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One-step flame synthesis of silver nanoparticles for roll-to-roll production of antibacterial paper

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Abstract

Nanoparticles are used in several applications due to the unique properties they possess compared to bulk materials. Production techniques have continuously evolved over the years. Recently, there has been emphasis on environmentally friendly manufacturing processes. Substrate properties often limit the possible production techniques and, for example; until recently, it has been difficult to incorporate nanoparticles into paper. Chemical reduction of a precursor in the presence of paper changes the bulk properties of paper, which may limit intended end-use. In this study, we present a novel technique for incorporating silver nanoparticles into paper surface using a flame pyrolysis procedure known as Liquid Flame Spray. Papers precoated with mineral pigments and plastic are used as substrates. Silver nanoparticles were analyzed using SEM and XPS measurements. Results show a homogeneous monolayer of silver nanoparticles on the surface of paper, which demonstrated antibacterial properties against E. coli. Paper precoated with plastic showed more nanoparticles on the surface compared to pigment coated paper samples except for polyethylene-precoated paper. The results demonstrate a dry synthesis approach for depositing silver nanoparticles directly onto paper surface in a process which produces no effluents. The production technique used herein is up scalable for industrial production of antibacterial paper.

Keywords: silver nanoparticles, liquid flame spray, antibacterial paper, roll-to-roll processing

1. Introduction

The use of nanoparticles in recent years is a result of their unique properties compared to bulk materials, which enables functionalization of different products. Currently, properties such as superhydrophobicity, improved electrical and photocatalytic properties have been achieved by incorporating nanoparticles into various materials and products [1,2]. Silver nanoparticles have been known for their inherent antimicrobial properties, and they may play a key role in fight against the drug-resistant bacteria with increasing prevalence especially in the hospital environment [3,4]. Studies have also shown improved control of pathogenic bacteria when silver nanoparticles are combined with commercial antibiotics [5]. It is necessary to emphasize the different production techniques available for these nanoparticles, as they are continuously changing.

Production techniques for nanoparticles have evolved over the years, and environmentally friendly methods have been under focus [6]. Another significant consideration is the possibility to reduce or eliminate production waste. The use of harmful chemicals such as sodium bromohydride in the production of nanoparticles has been one of the disadvantages that has resulted in production techniques being unattractive [7]. Subsequently, green synthesis of nanoparticles has been of interest, and several studies are currently ongoing [8-10]. Other techniques that avoid the use of harmful chemicals can yield high production costs due to energy requirement and specialized equipment [11]. Additionally, the ability to produce nanoparticles in large quantities would subsequently encourage their use in numerous industrial processes. These factors have contributed to the continuous evolution of production techniques for nanoparticles.

Typical production methods for nanoparticles can be classified into three groups as follows: physical, chemical, and bio-based production methods [12]. Physical methods include the use of external energy in the form of, for example, thermal or electrical energy in a process to produce nanoparticles [13,14]. Chemical production routes often use reagents that reduce compounds to form nanoparticles while bio-based production routes generally use biological reducing agents such as bacteria, eukaryotic and prokaryotic organisms, or extracts from plants to synthesize nanoparticles [9,15-18]. Silver nanoparticles have been produced using these methods. However, a combination of different production techniques such as physico-chemical process has also been used to successfully produce nanoparticles [19,20]. Unfortunately, chemical synthesis is primarily the predominant production route, which may not be environmentally friendly and economically feasible for producing nanoparticles in large scale. This reinforces the need to explore alternatives that can overcome the current inadequacies related to the production of nanoparticles.

Silver nanoparticles have been incorporated into several products, including paper, due to their inherent antimicrobial properties [21]. However, the end-use of a product as well as the substrate properties may set restrictions on the usable nanoparticle production technique. Hence, it has been difficult to produce antibacterial paper with silver nanoparticles until recently. The

challenge of producing paper with silver nanoparticles was overcome using chemical reduction technique [22,23]. Techniques that have successfully incorporated silver nanoparticles on paper substrates typically expose the whole paper matrix for chemical modification. Therefore, the used process also alters the properties of the paper matrix, which may hinder the final product usability for certain applications. To overcome this challenge, there have been attempts to attach silver nanoparticles directly on the paper surface [24]. Currently, such direct methods on paper surface are not economically feasible and suitable for industrial mass production.

