Novel thin functional coatings for paper by foam coating

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ABSTRACT: Conventional coating methods are currently being challenged by newly developed technologies that can result in products with interesting properties and competitive prices. Versatile foam coating is one such promising technology. Foam coating is carried out using different foam application methods, and the viscoelastic nature of the foam allows the use of a large variety of materials. Paper was coated with nanomaterials on a pilot scale using a method based on slot die application combined with foam destruction by absorption and infrared drying. A thin layer of titanium dioxide/zinc oxide (TiO_2/ZnO) functionalized cellulose nanofibrils (CNF) creates antimicrobial properties in the paper, and a thin layer of CNF-TiO_2 alone has substantial ability to oxidize nitric oxide (NO) and nitrogen oxides (NOx). These results illustrate methods that can be used to create novel functional properties for different end-use applications. Thin functional layers could bring added value to current products and also create opportunities to introduce novel products for several industrial areas using web-type manufacturing processes.

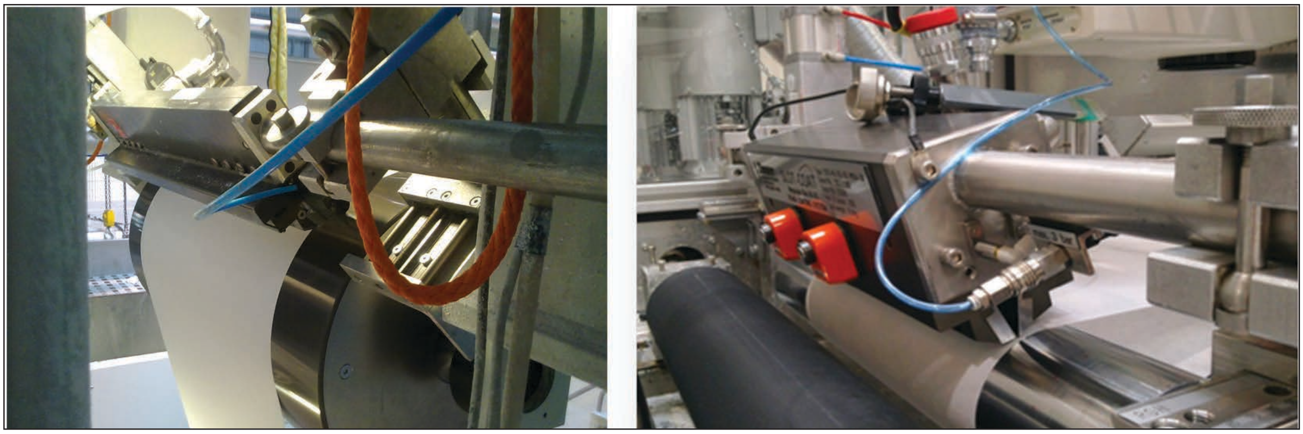
Application: The versatile foam coating method, using foam as a transferring media, allows manufacturers to modify web surfaces with a large variety of materials, from nanoparticles to highly viscous material such as cellulose nanofibers.

In foam coating, foam is commonly used as a carrier phase for applied chemicals or dispersions. In the textile industry, a wide variety of foam application techniques have been developed [1-9]. The foam application method reduces the problems caused by large amounts of liquid medium used in conventional treatments [10]. In paper applications, dispersions containing microcapsules with the natural foaming agents, gelatin, casein, and soybean extract have been applied [11]. Materials such as starch, wet strength resin, polyvinyl alcohol, wet end sizing agent, and direct dyes, as well as correction of an inherent curl in one-side-coated papers by water, have been applied using the foam coating method [12-14]. This system, referred to as the Foamcote system, is described in patents [15,16]. Eklund and Hobbs presented a more flexible application system for applying foamed starch to a paper web at high speeds [17].

Our earlier work [18-24] clearly shows that foam coating is a suitable method for applying nanoscale material to fiber-based substrates, and the method has several benefits compared with spray or film transfer technologies [21]. The technology offers a unique method for applying very light coat weights to the web surface with exceptional uniformity and absence of coating strike-in. Our earlier studies also showed that even a small amount of organic nanomaterial could change the surface properties of the paper [21]. The characterization of the coating layer having a small amount of material on the surface is challenging; however, in the case of inorganic particles, a combination of microscopic and spectroscopic procedures can be used for the analysis [23]. A thin layer of unmodified cellulose nanofibrils (CNF) increased hydrophilicity of paper and closed the paper surface, making it smoother and slightly less permeable [20,21].

