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Evaluation of Anti-Fouling Behavior of Silver Nanocomposites Made by Nano-Coating Fragmentation

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Abstract. Nano-coating fragmentation (NCF) is patented technology which allows producing thermoplastic matrix nanocomposites without the step of nano-particle preparation. Thermoplastic pellets are PVD (physical vapor deposition) coated by the metal of the desired nano-reinforce. In the following processing step, by extrusion or injection molding, nano-coatings are fragmented into nanoplatelets because of the action of the screw. In this study, nano-silver (Ag) filled nanocomposites with polypropylene (PP) matrix have manufactured by this innovative technique and tested in open environment for their anti-fouling behavior. PP pellets have been PVD coated into a large chamber with the aid of a rotating drum. Coated pellets were physically mixed with virgin in the percentage of 0, 5, 10, 20, and 100%. Consequently, the expected Ag percentage ranged from 0.036% wt to 0.103% wt. Square nanocomposite samples (80x80 mm^2 and 3 mm thick) were injection molded in a fully electric press. One sample for each nano-Ag content was selected to be exposed in open environment. A smart buoy, especially designed for water cleaning and monitoring, has been used for experimentation. Results show that Ag-NPs provide a significant contribution to reduce the growth of vegetation on the molded plastic surfaces. However, at very low contents, the negative effect of the Ag NPs on the surface morphology of the molded samples nullifies this contribution.

Introduction

The current critical issues on the presence of bacterial activity and different micro and bio-organisms on the surfaces of everyday life objects, which also damage marine structures require the use of proper strategies and materials to solve these criticalities. The presence of bacteria and harmful organisms is very dangerous for human health but also for the right operation of several devices. These issues are deeply felt in the fields of biomedical devices, tissue engineering, marine structures, packaging and electronic devices. In this view different materials with anti-bacterial and anti-fouling properties are properly designed and fabricated. Specifically, anti-bacterial behavior is referred to as the property of impeding the growth and proliferation of bacteria over surfaces through direct actions aiming at inhibiting bacterial growth or breaking the bacterial membrane. On the other hand, anti-fouling behavior is referred to as the property of avoiding the accumulation of organic and biological organisms (algae, bacteria, marine fouling such as molluscs) onto surfaces. The anti-fouling behavior is obtained by developing hierarchical surfaces, specifically super hydrophobic, which impede the settlement of organisms. On the other hand, the anti-bacterial behavior is obtained by releasing chemicals which react with the bacteria constituents [1, 2]. For these aims, current trends use nanomaterials (NMs) because they have optimal properties such as high surface area and area to volume ratio and consequently high reactivity [3]. Both inorganic and organic NMs are currently used. Among inorganic materials there are either metal oxides such as copper oxide (CuO), zinc oxide (ZnO), titanium dioxide (TiO_2), or metal-based NMs like gold (Au), silver (Ag), iron (Fe) and copper (Cu). Among organic based NMs graphene oxide (GO), carbon nanotubes (CNTs), nanodiamonds, cellulose nanocrystals and graphitic carbon nitride are commonly used. There are also 2D materials made of transition metals (M) and carbon and/or nitrogen (X) (MXene), metal organic frameworks and polymer nanocomposites (PNCs), that are polymers filled with nano-additives. All these materials are used in anti-fouling applications such as marine structures, water treatment and medical devices [4]. Polymeric materials with anti-bacterial and anti-fouling properties are also commonly used and modified by the addition of inorganic particles or active molecules. These additives allow modifications of the commonly developed polymeric membrane obtaining nanocomposite membranes (NCMs) with enhanced thermal stability, magnetic behavior and strength [5]. They are mainly fabricated with Polyethylene Glycol (PEG). Poly(oligoethylene glycol) Methacrylate (POEG-MA), Poly(2-oxazoline) (POx), Polyglycerol (PG), polyvinylidene fluoride (PVDF), polyvinylpyrrolidone (PVP) and zwitterionic polymers [6]. Apart from virgin materials, also recycled materials can be used for this aim. In fact, barrier properties of recycled polyethylene terephthalate (PET) sheets have been enhanced by aluminum (Al) nano-coating obtained through physical vapor deposition (PVD) sputtering [7]. Moreover, the commonly adopted technologies for polymers' processing can be redirected for PNCs with enhanced anti-bacterial and anti-fouling behaviours. These processes include melt processes, solvent casting, electrospinning, solution blow spinning, solid state methods (high energy ball milling), sputtering, plasma treatment and nanopatterned surfaces [8].