This study presents an alternative production method for silver nanoparticle production using liquid flame spray (LFS) technology [25]. This technique is suitable for direct deposition on paper surface, and it has successfully been used in the production of nanoparticles [26]. LFS is an aerosol process, in which a liquid precursor is atomized in the presence of hydrogen, oxygen and nitrogen, and then combusted in a flame that causes the precursor to undergo a nucleation process. The result is the formation of solid nanoparticles. A detailed description of the LFS process is given in literature [27,28]. Nanoparticles can directly be deposited onto substrates such as paper, ceramic, or textiles [29,30]. Recently, TiO_2 nanoparticle coatings were produced using LFS on paper resulting in a superhydrophobic surface [31]. The LFS has been used in a roll-to-roll process to produce nanoparticles at speeds up to 300 m/min, and it is possible to upscale this to higher production speed suitable for industrial environment [25,32]. Additionally, a large matrix of metal and metal oxide nanoparticles can be produced depending on the precursors used, which adds to the flexibility of LFS in the production different nanoparticles. LFS is used in this study as a dry synthesis technique to produce large area antibacterial surfaces that utilize silver nanoparticles. The silver nanoparticles produced are analyzed using a scanning electron microscopy (SEM) and an X-ray photoelectron spectroscopy (XPS) while the antibacterial properties are examined using Escherichia coli (E. coli, gram negative) bacteria as a test organism.

2. Materials and methods

2.1 Liquid flame spray coating (LFS)

A liquid precursor was prepared by dissolving silver nitrate ($AgNO_3$, 99.9+%, Alfa Aesar) into deionized water. Concentration of the silver nitrate solution was 250 mg/ml. The liquid precursor was then injected into a flame where decomposition and nucleation resulted in the formation of solid silver nanoparticles. Oxygen, hydrogen and nitrogen are used as the combustion gases in the flame pyrolysis process to produce the nanoparticles that are deposited directly onto paper surface. The precursor feed rate through the nozzle was 2 ml/min and the gas flow rates ($H_2/O_2/N_2$) were 20/10/5 l/min. The distance between the nozzle and the sample surface was 20 cm with paper substrates attached to a rotating carousel above the flame. As the carousel rotated, the paper passed across the flame and nanoparticles were deposited onto the paper surface. The paper was passed through the flame 3, 10 or 30 times to produce samples with different amount of silver nanoparticles. Subsequently, PE_30X represents PE paper coated through the flame 30

times. The line speed of the carousel was 50 m/min. Figure 1 shows an image and a schematic representation of the nanoparticle deposition process.

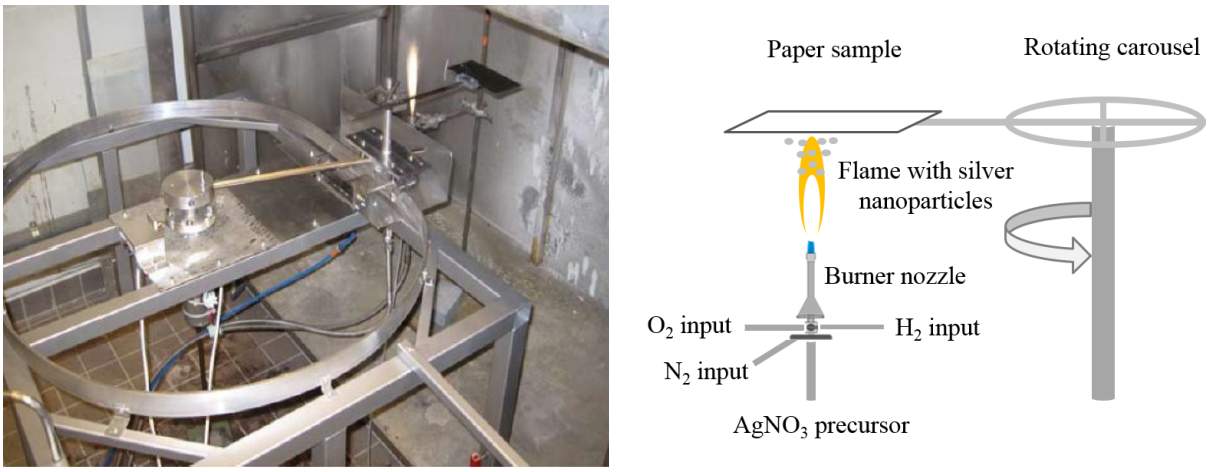


Figure 1: The setup and a schematic representation of the LFS process used to deposit silver nanoparticles on paper surface.