Silver has been known to be an antimicrobial agent. Polycrystalline titanium dioxide (TiO_2) powders, on the other hand, have been intensively used as white pigments in diverse applications, including papermaking [25]. The photocatalytic properties of TiO_2 were first discovered 40 years ago [26]. TiO_2 is also a photoactive material with the ability to promote oxidation of organic adsorbates at the surface because of photogenerated electron-hole pairs after ultraviolet irradiation [27, 28]. The optical activity of TiO_2 has also been intensively exploited in the field of heterogeneous photocatalysis and solar energy conversion. Moreover, extended studies have been carried out on photodegradation of volatile organic compounds. More recently, there has been great interest in TiO_2 as a photoactive antimicrobial agent. In addition, the antimicrobial properties of zinc oxide were considered to be of interest [29].

In this paper we describe how the aforementioned properties of TiO_2 and zinc oxide (ZnO) were exploited for the first time in CNF-based products by foam coating of paper [24]. In addition, we tested the thin layer of CNF-TiO_2 alone for the oxidation of nitric oxide (NO) and nitrogen oxides (NOx).



1. The narrow slot-type foam applicator. The left side applicator has a labyrinth design, and the foam enters the applicator chamber in the middle of the unit. The right side applicator has a chamber design, and the foam enters the applicator from both ends.

2. Cellulose nanofibrils (CNF) with solid content 2.9 % (left), and foamed CNF containing 90% air (right).

EXPERIMENTAL

In the foam coating process, dry foam with air content greater than 80% and preferably 90%–95% (corresponding to foam densities of 50–200 g/L) is used as a carrier phase for the coating material. The coating material is located in the bubble pockets (vertices) and on the bubble surfaces. The main components in a foam coating line are a foam generator and a foam applicator. These trials used the Top-Mix type 60 foam generator (Hansa Industrie-Mixer GmbH & Co. KG; Stuhr, Germany). The starting material feeding capacity of the generator is 6–60 kg/h, and it can produce foam with densities of 50–400 g/L. The generator is connected to the compressed-air pipeline of the pilot plant. The coating material and foaming agents are mixed with pressurized air in a mixing head equipped with a rotor-stator unit.

For the application of the foam, two different Magnojet narrow slot-type applicators (J. Zimmer Maschinenbau GmbH; Klagenfurt, Austria) were employed and are shown in **Fig. 1**.

Foam coating trials

In the trials of CNF functionalized with nanoparticles, we used a noncontact, curtain-type foam application method, where foam collapses through the absorption of the paper web and through infrared (IR) drying. This method is suitable especially for small application amounts with hydrophilic substrates with fast absorption properties.

The trials were performed at the Zimmer coating line in Austria. The chamber-type applicator was used in the trials. A 400-µm gap between the slot and the moving web was used. The IR drying, with the dryers on both sides of the web, was followed by drying with hot-air ovens at a temperature of 150°C. The maximum speed of the line is around 180 m/min, but the coatings were applied at 100 m/min. This coating speed allowed for the low consistency of CNF coating to achieve the desired coat weights, while providing enough speed to obtain a good quality coating layer. The applied coat weights in trials at Zimmer were 0.4–1.8 g/m^2. The maximum speed we have obtained so far with the KCL pilot coater is 400 m/min (KCL Pilot Plant; Espoo, Finland). Technical improvements are nonetheless needed to increase the speed and to improve the evenness of the coating layer.

Property	±	TiO_2	ZnO
Concentration, %w/w	0.5	6.0	1.0
Density, g/cm³	0.05	1.20	1.12
Viscosity (25°C), mPa/sec	0.1	2.00	ND
Particle dimension, nm	-	40.0	45.0
Polydispersity index	0.05	0.25	0.20
pH	0.5	1.0	ND
Cationic surfactant, %w/w	0.05	0.1	-