In polymeric materials a commonly implemented strategy is using nanoparticles (NPs) as additives to enhance the anti-fouling and anti-bacterial behaviours. The presence of metal additives like Ag, develop Ag^+ ions that react with the bacteria surface inhibiting their action. Another common solution is reducing Ag in nanoparticles (AgNPs) to be added in polylactic acid composite fibers containing cerium oxide to enhance antibacterial performances [9] or used in combination with TiO_2 to be stabilized and using (3-Aminopropyl) triethoxysilane (APTES) crosslinking for holding with PVDF membrane [10]. TiO_2 can also be used in ternary nanoparticle membrane with Ag, TiO2 and multiwalled carbon nanotubes (MWCNTs) $AgTiO_2MWCNT$, embedded in PVDF/PVP membrane so enhancing the anti-fouling performances [11]. Membrane's anti-fouling properties can be also enhanced through the incorporation of other NPs such as Ag, Fe, silica, Al, Ti, Zn, Cu and their oxides [12]. Bifunctionalized zeolites (FAU) with hydroxylic groups (OH) and silver ions (FAU-Ag-OH) were incorporated through interfacial polymerization into a polyamide (PA) layer of a thin film NCM increasing its water permeance for wastewater treatment management as well as anti-biofouling ability with a flux recovery ratio up to 85% [13]. An easy and fast deposition of AgNPs can be achieved by electrospray even if the lack of knowledge on the behaviour of engineer NMs requires deep investigation on process parameters. Good results can be obtained by coupling electrospray with polymeric bonding to activate reaction mechanisms able to enhance the functionalization of permeable membrane [14]. AgNPs can be also treated by amine, thiol, carboxyl, and the thiol-polyvinylpyrrolidone functional groups to enhance its immobilization inside the polymeric matrix of NCMs for membrane bioreactor [15] and improving anti-microbial effect. In situ reducing of AgNPs taking advantages by phenolic hydroxyl groups of substances such as urushiol-based benzoxazines are generally adopted ways to obtain composite coatings [16].

Moreover, the in-situ reducing of silver nitrate ($AgNO_3$) on the surface of GO with sodium borohydride three ($NaBH_3$) has been used to prepare graphene oxyde with AgNPs (GOA) and was dropped coating in polypropylene matrix for sensor housing applications [17]. The same in situ reducing can be obtained on polyurethane (PU) substrates by a polydopamine coating directly applied to PU surfaces [18]. Wetness impregnation of silica, titania and mechanically mixed silica-titania powders was used to prepare NMs based on Cu and Ag nanoparticles and they have been further bonded with a commercial topcoat [19]. Wet impregnation of CNTs with AgNPs and further vacuum filtration on a PVDF substrate also allow enhancing anti-fouling performances [20]. Spin coating of a hybrid polymer nanoparticles mixes based on poly vinyl alcohol (PVA)-glutaraldehyde (GA) and $Ag\text{-}TiO_2$ onto a commercial gel coated fiber reinforced polyester substrate revealed the higher anti-fouling properties when compared to AgNPs and TiO_2NPs singularly [21]. Very recent trends aimed at developing even more advanced materials able to release Ag ions to interact with bacteria as for the new composite with a core shell structure of halloysite by loading AgNPs and subsequently coating with chitosan. Chitosan serves as a ph-sensitive gatekeeper to release silver ions [22].

All the mentioned techniques are mainly diffused at a lab scale and realize AgNPs in a separate step; unfortunately, this choice makes the transfer to industries difficult. An innovative approach aiming at developing AgNPs within polymers during the manufacturing processes has been developed, patented and termed as nano-coating fragmentation (NCF) [23. 24]. This process produces nanometric metal-based layers onto polymeric precursors (pellets) via a PVD sputtering process, which are then properly mixed with virgin PP pellets and processed by extrusion or injection moulding. In this way, PNCs with enhanced anti-bacterial properties can be obtained [25, 26]. This approach can be used also for producing a new class of composite starting from biopolymers [27]. Moreover, a non-destructive technique based on pulsed phase thermography has been also used to evaluate the dispersion of the Ag content in the bulk material [28].