2.2 Paper sample description

Silver nanoparticles were deposited on four different paper substrates. Two substrates were precoated with mineral pigments whereas the other two were extrusion coated with plastic. Pigment coated samples were commercially available papers with grammages 200 g/m^2 (NAT) and 90 g/m^2 (LFP), obtained from Stora Enso Oyj. The plastic on paper was either polyethylene (PE) or polyethylene terephthalate (PET), both with thickness of approximately 20 µm.

2.3 Scanning electron microscopy (SEM)

The paper sample surfaces were imaged using an SEM (Jeol JSM-6335F) before and after coating with nanoparticles. An accelerating voltage of 2.7 kV was used with a working distance of 5-6 mm. The morphology and particle distribution on the paper surface was measured. ImageJ analysis software was used to quantify the particle size distribution from the SEM images [33]. The samples were sputtered with a thin carbon coating before imaging.

2.4 X-ray photoelectron spectroscopy (XPS)

XPS was used to determine the surface chemical composition of paper with silver nanoparticles. The amounts of different chemical constituents were measured in atomic percentage relative to the total atomic composition of the surface. XPS spectra were obtained using PHI Quantum 2000 (Physical Electronics Instruments, USA). Samples were irradiated with monoenergetic Al source through an aperture of 200 µm with a pass energy of 117.4 eV. The corresponding peaks in XPS spectra were used to identify and quantify silver.

2.5 Antibacterial testing

Antibacterial activity of silver nanoparticle coated PET paper samples was examined using *Escherichia coli* (*E. coli*) ATCC 25922 type strain as test bacteria. Samples examined for antibacterial properties are coated with 1X, 3X, 5X, 10X, and 30X flame sweeps. Antimicrobial testing was performed with modified replica plating method (T. Paavilainen et al. 2000) [34]. Bacterial colonies were diluted with 0.9 % NaCl solution to make bacterial suspension equal to 0.5 McFarland standard (approximately $1.5x10^8$ CFU/mL). This bacterial suspension was then pipetted on top of the nanoparticle coated samples and then samples were incubated for 24 hours at room temperature. After the incubation viable bacteria from paper surface were replicated by stamping sample on a blood agar plate. Plates were then incubated at +37°C and the colony forming units (CFU) were counted on the following day.