I. Physicochemical characterization of inorganic nanoparticles (TiO_2 = titanium dioxide; ZnO = zinc oxide).

Materials

Functionalized cellulose nanofibrils

This work was carried out as part of the European SUNPAP (Scaling Up Nanoparticles in Modern Paper Making, 2009-2012) project targeting CNF applications. However, other nanomaterials, such as silica, can be used as a carrier for the functional materials. CNF are nanosized cellulose fibrils with a high aspect ratio (length-to-width ratio). The width of fibrils is typically 5–20 nanometers and the length ranges widely from tens of nanometers to several microns. The more nanoscale particles present in the CNF material, the more viscous and transparent the material will be. The benefit of foam coating is that high-viscosity CNF does not need to be diluted for the coating process. **Figure 2** shows the case where the starting CNF had a solids content of 2.9% and did not flow by itself into the screw pump. However, the foamed CNF from the generator (with an air content of 90%) was a suitable material for the coating process. The foamed CNF was pushed manually to the pump through the pipe (10 cm length and 4 cm diameter), combining the feeding tank and the screw pump.

The CNF for the trials was produced by the Centre Technique du Papier, Gières, France (CTP). The CNF fibers were produced by an initial enzymatic pretreatment of cellulose fibers followed by low consistency refining. Then, the pretreated and refined fibers were disintegrated to nanoscale particles by running them through a homogenizer several times. This produced CNF with a solids content of 2.3%, which was then functionalized by Innovhub (Milan, Italy). For functionalization of the CNF, inorganic nanoparticles TiO_2 and ZnO were produced by Colorobbia (Vinci, Italy). **Table I** shows the physicochemical characterization of inorganic nanoparticles.

The functionalization of CNF was achieved on the basis of direct physical adsorption of inorganic nanoparticles to CNF. The nanocomposites were prepared by mixing CNF (2.3% in water) with TiO_2 (6% in water) or ZnO (1% in ethylene glycol), or both, for 15 min. The mixtures were then washed three times with water followed by centrifugation to eliminate non-adsorbed inorganic fillers. CNF and inorganic nanoparticles suspensions were mixed together as supplied by an immersion mechanical blade stirrer. Centrifugations were performed and washings were carried out to eliminate excess TiO_2 or ZnO. After each washing, homogenization by stirring was carried out. The centrifuge took approximately 2 min to reach 7000 rpm, which was then maintained for 5 min. After centrifugation, the water supernatant was discarded. The pellet produced was suspended in the same volume of fresh water that had been removed by ultracentrifugation and then mixed with the mechanical stirrer. For the trials, 25 kg of CNF-TiO_2 (2.8% dry weight) and 15 kg of CNF-ZnO-TiO_2 (4.8% dry weight) were produced. The concentration of TiO_2 and ZnO in the final nanocomposites was determined by inductively coupled plasma analysis. **Table II** shows the mixing ratios and the contents of inorganic nanoparticles in the prepared composites.

The foams were produced by mixing the foaming agent and CNF containing nanoparticles with pressurized air in the mixing head of the foam generator. The air content of the foams was adjusted to 80%–90%, with foam density 100–200 g/L. The anionic foaming agent used was sodium dodecyl sulfate, in a ratio of 0.2% of CNF composite volume. The addition of a surface active agent was on the basis of the liquid volume.

Base paper and paperboard

The base paper used in the CNF trials was 80 g/m² uncoated and uncalendered fine paper produced from hardwood and softwood fiber without hydrophobic sizing agents with fast absorption properties.

Characterization

Using the paper samples coated by CNF-TiO_2 or CNF-ZnO, or CNF- TiO_2 and ZnO, the following tests were performed:

Nanocomposite	Solids Content, %	Initial Mixing Ratio of the Preparations, as dry weight	TiO_2 Content, % dry weight	ZnO Content, % dry weight
CNF-TiO_2	2, 8	1:1	24	-
CNF-ZnO and TiO_2	4, 8	10:1:10	42	2

II. Analysis of CNF/inorganic nanoparticles suspensions (TiO_2 and ZnO determined by inductively coupled plasma analysis).

Data Type	Counts
Image resolution	1024 by 1024
Image pixel size	1.06 µm
Map resolution	256 by 256
Map pixel size	4.24 µm
Alternating current (AC) voltage	12.0 kV
Magnification	100

III. The information of scanning electron microscopy analysis of TiO_2/ZnO modified CNF–containing paper.

- Antimicrobial activity (AATCC Test Method 100 "Quantitative assessment of antibacterial activity") toward gram-positive *Staphylococcus aureus* and gram-negative *Klebsiella pneumoniae* [24].
- Photocatalytic activity by using a device designed by Colorobbia Italy [24].