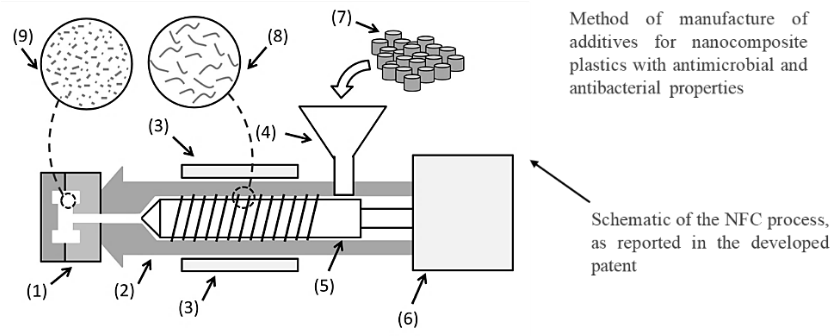
In the current study the NCF process has been used to produce different samples of polypropylene-based nanocomposites with different content of AgNPs. The developed PNCs were placed on a new concept of smart buoy equipped also with oleophilic filters, obtained by cold compaction of pyrolytic carbons by recycling of tyres [29]. The buoy was settled in open environment with the expected presence of several harmful substances and organisms to evaluate the anti-fouling properties.

Materials and Methods

Materials. The PNC plates used for exposure in a harsh environment are obtained from commercially available materials. The matrix is an injection-molding-grade polypropylene (PP) (Moplen HC500N, Lyondellbasell Europe). It is supplied in the form of pellets and commonly used in industrial applications such as nonwoven fabrics, biomedical devices, packaging and so on. Moreover, pellets have a nominal density of 0.9 g/cm^3 and a characteristic dimension (diameter) ranging from 2.5 mm to 4.8 mm. The reinforcement was a 99.99% pure silver (G. Gambetti Kenologia Srl, Italy) which was used as a rectangular target with nominal dimensions of 300×125 mm^2 for PVD sputtering.

Nanocoating Fragmentation and PNCs Manufacturing. The manufacturing of the PNCs is obtained through the patented solution shown in Figure 1. The process is divided into two main steps which are shown in Figure 2. The first step consists in obtaining nanometric silver coating on PP pellets via a magnetron sputtering PVD system (MITEC s.r.l., Italy) with 400 W as DC power input, 20 min as deposition time, and 29 rpm of rotation speed of the pellets during the sputtering phase. These parameters already used in a previous study [26] allowed to deposit an Ag percentage of 0.046 ± 0.001% over the pellets' surface.

In the second step, or molding step, the obtained coated pellets are first mixed with virgin pellets of the same polymer in properly chosen proportions and then processed by extrusion or injection moulding. During the process, shear forces fragment the pellets and AgNPs are directly dispersed inside the polymer to obtain PNCs with enhanced anti-bacterial properties.



Method of manufacture of additives for nanocomposite plastics with antimicrobial and antibacterial properties

Schematic of the NFC process, as reported in the developed patent

Fig.1. Patent for nano-coating fragmentation [30].

