accurate measurement
4. background noises were often reported as a source of variability.

In this study, NEPPH protocol for assessing the exposure of nanomaterials has been evaluated. The protocol has been replicated, but deficiencies have been found and corrected. This has helped in the development of a new methodology in SIRENA. This paper describes the development of a

prototype to assess the release of nanoparticles by mitigating or eliminating the limitations associated with previous study [12] like reduction of contamination sources, and controlling the process parameters. This system has been characterized, and preliminary testing on cutting and drilling nanocomposites for validation has been conducted. In addition, the particles released from the testing (airborne particles and deposited fraction of particles) have been characterized using several analytical techniques. Further improvements of the prototype are also suggested.

Materials and methods

Materials

For the replication of NEPHH protocol, 3-phase nanocomposites made of polyamide-6 reinforced with 30 wt.% of glass fiber and different contents of nanosilica (nano-SiO2), 0.5, 1, 1.5 and 3 wt.%, or organically modified montmorillonite (OMt), 5, 7.5 or 10 wt.% were used. The samples were 'donut'-shape rings of dimensions 160mm for the external diameter, 100mm for the internal diameter, and 4mm for the thickness.

For testing the new protocol al prototype developed within SIRENA, the samples used were manufactured by Tecnalia Corp. (Spain). The nanocomposites were based on polyester (PS) with

two types of nanofiller, nano-silica and nano-alumina, with 2 or 5 wt%. Unsaturated orthophtalic polyester was supplied by Gazechim Composites (France), and nanofiller, nano-silica and nanoalumina by Torrecid Group (Spain). Composition and nomenclature of the samples are provided in Table 1.

Replication of NEPPH protocol

The system in NEPHH was composed of a close chamber (dimensions 690x330x560 mm) equipped with gloves, a manual drill 'Makita BDA351Z 18V LXT Angle Drill', and a portable scanning mobility particle sizer and particle counter ('SMPS+C') from Grimm Aerosol, with a particle size resolution of 44 channels over a size range of 11.1-1083.8nm. The protocol applied was a first step of purging the chamber for 20 minutes with air from the room. Then the sample was fixed into the chamber. The manual drill was totally enclosed in the chamber throughout the measurement cycle. The 'SMPS+C' was connected to the chamber using an antistatic hose via a hole on the top of the chamber. After closing the chamber, the measurements started by monitoring for 30 minutes the background noise (air inside the chamber) using the 'SMPS+C', then the sample was drilled for 7 minutes (time that the 'SMPS+C' needs to carry out one scan), and the cycle finished with 60 minutes measurement of post-drilling. The angle drill was deployed at its

maximum speed of 1800 rpm; two different sizes of drill bits were studied: 5mm and 8mm diameter; and two different feed rates: 4mm/min and 1.14mm/min. The experiment was repeated three times for each material composition and drill bit size. In addition, every morning one measurement cycle was conducted in order to record the noise of the drill, itself. The manual drill was switched on and run, but no materials were drilled. The data for the total number of particles (C, cm^{-3}) and particle size distribution was obtained from the 'SMPS+C'.

Description of new prototype

A new set-up was developed in order to deliver robust and repeatable experiments. A schematic representation of the system is presented in Figure 1. The main features and elements of this system are:

'Environmental control system' comprising of a sealed chamber with a fan, BenchVent I100-4 and a pre-filter and HEPA filter (category H14) that were used to clean the air inside the chamber and air recirculation system to reduce the amount of 'dirty' air outside the chamber.

'Automatic machining system' comprising of a CNC machine designed and built at Cranfield University to ensure the precise control of drilling parameters (feed rate, spindle speed, etc.) and a water cooled spindle drill to avoid background noise or particles produced by the motor; the motor

being totally sealed.

'Dust collection system' to collect the deposited fraction of particles and composed of a petri dish with lid, adapted for the drilling process and located on the surface of the sample. The petri dish was sealed after the experiment and used as a container.