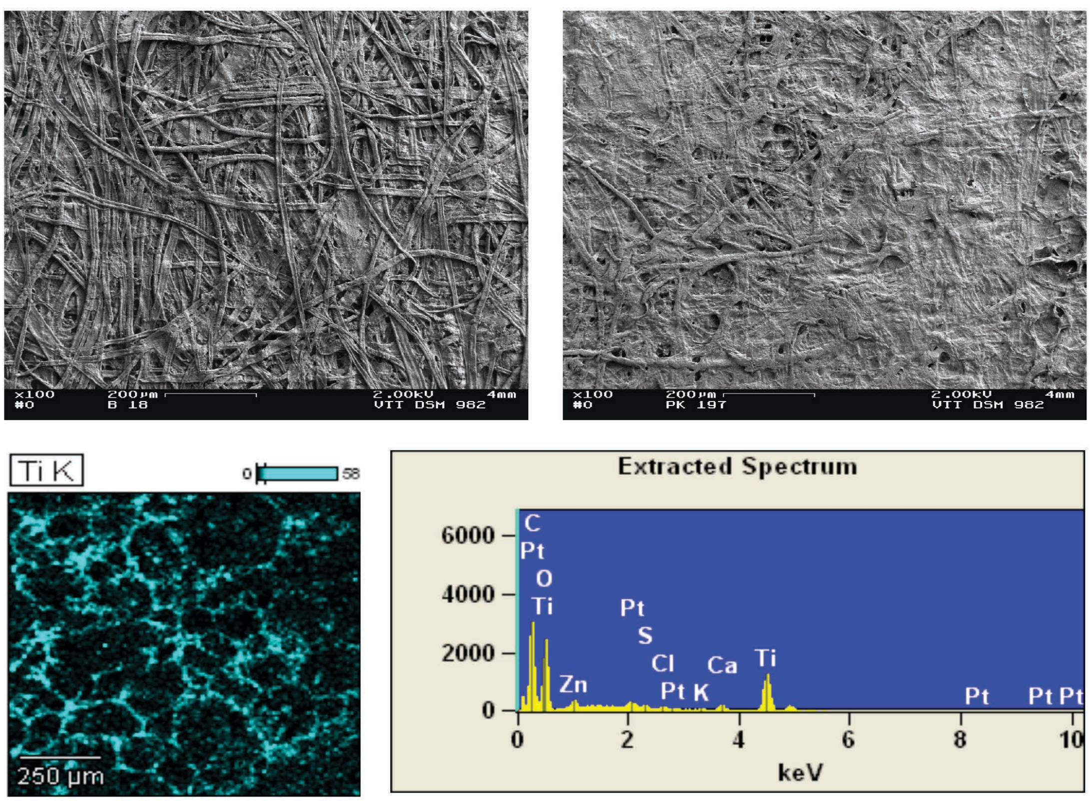
Before the tests, the TiO_2/ZnO modified CNF-containing papers were analyzed with scanning electron microscopy (SEM) imaging using a backscattered electron (BSE) detector and titanium element mapping. The information of SEM analysis is seen in **Table III**.

Titanium element maps show that material on a paper surface is attached on the fiber surfaces to form a structure that resembles a honeycomb (**Fig. 3**).

Antimicrobial activity of the foam-coated papers

Photoactivation

In the presence of TiO_2 and ZnO nanocomposites, the antibacterial tests were carried out on photoactivated samples as follows. The samples were sterilized before testing and the antibacterial tests were conducted by spreading a known number of living cells on the paper surface to yield a population of 10^5 colony forming units (CFU). The paper samples were subjected to light exposure (4 h by standard solar lamp 6000 lux) to induce photoactivation of inorganic nanoparticles (TiO_2 or ZnO, or both). Then the samples were incubated by adopting testing conditions (temperature and nutrients) that promote bacterial growth. In addition, some tests were also run in darkness. The inoculated paper samples were maintained 20 h under optimal nutrient and temperature conditions for bacterial growth. At the end of the test, the paper samples were extracted and the number of living cells determined by plating serial dilution on nutrient agar.