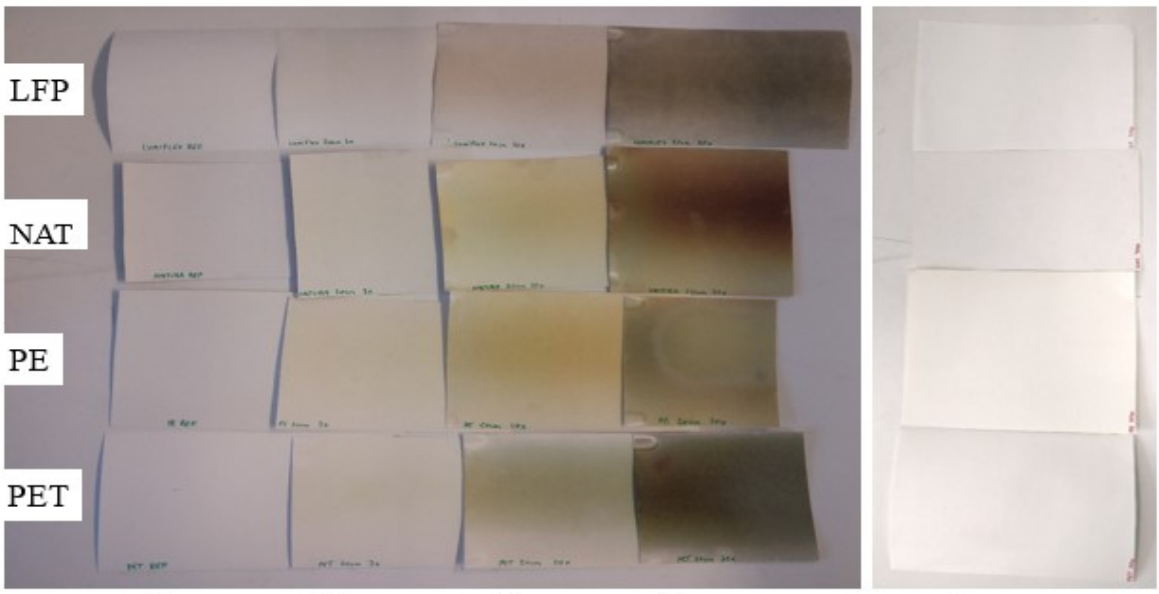


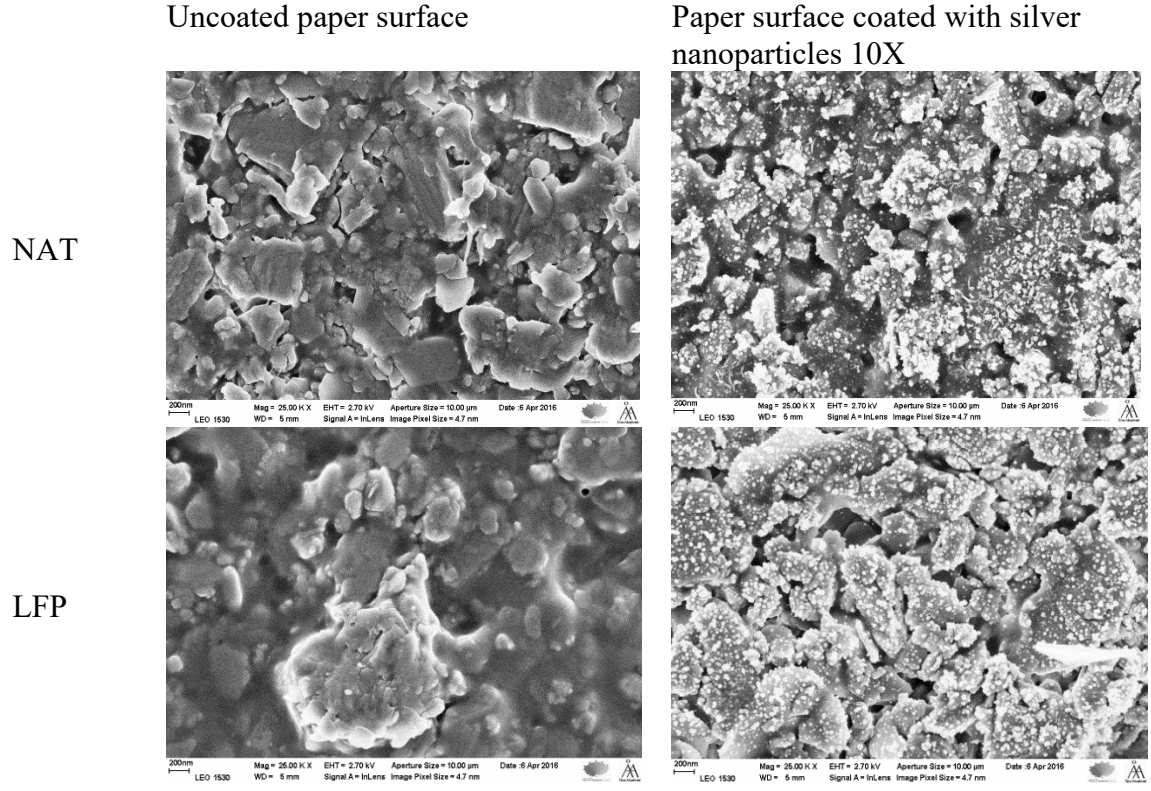
Figure 2: Paper samples after coating with silver nanoparticles using liquid flame spray. Each sample shows an uncoated reference, 3X, 10X and 30X in (a), (b), (c), and (d), respectively. Sample (e) shows paper samples treated with flame without silver nitrate in the precursor (30X). LFP and NAT are pigment coated paper samples. Paper samples precoated with polyethylene and polyethylene terephthalate are labeled as PE and PET respectively.