'SMPS+C' was used to monitor the nanoparticles released. This equipment was connected to the chamber using antistatic hoses. The particle size range measured ranged from 11.1 to 1083.8nm distributed in 44 channels.

Preliminary testing: drilling nanocomposites

Firstly, in order to compare the new protocol with the protocol developed in the NEPHH, the manual drill used in the NEPHH experiments was monitored with the new prototype. The manual drill was working for 7 minutes with a drill bit of 8mm diameter at a maximum speed (1400 rpm), but no sample was machined. The air inside the chamber was monitored prior to using the manual drill and the airborne particles released by the drill. The hose for air inlet to the 'SMPS+C' was placed near the drill bit. Another test was conducted but without the air recirculation system running. Likewise, the CNC machine was characterized with the same procedure.

The parameters for the drilling experiments of the nanocomposite samples were selected to be as

similar as possible to the industrial processes: [17, 18]

1. Cutting tool: HSS plain shank short drill bit, 3.5mm diameter
2. Spindle speed: 8500 rpm
3. Feed rate: 200mm/min

Only one hole of 5mm depth was drilled in each sample, so that the same quantity of material was drilled. The airborne particles were studied using the 'SMPS+C' and the deposited particles were collected inside the Petri dish and characterized. Before drilling commenced, a baseline measurement of the Petri dish was undertaken with the 'SMPS+C' in order to compare with the airborne particles released during drilling.

Characterization of the airborne particles

Number concentration of particles (C, cm^{-3}) and particle size distribution were determined using a 'Scanning Mobility Particle Sizer' and 'Particle Counter' ('SMPS+C') from Grimm Aerosol, with a particle size resolution of 44 channels over a size range of 11.1-1083.8nm. The' SMPS+C' comprises of a Condensation Particle Counter (CPC) model 5.403 with a classifier type Vienna, long U-DMA, for the measurement of the airborne particles. Each measurement cycle or scan lasted approximately 7 minutes.

Characterization of the deposited particles

Scanning electron microscopy (SEM) was used to characterize the dust collected from the drilling or cutting experiments. The microscope used was a high resolution scanning electron microscope FEI XL30 SFEG analytical. Specimens were gold-palladium sputtered to minimize charging of the samples.

Fourier transforms infrared spectroscopy (FTIR) was performed on the samples and on the dust collected using the Petri dish. FTIR spectroscopy model Jasco 6200 with accessory ATR IR from Pike- model miracle window- diamond/ZnSe was used. The scan range was from 4000 to 500 wavenumber.

Results and discussion

Deficiencies observed during the replication of NEPPH protocol

Influence of the drill bit size

The evolution of the number concentration of airborne particles (C, cm^{-3}) versus time for all the

samples when drilling with 5mm and 8mm diameter drill bit (Fig. 2), showed that there was no clear trend in the nanoparticle release. In all the cases, C increased when the drilling started and decreased following completion of the drilling process. However, the maximum value of C varied depending on the material and the drill bit. With the 5mm drill bit, the samples with low content of nano-silica (0.5 and 1 wt.%) appeared to release more particles than the samples with the highest concentration (3wt.%). In the set of samples with OMt, the sample with the highest concentration (10 wt.%) released the highest number of particles. In the case of the 8mm drill bit, all the samples with nano-silica released a similar concentration of particles. On the contrary, the samples with OMt when drilled with the 5mm drill bit, it was the sample with the lowest concentration which released the highest number of particles.

With the 5mm drill bit, the maximum value of C for the samples varied in the range 100000 to 550000, whereas with the 8mm drill bit, the range was 150000 to 1200000. This could be explained by the fact that the volume of material drilled using the 8mm drill bit was higher compared to the volume of material drilled with the 5mm drill bit.

Influence of the feed rate

As the angle drill used was manually operated, the feed rate was only controlled by the pressure

exercised by the drill on the plate, thus the pressure exerted by hand. This parameter was clearly difficult to control and replicate. Two different feed rates were studied, 4mm/min and 1.14mm/min. It was found that at a slow feed rate, the number of particles produced was 100 times higher than at a fast speed rate. This result is remarkable due to the fact that at a fast feed rate the amount of material drilled is higher than at a slow feed rate. Also, the mean diameter of particles was smaller at the slow feed rate (around 20nm) than at the high speed rate (around 70nm). These results reveal that the feed rate is an important parameter to control in order to produce repeatable results as it has a significant influence on the size distribution and number of particles released.