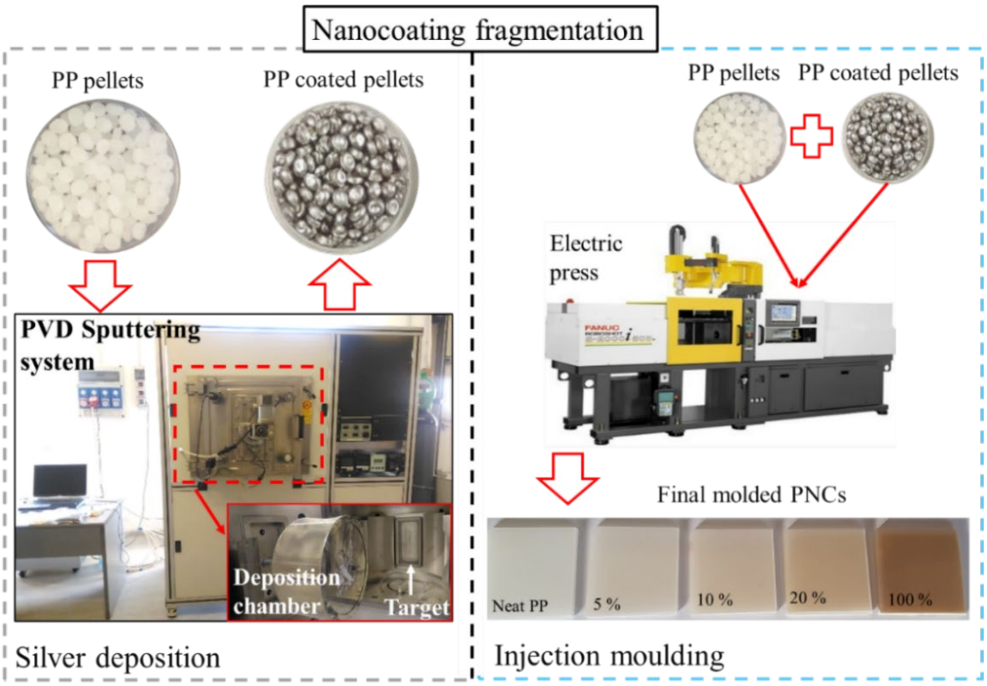


Fig. 2. Procedure for manufacturing anti-fouling PNC plates.

This second step was performed through an electric press Fanuc Roboshot S-2000i50B of 50 t (Fanuc Corporation, Japan). Square plates with nominal sizes of 80×80 mm^2 and nominal thickness of 3 mm were produced either with only PP pellets, termed as Neat and either with coated pellets mixed with virgin ones and termed as PNCs. Specifically, the coated pellets were mixed with virgin ones in 4 different amounts, that is in percentage of 5% wt, 10% wt, 20% wt and 100% wt with the virgin ones. These percentages correspond to Ag contents varying from 0.036% wt to 0.103% wt. The adopted Ag can be considered in the form of nano particles as the sputtering step allowed to obtain a nanometric coating of about 25.3 ± 0.5 nm.

Microscopic images of the moulded samples before and after testing have been acquired through a 3D digital microscope Hirox HR-2016E (Hirox, Japan).

Design of Smart Buoy. The PNC plates were housed around the perimeter surface of a smart buoy and mechanically joint through screws. The buoy is properly designed and produced to house oleophilic filters obtained by pyrolytic carbon from tires' recycling. In the current study such filters were replaced by PNC square plates. The smart buoy's configuration guarantees a fixed waterline to wet the nanocomposite plates for 1/3 of their height. Moreover, the buoy has a nominal diameter and height of 610 mm and 390 mm respectively and a nominal mass of 7 kg.


Buoy equipped with anti-fouling plates

Beginning of exposure test: January 2025

End of exposure test: September 2025

Fig. 3. Positioning of the PNC samples on the smart buoy, by replacing some pyrolytic carbon filters, and exposure in open environment for the experimentation.

Exposure in Harsh Environment. The anti-fouling behaviour of the manufactured PNC plates was investigated after 8 months of exposure in a drain of an industrial site, which could be potentially affected by different kinds of micro (i.e. bacteria) and macro (i.e. algae) living organisms, and suitable for understanding the whole properties of the produced nanocomposites. The anti-fouling system layout and the selected harsh environment are shown in Figure 3. After exposure, the buoy was retrieved and the PNC samples removed for analysis.