3. Results and discussion

In this study, silver nanoparticles were deposited directly on paper surface using the LFS process. Figure 2 shows paper samples with silver nanoparticles deposited using LFS in (b), (c), and (d). In figure 2 (e), paper samples are passed through a flame without silver nitrate in the precursor thirty times (30X), and no color change is observed. We can clearly see the difference in color induced by silver nanoparticles in comparison to paper samples that passed only the flame without silver nitrate in the precursor. This means that the used flame does not discolor the surface of paper samples. Hence, color change on the surface of paper samples is a result of silver NPs deposited onto the surface. It has been shown in literature that colloidal silver nanoparticles

have different colors based on the synthesis procedure depending on, for example, concentration and particle size distribution [35]. It has also been shown that a color change is observed as result of incorporating silver nanoparticles into films [36]. In this study, it is observed that the color change in the sample surface is dependent on the number of times samples are coated in the LFS. Samples coated three times (3X) showed less color change in the surface compared to samples coated thirty times (30X) as seen in figure 2 (d). The distinct color change also indicates that more silver NPs are deposited onto the surface for 30X samples. Therefore, the color change can be controlled by reducing the number of flame sweeps. It is noteworthy that the color change is particular to silver NP coatings since other nanocoatings produced using LFS do not show any color change even at 30X. Nevertheless, this color change could result in aesthetic properties that would be undesirable depending on the consumer preference.

Silver NPs have been incorporated into paper to produce antimicrobial filters both for air and water purification [37,38]. Using LFS it is possible to replicate these applications in a roll-to-roll process since the LFS nanoparticle deposition is done using a custom-built carousel that mimics a roll-to-roll process flow with a line speed of 50 m/min. The speed of LFS coating process can be scaled up to several hundreds of meters per minute. However, silver NP adhesion onto different paper substrates need to be studied before practical use can be implemented. The SEM images of paper surfaces before and after silver nanoparticle deposition are shown in Figure 3. Pigment coated paper shows the accumulation of silver nanoparticles as bright particles on the mineral pigments. Due to porosity of the pigment-coated surface, some nanoparticles can settle within the pores. The uniform coverage of paper by nanoparticles can be clearly seen on



PET

PE

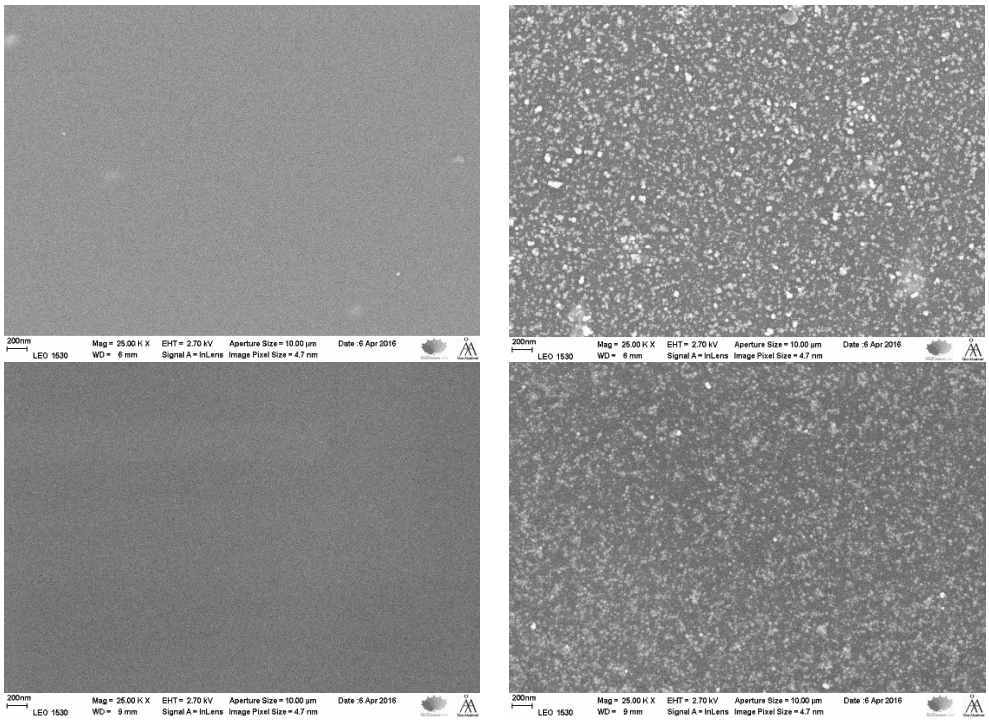


Figure 3: SEM images of paper surface before and after coating with silver nanoparticles using LFS. Silver coated paper surfaces shown here represent ten times flame sweep (10X) on the right and reference on the left.