Noise from the manual drill

Each day one cycle measurement was carried out in order to record the background noise of the drill, itself. This was conducted without the drilling of any samples. It was found that the drill generated a significant amount of nanoparticles, and was highly variable. Different activities were carried out in the workshop at the same time as the drilling experiments. Consequently, they could have effected changes to the environment. Some studies regarding machining such as cutting, drilling or sanding had already reported the effect of the environment and tools such as noise from airborne

nano-sized particle measurements. [19–22] Background noise from the manual drill varied from around 70000cm^{-3} to 700000cm^{-3}. According to the fact that the background number of particles without any activities is around 10000cm^{-3}, the particles produced by the drill itself are not negligible and indeed are very significant. This means that all the previous results can be contaminated by the drill.

These results demonstrate that the protocol developed in the NEPHH project exhibited a large number of deficiencies. Drilling parameters (in particular feed rate and spindle speed) have a significant effect on the release of nanoparticles; and are not possible to be controlled when using a manual drill, including the angle of drilling. The contamination from the manual drill was a critical aspect. The evaluation of the particles released from the drill shows that any result obtained from the NEPHH protocol does not simulate the release of particles from the nanocomposites in 'service life'. Additionally, there was no control over the background noise. This depended on the quality of the room's air as the chamber was purged before each experiment.

Results from application of SIRENA protocol

Contamination from the manual drill

The results for total concentration of particles released from the manual drill in both situations (with and without air recirculation) are shown in Table 2. The manual drill released a considerable number of particles (C), compared to the air inside the chamber (approximately 10 times greater). This value was even higher when the air recirculation system was not working (>65000 cm^{-3}). In both cases, the higher concentration of particles was found in the size range 10mm to 50nm. This demonstrates that in the NEPHH protocol a considerable number of the particles measured were a contribution of the manual drill and not as a result of the samples tested. For the CNC machine, the total number concentration of particles went from an average of 590 ± 75cm^{-3} inside the chamber to 905cm^{-3} with the air recirculation system running. With the new prototype and protocol it was demonstrated that the equipment used in the NEPHH protocol, specifically the manual drill, is an important source of contamination. This compromises all the results obtained when the NEPHH protocol was applied. The particles released from the manual drill are likely to be metallic ones produced by the motor of the manual drill which utilize metal brushes. At the same time, it was demonstrated that the new configuration (CNC machine with isolated and water cooled motor) was not a contamination source, as there was not a significant increase in the number of particles when the motor was on.

Drilling experiments

The total number concentration of particles and particle size distribution for the three nanocomposite samples are presented in Table 3 and Figure 3, respectively. The total number of particles released during the drilling experiment was not significantly different from the baseline experiment. The difference with the background was below 10%. This behavior can be explained by the fact that the amount of material drilled was very small (approximately 48mm^3) or because the drilling time (a few seconds) was very short compared to the measurement time of the 'SMPS+C' for a single cycle (7 minutes). To overcome this problem, a new design of the drilling experiment will be established. Applying the same drilling parameters, a large number of holes can be drilled in the sample. Thus, the drilling time can be extended to 7 minutes. Regarding the particle size distribution, the three samples present a similar profile to the baseline experiment (inside the Petri dish before drilling). Only the sample D-P presents a slight increase of particles in the size range 17mm to 115nm.