Results and Discussion

The appearance of the exposed samples is reported in Figure 4a). They have been partially cleaned by soft water flow because of the presence of many residuals, not stuck on the sample surfaces. In fact, during 8 months in the small water channel of Figure 3, the buoys have experienced many intense natural events such as floodings and shallows. Moreover, it is reasonable that samples shown in Figure 4a) have been subjected to more severe conditions for longer times than others which have been previously tested in laboratory, as the buoy tended to take a stable position into the water flow. Direct sun exposure or shadow presence could have affected differently the sample surfaces, but a direct quantitative effect evaluation was difficult to perform. Infect, during the 8 months of exposure each sample was subjected to different and non-quantifiable sun exposure or shadow presence. Nevertheless, the very long exposure time has been able to show such interesting occurrences. A part of 1/3 of the samples has been fully immerged into the water, and this is clearly visible in all the samples, as the largest amount of vegetation (green and brown color) is present in this part. Nevertheless, samples with 20% and 100% coated pellets, during manufacturing, show very small traces in comparison with the others. Moreover, these samples are almost fully clean on the 2/3 rest of the surface, differently from the samples with 0%, 5% and 10%. The positive effect of the Ag NPs seems to be verified from this first experimentation. The most affected sample is that with 10% of coated pellets, as the neat sample is better. Microscopic images of the specimen with 10% and 20% of coated pellets, which exhibited a bad and good anti-fouling behaviour respectively, are shown in Figure 4b). Apart from possible fluctuations of the exposure conditions, it is possible to identify a mechanism. In fact, NPs partially affects the roughness of molded surfaces.

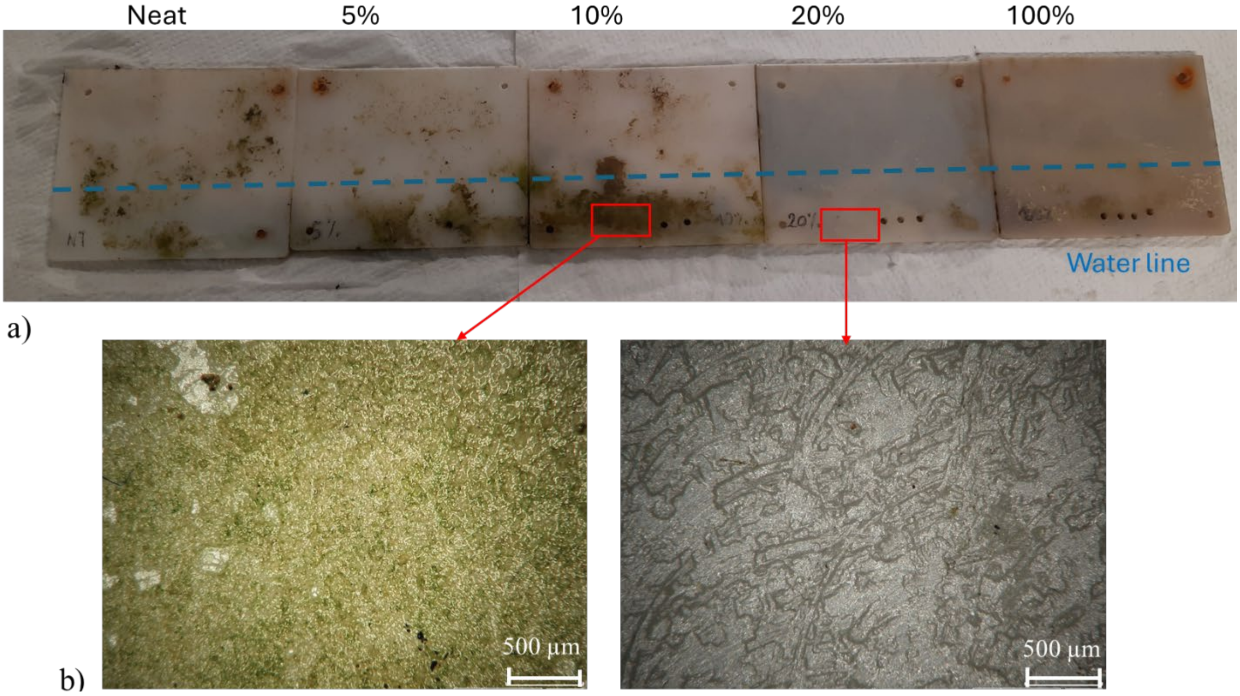


Fig. 4. PNC samples after their exposure for 8 months in open environment a) and microscopic images of the sample with a bad (10%) and with a good (20%) anti-fouling behaviour b).