3. Titanium element scanning electron microscopy/energy-dispersive x-ray spectroscopy (SEM/EDS maps) from calendered TiO_2/ZnO modified CNF–containing paper showing that material on the paper surface is attached on the fiber surfaces (top left–base; top right–coated surface).

4. *The photodegradation kinetics of nitrogen oxides (NOx) in gaseous phase by foam-coated papers were tested using a photoreactor equipped with chemiluminescence and gas chromatography–mass spectrometry.*

Testing conditions

The tests were performed according to AATCC Test Method 100 under static conditions. The foam-coated paper samples were tested for antibacterial activity toward gram-positive *Staphylococcus aureus* and gram-negative *Klebsiella pneumoniae*. The inoculated samples were subjected to overnight incubation (about 18 h) at 30°C or 37°C, depending on the optimal temperature for microbial growth. After incubation, the bacteria were extracted from the samples under investigation by using a neutralizing solution. CFU, the number of living cells in the extracted suspension, was evaluated by the count plate agar method. The antibacterial activity of the sample, as bacteria log reduction, was calculated as follows in Eq. (1):

$$\text{log reduction} = \log \text{CFU } T_{18} \text{ blank (control)} - \log \text{CFU } T_{18} \text{ sample} \quad (1)$$

where CFU T_{18} is the colony forming number corresponding to the bacteria living cells at incubation of 18 h.

In general, two antibacterial effects can be distinguished:

1. Bactericidal — Reduction (killing) of the number of bacteria initially inoculated (CFU at time 0).
2. Bacteriostatic — Inhibition of bacteria growth under testing conditions favorable to bacteria proliferation, evaluated with respect to growth in the blank sample (CFU at time 18 h).

Untreated CNF was used as a blank untreated control (CNF control).

Photocatalytic activity of the foam coated papers

The catalytic activity of the treated paper samples was tested using a device developed by Colorobbia Italy consisting of a glass chamber with a quartz window, in which the treated samples were exposed. In the chamber, a NOx-polluted atmosphere was created and the NOx concentration, humidity, and temperature were measured at fixed sampling time intervals. The measurement of NOx concentrations was carried out by a Model 42i chemiluminescence apparatus (Thermo Fisher Scientific; Waltham, MA, USA) (**Fig. 4**). The samples were continuously irradiated by an Ultravitalux 300W mercury vapor lamp (Osram Light AG; Munich, Germany) to induce photoactivation. The measurements were made by passing the polluted air passed through a closed circuit, including the reaction glass chamber, a bag in which the polluted air was stored, and a pump. During the entire experiment, the lamp remained on for activation of the samples. The data collected were used to assess the kinetics of the photocatalytic process.

RESULTS AND DISCUSSION

Antibacterial activity of the foam coated papers

Table IV reports the results obtained with the different paper samples (logarithmic reduction of the number of living cells) for tests run under light exposure conditions. Bacteria grown on the untreated reference is at a level of 10^6–10^7 CFU (6–7 log). One sample from earlier trials at KCL, namely the CNF/ZnO (0.8 g/m^2) sample, was tested at the same time with these new samples because the preparation procedure of modified CNF and the base paper were the same.

The results reported in Table IV show substantial antibacterial activity for paper samples treated by CNF-TiO_2 for TiO_2 content around 0.3%, while for paper samples treated by CNF-ZnO, a much lower inorganic filler content is needed to obtain similar effects. In general, bacteriostatic activity was obtained while bactericidal activity was not detected with these amounts of inorganic particles. Bacteriostatic activity was also demonstrated for CNF-TiO_2 foam-coated papers under conditions of darkness with respect to *Staphylococcus aureus*, as reported in **Table V**. The results show that in the absence of light, TiO_2 or ZnO modified CNF coated papers had only a slight effect on bacteria growth; that is, they need light exposure to show antibacterial activity.