The characterization of the deposited fraction of particles with SEM (Figure 4) shows that the shape and size of the fragments produced during drilling are similar in the three samples. The similarity in the surface patter can be attributed to the drilling parameters. .Following industrial guidelines, feed rate and cutting speed were kept as high as reasonably possible to prevent melting of the matrix. Low feed rate and/or cutting speed can result in long processes, increasing the temperature of the

sample at the cutting point up to the glass transition point and melting the polyester. In the images of scale 20, 10 and 2 μm, small particles can be identified. In more detail, nanoparticles can be observed in the images of scale 500 nm. The sample with no nanofiller (D-P) presents less nanoparticles than the samples with nanosilica and nanoalumina. These nanoparticles could be attached or adhered to the surface. Presumably, the nanoparticles measured with the 'SMPS+C' came from the physical degradation suffered by the surface of the sample. However, it is difficult to distinguish if there were nanoparticles completely separated from the surface, or adhered to it. One of the contamination routes is through skin contact. Consequently, it is important to determine if the large fragments can be a source of contamination for human beings.

FTIR spectroscopy was carried out in order to identify any possible chemical change in the particles released. The test was conducted on the original samples and in relation to the dust collected during drilling. The characteristic peaks of polyester, nanosilica and nanoalumina are summarized in Table 4.[23–26] No significant changes in the FTIR spectra can be observed (Fig. 5). The positions of peaks, shoulders, etc., in the FTIR spectra of the drilled and non-drilled samples were the same. This indicates that the dust generated has the same composition as the original bulk sample and that no chemical changes occur during the physical processing (drilling). On the other hand, it is not possible to identify clearly the peaks related to nanosilica and nanoalumina. The FTIR of the PS used in these samples also presented some absorption peaks in the characteristic

area of the nanofillers (nanosilica and nanoalumina). The low content of nanofiller and the thickness of the non-drilled sample (5mm to 8mm) could explain the fact that the characteristic peaks of the nanofillers did not appear, or had a low intensity and were overlapped with other peaks.

Conclusion

The NEPHH protocol for assessing the release of nanoparticles from nanocomposites has been evaluated. It has been demonstrated that this protocol exhibits a significant number of deficiencies, including reproducibility and was compromised by sources of contamination. In conclusion the protocol is not sufficiently robust for this purpose and thus cannot be applied to the study of the release of nanoparticles from nanocomposites.

A new prototype and protocol have been developed to assess the release of nanoparticles from nanocomposites. An automated system controls the process parameters and there is precise control over the environment in the chamber where the experiments take place.

An experiment for drilling nanomaterials was designed, based on industrial machining guidance. According to this, process parameters such as feed rate and cutting speed were selected to be sufficiently fast enough to prevent the resins from melting. The results show that the concentrations of airborne particle released when drilling one single hole were similar to the

312 concentrations of particles in the background (container). The drilling time was a few seconds, very short when compared to the measurement cycle of the 'SMPS+C' (7 minutes). To overcome this problem, the drilling time can be increased by drilling more holes in the samples. This will increase time and the quantity of material drilled. On the other hand, the SEM analysis of the deposited particles demonstrates the existence of nanoparticles that could be attached or adhere to the surface of the fragments generated during drilling. FTIR spectra show that no significant chemical changes occurred in the deposited fraction of particles during the drilling process.

In general, it can be concluded that the new prototype provides reproducibility and reliability, overcoming issues extant in the previous protocol which included contamination from the manual drill, lack of precise control of process parameters and contamination from the background environment.

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FIGURE CAPTIONS

Figure 1. Schematic of the new configuration for the drilling prototype with dust collection system

Figure 2. Number concentration of particles (C) vs time during the replication of NEPHH protocol for the 5mmØ drill bit and for the 8mmØ drill bit

Figure 3. Particle size distribution of the samples and baseline during drilling experiment

Figure 4. SEM images of the deposited fraction of particles released during drilling