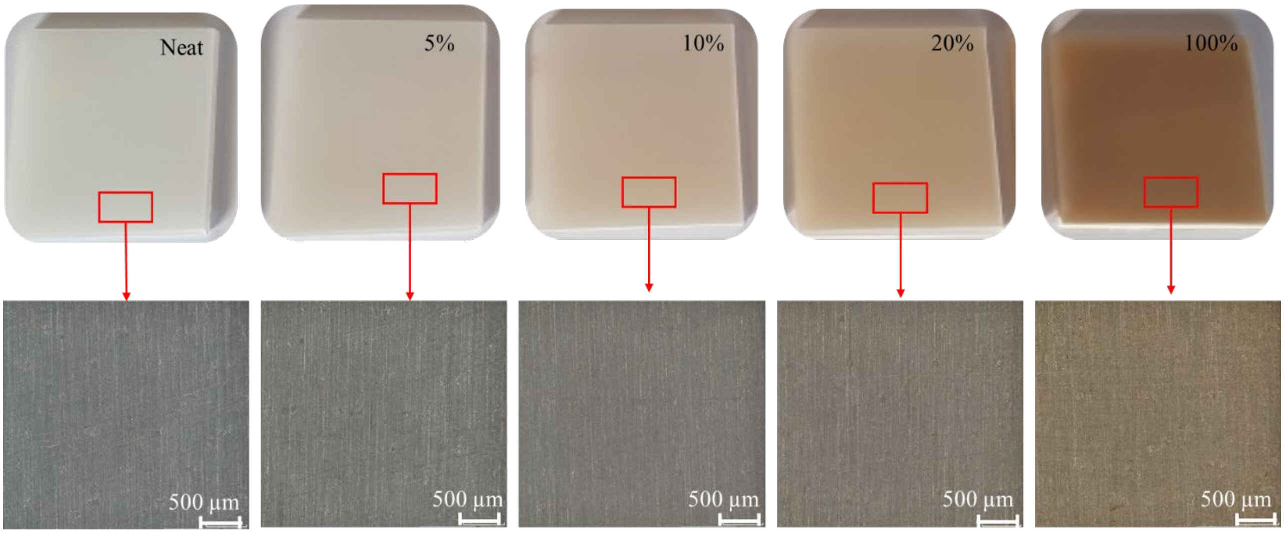


Fig. 5. Microscopic images of all samples before long term exposure.

The worst performances of the samples with 5% and 10% of coated pellets depend on the surface morphology. The average roughness Ra and the average height of the profile elements Rz of all sample range between narrow intervals, being 0.4-0.45 µm for Ra and 2.9-3.4 µm for Rz respectively. On average, samples with 5% and 10% coated pellets exhibited higher values. Moreover, the mean spacing of profile elements Rsm exhibited higher values for the samples with 5% and 10% of coated pellets as well and, among these, the highest values were measured for the specimen with 10% of coated pellets [26]. Higher Rsm values are correlated to the development of larger stagnation zones that promote the accumulation of marine vegetation. Samples with 20% and 100% coated pellets, did not exhibit a significant increase in roughness and had lower values or Rsm. Instead, roughness had small values in the case of virgin polypropylene and this characteristic, together with the hydrophobicity of this polymer, provides a certain degree of anti-fouling to molded PP products, at least at low times of exposure. Rough surfaces improve the adhesion of living organisms, partially reduce this intrinsic anti-fouling behavior. In Figure 5 a comparison of microscopic images of all samples before their exposure is shown. Surface textures are similar for all samples due to the injection moulding process. By adding Ag NPs, there are 2 opposite effects, one is on the plastic surface morphology which affects the intrinsic PP anti-fouling, and the other is the anti-fouling behavior of silver. In the end at very low content, a positive effect of the Ag NPs is covered by the worsening of the surface morphology, whereas, at higher contents, this positive effect becomes predominant. The injection moulding process develops surface morphology with variation of Ra, Rz and of Rsm which reduces the positive effect of the Ag NPs. Surface morphology can be considered a significative factor influencing the anti-fouling behaviour if a correlation between topological parameters and biological activity can be found independently by the chemical composition of the developed PNCs.

Summary

In the current study, the anti-fouling behavior of molded polypropylene nanocomposites, made by the patented nano-coating fragmentation technique, has been tested in open environment for a long time. Results show that nanocomposites may provide a positive anti-fouling effect, if a given threshold in the Ag NPs content is reached. In fact, at very low contents, the altered surface morphology of the molded plastic can cover the positive contribution of the nanoparticles. However, silver content remains very low in absolute terms, reaching 0.1% wt in the highest case.

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