Photocatalytic activity of the foam coated papers

The paper samples foam coated by nanocomposites CNF-TiO_2, CNF-ZnO, and CNF-ZnO and TiO_2 were characterized for their photocatalytic efficiency. As shown in **Table VI**, paper samples foam coated by nanocomposites containing TiO_2 display

Paper Sample (coat weight)	ZnO, ppm	TiO_2, %	*Staphylococcus aureus*		*Klebsiella pneumoniae*	
			Bacteriostatic Activity, log reduction	Bactericidal Activity, log reduction	Bacteriostatic Activity, log reduction	Bactericidal Activity, log reduction
Untreated paper	-	-	0	0	0	0
CNF-TiO_2 (0.4 g/m²)	-	0.034	0	0	0	0
CNF-TiO_2 (0.9 g/m²)	-	0.167	1.3	0	0	0
CNF-TiO_2 (1.8 g/m²)	-	0.291	Total bacteriostatic	1.0	1.8	0
CNF-ZnO and TiO_2 (0.6 g/m²)	38.4	0.115	0.7	0	0	0
CNF-ZnO&TiO_2 (1.3 g/m²)	104.6	0.307	Total bacteriostatic	0.1	0.2	0
CNF/ZnO (0.8 g/m²)*	180	-	Total bacteriostatic	0.3	2.1	0

* A sample from KCL coating machine trials. The procedure of CNF/ZnO composite preparation and the base paper were the same as in Zimmer trial samples.

IV. Antibacterial activity of foam-coated paper samples, under conditions of light exposure.

Paper Sample (coat weight)	ZnO, ppm	TiO_2, %	*Staphylococcus aureus*	
			Bacteriostatic Activity, log reduction	Bactericidal Activity, log reduction
Untreated paper (control)	-	-	0	0
CNF-TiO_2 (1.8 g/m²)	-	0.291	1.7	0
CNF-ZnO (0.8 g/m²)	180	-	1.2	0

V. Antibacterial activity of foam-coated paper samples, under conditions of darkness. Bacteria grown on untreated reference: 106–107 (6–7 log).

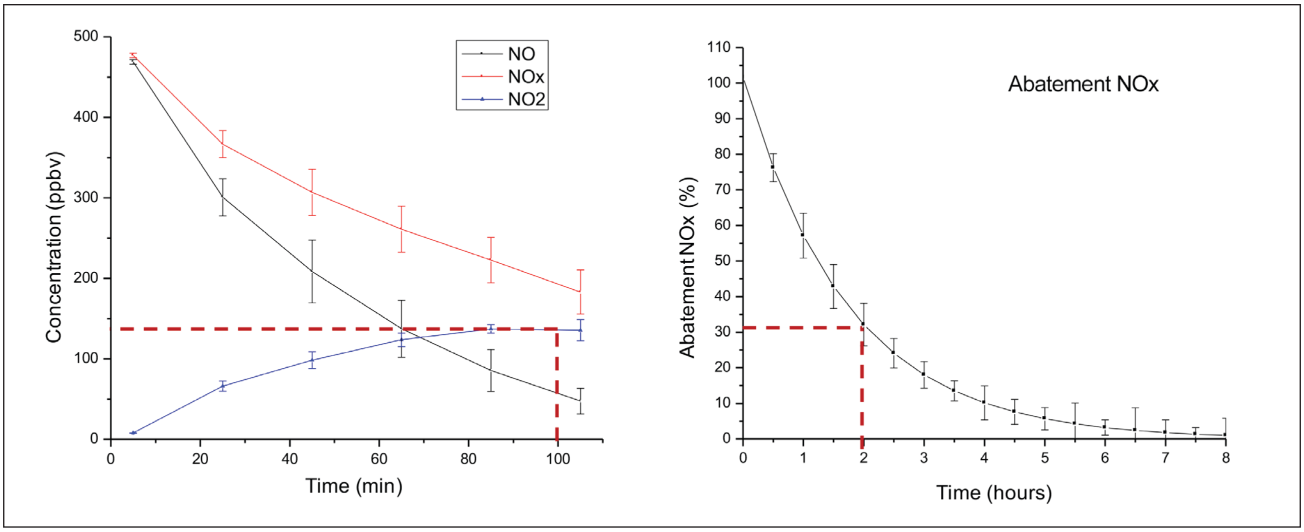
photoactivity for the degradation of volatile compounds at TiO_2 content higher than 0.1%. In contrast, ZnO presence seems to have little influence on the photoactivity of the paper. Photo-oxidation of volatile substances depends only on the TiO_2 content of the paper.

Figure 5 shows the NOx photodegradation in gaseous phase by CNF-TiO_2 foam-coated paper. Around 70% NOx oxidation occurred within 100 min, showing that foam-coated paper with CNF-TiO_2 had significant activity for the oxidation of NO and NOx. At a coat weight of 0.9 g/m², the corresponding TiO_2 dry weight on paper was 0.162%; this was enough to have an influence of 72.6% NO degradation after 105 min.