Figure 5. FTIR spectra of the drilled samples

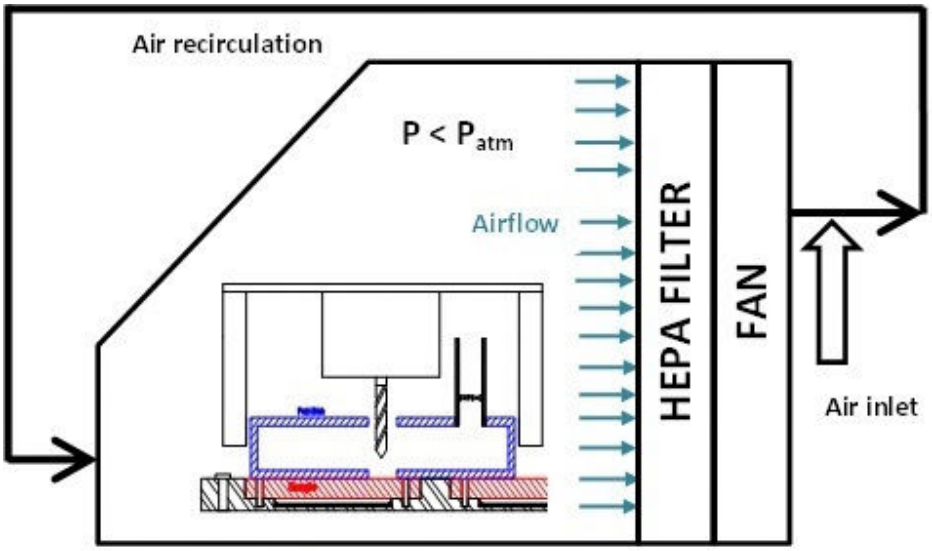
Air recirculation
P < P atm
Airflow
HEPA FILTER
FAN
Air inlet

Fig. 1

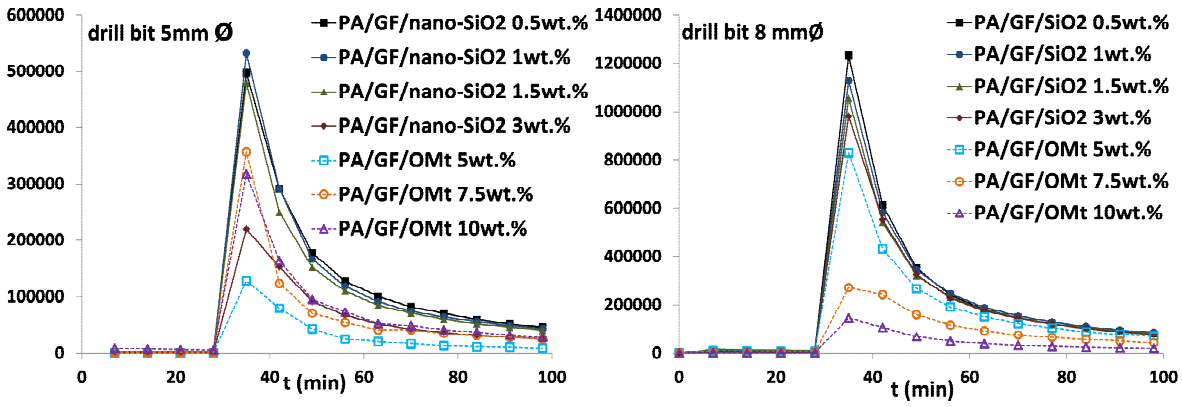
drill bit 5mm Ø
PA/GF/nano-SiO2 0.5wt.%
PA/GF/nano-SiO2 1wt.%
PA/GF/nano-SiO2 1.5wt.%
PA/GF/nano-SiO2 3wt.%
PA/GF/OMt 5wt.%
PA/GF/OMt 7.5wt.%
PA/GF/OMt 10wt.%
t (min)
drill bit 8 mmØ
PA/GF/SiO2 0.5wt.%
PA/GF/SiO2 1wt.%
PA/GF/SiO2 1.5wt.%
PA/GF/SiO2 3wt.%
PA/GF/OMt 5wt.%
PA/GF/OMt 7.5wt.%
PA/GF/OMt 10wt.%
t (min)