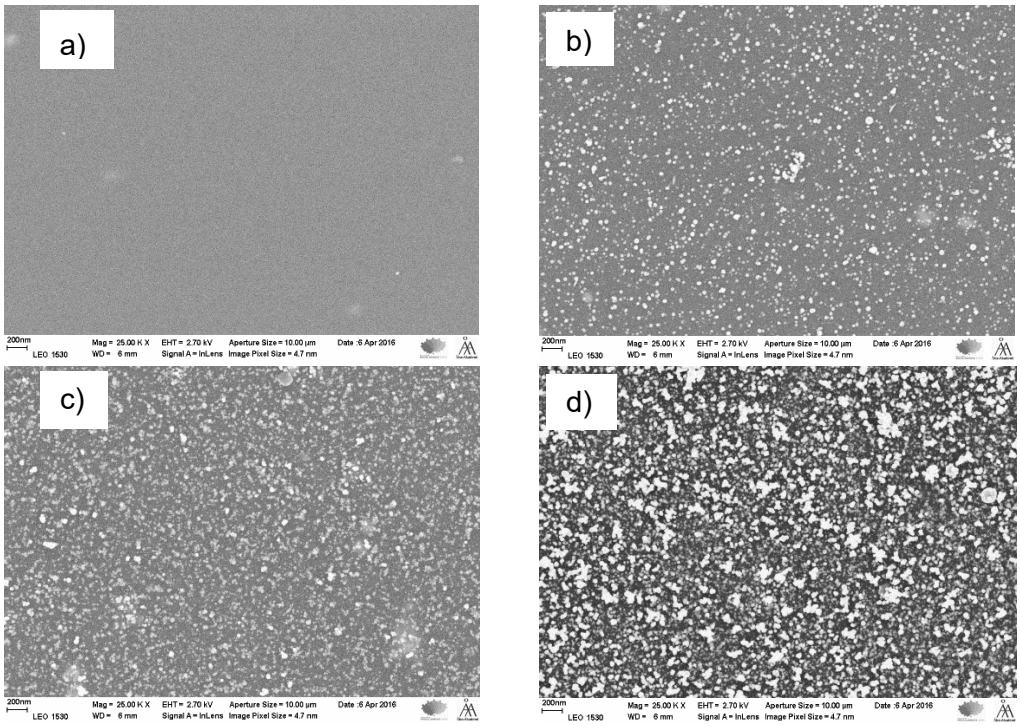


Figure 4: SEM image showing how nanoparticles on the surface increase as the number of flame sweeps is increased. a), b), c) and d) represent uncoated PET surface, 3X, 10X, and 30X flame sweep respectively.

the plastic-coated paper samples. The nanoparticles appear spherical in the SEM images. As expected, increasing the number of sweeps a sample passes through the flame increases the amount of deposited nanoparticles, as shown in Figure 4. The particle spacing in the monolayer of homogeneously distributed nanoparticles depends directly on the number of flame sweeps *i.e.* the area covered by nanoparticles increases with the number of sweeps. An exception is the sample PE_30X, which does not show silver nanoparticles distinctly on the surface. Image analysis of the SEM images reveals that the average particle diameter is approximately 30 nm. The particle size distribution and the frequency of occurrence are shown in Figure 5. Even though the particle size distribution was within a range from 10 to 100 nm, about 50% of particles on the surface have size less than 30 nm. The average particle diameter appears to increase with the increased number of flame sweeps as observed in Figure 4. This may result from nanoparticle agglomeration on the surface, which makes it difficult to distinguish individual nanoparticles from SEM imaging.