Photo-oxidation of NOx has important applications for the control of air pollution. In the case of active paper it could be used for wallpaper, for example, to control indoor pollution. However, the photo-oxidation by TiO_2 can also work to oxidize or breakdown organic volatile compounds (not only for inorganic materials such as NOx). These could include formaldehyde or other air pollution organic compounds (e.g., acetaldehydes) or ethylene produced by fruits that are responsible for the ripening and fast spoilage of fruits and vegetables in packaged produce systems.

CONCLUSIONS

Foam coating technology offers a unique opportunity to apply coating to a web surface thinly enough to be economically viable. The method is suitable for production of functional construction materials, filters, and disposable textile materials for hospital use or for active packaging solutions with antimicrobial, photoactivity, or barrier properties. The results ob-



5. NOx photodegradation in gaseous phase by CNF-TiO_2 foam-coated paper. Note: coat weight below 1 g/m².

Paper Sample (coat weight)	ZnODry Weight on Paper*, ppm	TiO_2Dry Weight on Paper*, %	NO Degradation (after 105 min), %	NO Degradation (after 105 min), %
Untreated paper	-	-	0	0
CNF-TiO_2 (0.4 g/m²)	-	0.008	16.8	12.6
CNF-TiO_2 (0.9 g/m²)	-	0.167	72.6	46.0
CNF-TiO_2 (1.8 g/m²)	-	0.291	79.9	61.6
CNF-ZnO and TiO_2 (0.6 g/m²)	38.4	0.115	81.7	64.9
CNF-ZnO and TiO_2 (1.3 g/m²)	104.6	0.307	90.9	78.5
CNF/ZnO (0.8 g/m²)	180	-	0	0

* From inductively coupled plasma analysis.

VI. Nitric oxide (NO) and NOx degradation by foam-coated papers.

tained from the foam coating trials with TiO_2 and ZnO functionalized CNF show that it is possible to create antimicrobial and photocatalytic properties of paper and paperboard.

The results obtained from the foam coating trials with functionalized CNF showed that by using modified CNF, it is possible to activate properties on the paper surface even at extremely low coat weights (i.e., below 1 g/m²). For the foam coating trials, functionalized CNF was prepared using a physical absorption based method. Nanoscale inorganic particles TiO_2 and ZnO were mixed with CNF to create nanocomposites CNF-TiO_2, CNF-ZnO, and CNF-ZnO and TiO_2 .

The paper coated by the functionalized CNF showed antibacterial activity mainly depending on the TiO_2 or ZnO content, or both. The antibacterial activities obtained were mainly bacteriostatic. The bacteriostatic property of CNF-TiO_2 or CNF-ZnO, or both, was obtained even at low coat weights of modified CNF. The TiO_2 content was around 0.3% dry weight on paper, and ZnO content was even lower.

Regarding the photo-oxidation of NOx compounds in gaseous phases, only paper samples coated by CNF-TiO_2 showed efficient photoactivity. At a coat weight of 0.9 g/m², the corresponding TiO_2 dry weight on paper was 0.162%; this was enough to result in a 72.6% NO degradation after 105 min. The degradation depended strongly on the TiO_2 content, while the paper samples containing only CNF-ZnO nanocomposite did not show any photoactivity, indicating that this additive is not sensitive to photoactivation.

Further work with more defined targets for different end applications, including product and process development at high speeds, would serve to optimize the effects reported here. **TJ**

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ABOUT THE AUTHORS

The paper and board industry needs new tools for renewal. We chose this research topic because foam based technologies in several application areas are such promising tools. This research is a continuation of earlier foam coating research focusing on the application of unmodified cellulose nanofibrils.

The most difficult aspect of this work was finding the facilities for starting the research at pilot scale. It was difficult because no relevant laboratory scale facilities exist. The most interesting finding was that the foam coating technology was able to transfer the effectiveness of the functionalized cellulose nanofibrils to the paper surface. The research shows that the foam based coating technology is very versatile. It allows the use of a wide variety of materials, which might challenge the conventional coating technologies.

For this author (Kinnunen-Raudaskoski), the next step is to continue product development based on foam technologies at Paptic Ltd., a spinoff company of VTT.

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