Fig. 2

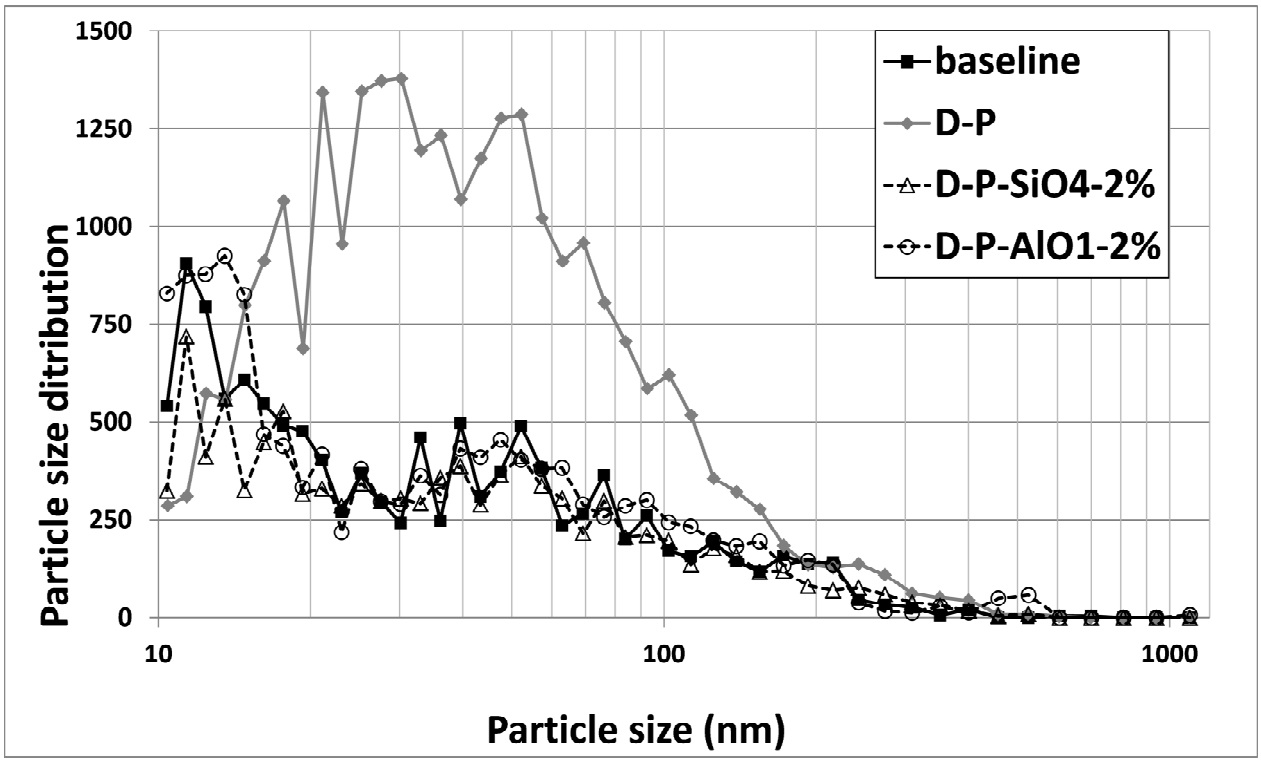
1500
1250
1000
750
500
250
0
Particle size ditribution
10
100
1000
Particle size (nm)
baseline
D-P
D-P-SiO4-2%
D-P-AlO1-2%