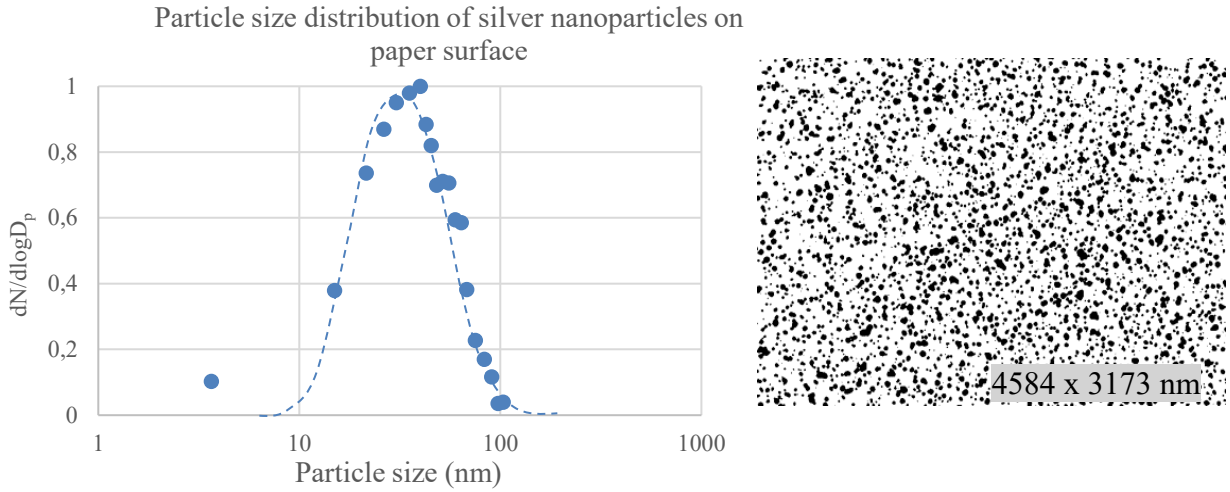


Figure 5: Size distribution of silver nanoparticles on paper surface. Left: the frequency of occurrence ($dN/dlogD_p$) as a function of particle size. Right: Processed SEM image of PE_5X sample obtained from ImageJ.

3.1 XPS results

XPS, which has a penetration depth of about 10 nm, was used to detect the presence of silver on the sample surfaces. A representative XPS spectrum in Figure 6 shows the difference between silver nanoparticle coated paper and uncoated paper surface. While there are several peaks that identify silver in the XPS spectrum, the photoelectron double peak that occurs between 365-380 eV is used to identify silver in the XPS analysis. The corresponding silver peaks are located at 368 and 374 eV with binding energy difference of about 6 eV. XPS was also used to quantify the amount of silver nanoparticles deposited on paper surface by measuring the silver amount in

atomic percentage relative to other elements present on the surface. Typically, for uncoated PE, over 50 % of the atomic composition on the surface was carbon.

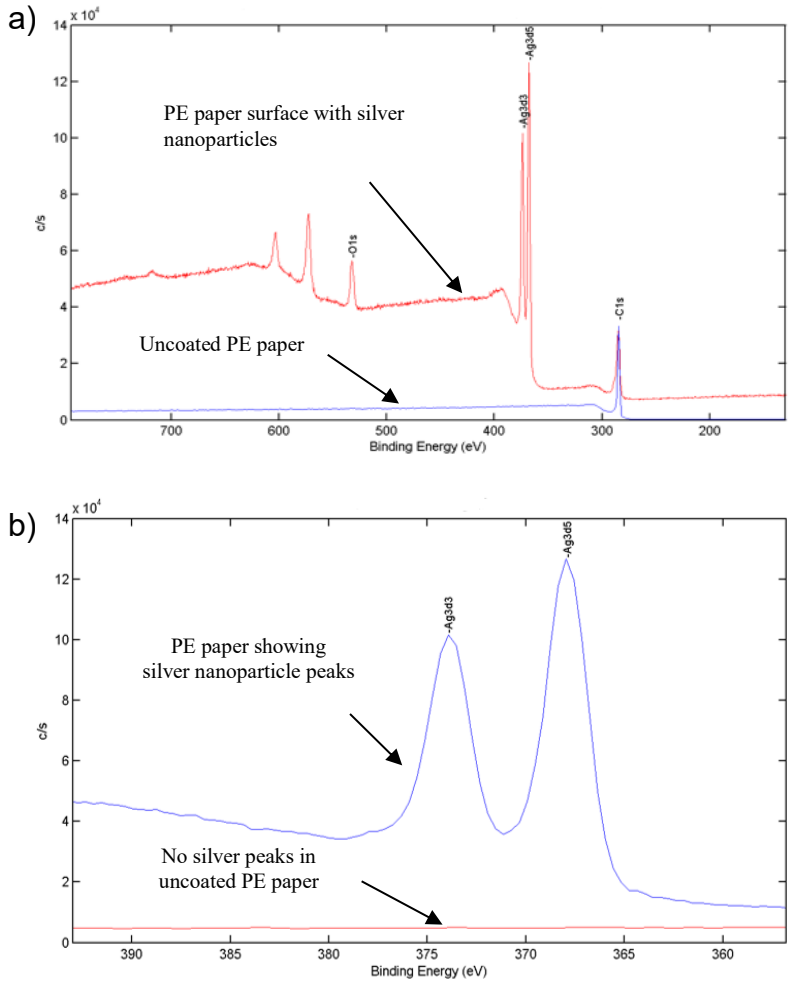


Figure 6: a) XPS spectra for PE paper surface before and after coating with silver nanoparticles. b) magnification of the analyzed silver peak.