Fig. 3

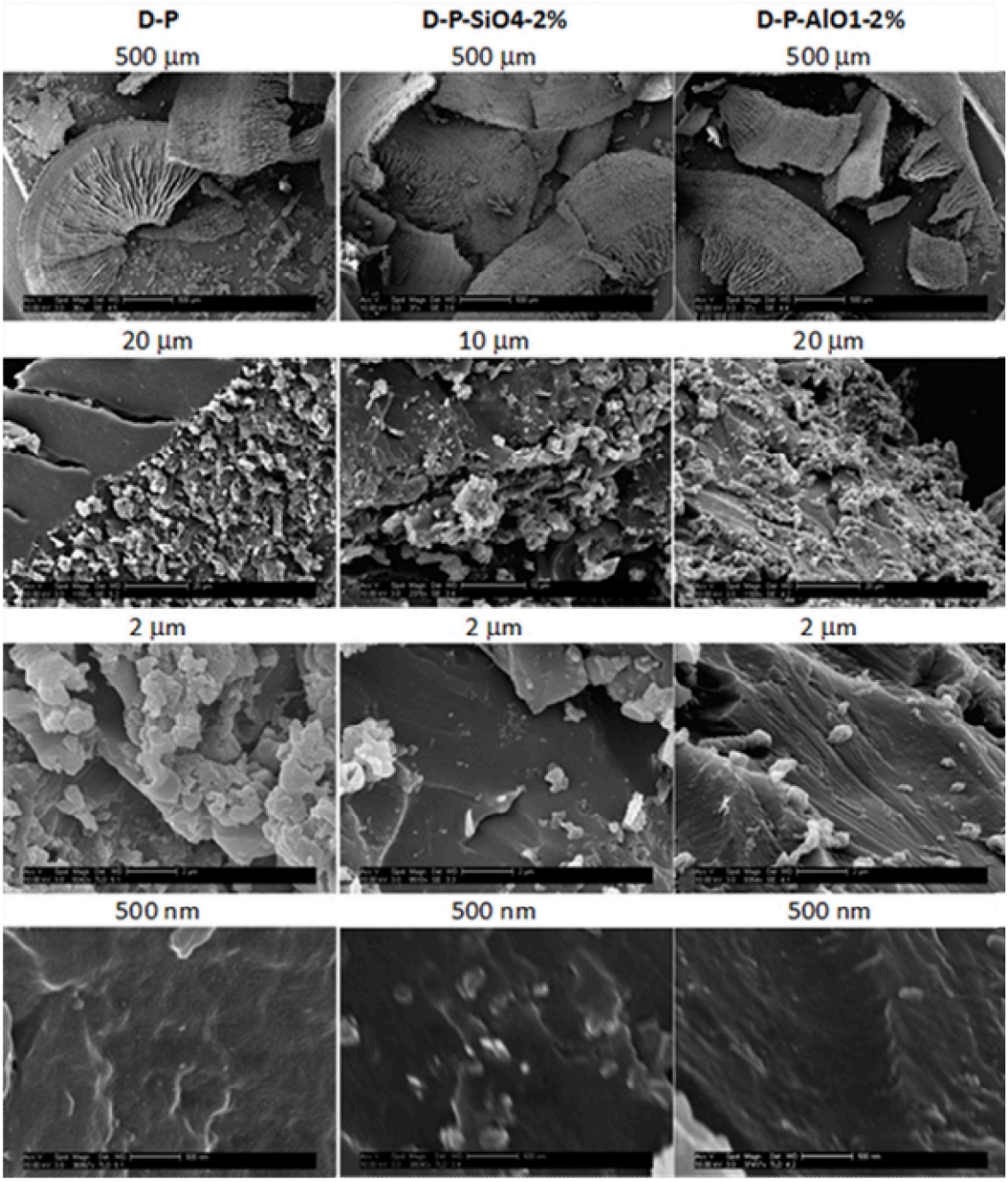
D-P
D-P-SiO4-2%
D-P-AlO1-2%
500 µm
500 µm
500 µm
20 µm
10 µm
20 µm
2 µm
2 µm
2 µm
500 nm
500 nm
500 nm

Fig. 4

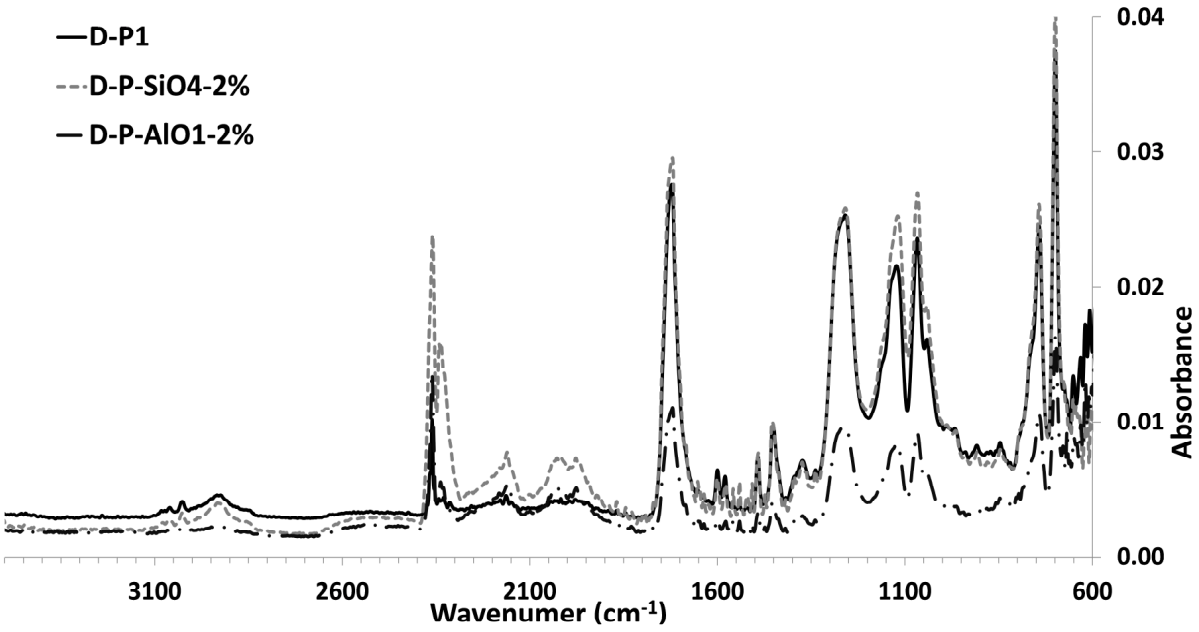
D-P1
D-P-SiO4-2%
D-P-AlO1-2%
0.04
0.03
0.02
0.01
0.00
Absorbance
3100
2600
2100
1600
1100
600
Wavenumer (cm-1)

Fig. 5

Table 1 Samples used for the drilling experiments in the CNC prototype

Sample	Matrix	Nanofiller	Content
D-P	polyester	--	--
D-P-SiO4-2%	polyester	nano-silica	2 wt.%
D-P-AlO1-2%	polyester	Nano-alumina	2 wt.%

Table 2 Total number concentration of particles for the experiments with the manual drill

Number concentration of particles C (cm^{-3})	Air recirculation	No Air recirculation
Inside Chamber	411	560
Manual Drill	4140	65015

Table 3 Number concentration of particles C (cm^{-3}) measured during the baseline test and during drilling experiment

Sample	Number concentration of particles C (cm^{-3})	
	Baseline	Drilling
D-P	1639	1796
D-P-SiO4-2%	1104	944
D-P-AlO1-2%	1134	1219

Table 4 Characteristic peaks in FTIR of polyester, nanosilica and nanoalumina [23-26]

Polyester		Nanosilica		Nanoalumina	
Band (cm^{-1})	Assignment	Band (cm^{-1})	Assignment	Band (cm^{-1})	Assignment
3448	O-H stretch	3457	O-H stretch in silanol hydroxyls	1200-950	Al-O-M bonds
3060	Aliphatic C-H stretch	1268-1132	Si-O-Si stretch	3092	-OH groups in alumina
3026		966	Si-OH bond	2090	
2982		959	Si-O asymmetrical stretch in SiO_4	1920	
1728	C=O stretch	938			
1599	Aromatic ring stretch	875			
1580		810	Si-O asymmetrical stretch		
1493		525	O-Si-O out-of-plane bending		
1453	CH_3 asymmetrical bend	511			
1380	CH_3 asymmetrical bend	450	O-Si-O in-plane bending		
1284	CH_2 twist				
1121	C-O stretch				

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Development of CNC prototype for the characterization of the nanoparticle release during physical manipulation of nanocomposites

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