This was largely due to the composition of the paper surface. Salts such as magnesium and sodium were present in quantities less than 1% after silver deposition but absent prior to deposition of silver. These salts may also originate from the constituents of the precursor used. The amount of silver measured on the surface depended on the number of flame sweeps, which is consistent with the SEM results. Figure 7 shows the silver amount measured using XPS for the different paper samples as a function of number of flame sweeps.

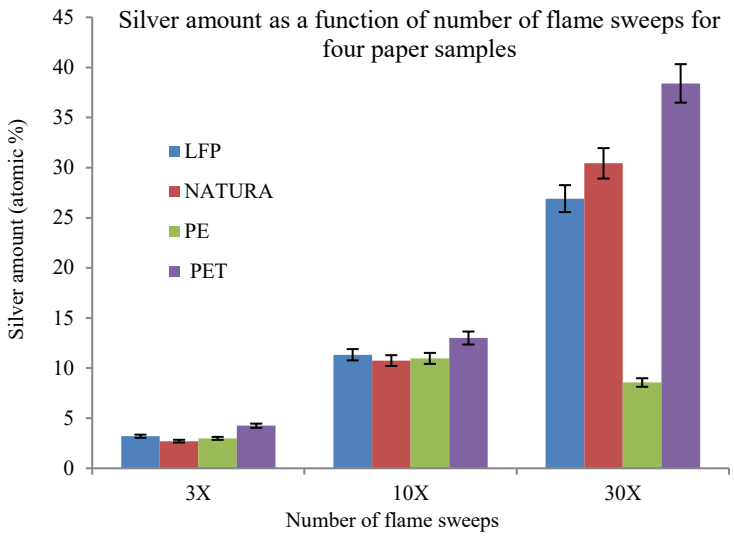


Figure **7**: Silver amount in atomic percentage on paper surfaces as a function of the number of flame sweeps measured with XPS.

Similarly, to the SEM images, quantitative XPS analysis shown in Figure 7 reveals a systematic increase of silver on sample surfaces as the number of passes over the flame is increased. 3X samples all contained 3.3 ± 0.4% whereas 10X and 30X had 11 ± 1% and 32 ± 1% silver, respectively. For 30X coated samples, a similar amount of silver nanoparticles was observed for all the paper samples except for the sample PE_30X. Precoated plastic paper surface appears to have slightly more nanoparticles on the surface compared to the mineral pigment coated papers. This can be due the porosity of the pigment coating allowing some nanoparticles to penetrate the coating layer beyond the detection depth of XPS. Unlike other paper samples, the SEM image of PE_30X does not show as much silver nanoparticles on the surface compared to the other 30X coated samples. XPS results also confirmed a reduction in the amount of silver nanoparticles on the surface for PE_30X. Particles appear to have penetrated into the structure and settled within the PE layer on the paper surface. This was observed as indistinct spots in the SEM image for PE_30X. Since the penetration depth for XPS measurement is only about 10 nm from the surface, silver nanoparticles that have penetrated beyond this depth would not be observed in XPS measurement. This can explain the low amount of silver nanoparticles seen on the surface of PE_30X.

3.2 Antibacterial properties

Silver nanoparticles have been extensively cited in literature for their antibacterial properties [39-41]. For antibacterial measurements, additional samples (1X and 5X) were prepared to obtain more information about the minimum amount of silver required to achieve antibacterial properties. Antibacterial activity of silver nanoparticle coated paper surface was demonstrated by counting of colony forming units (CFU). Paper surface with only PET surface coating was used as the reference, and it showed about 10^4-10^5 CFU of bacteria. After the LFS coating, PET_1X

which had passed through the flame once, showed less than 10^3 CFU of bacteria. However, no bacteria colonies were observed for the other coated samples. Paper samples coated using LFS show antibacterial properties against *E. coli* bacteria as shown in Figure 8. The minimum amount of silver required to achieve the antibacterial effect was not determined, even though 3X coatings and beyond had similar antibacterial effect. This suggests that coating silver in at least three flame sweeps may be enough to control E. coli considering the process parameters used. However, further investigation is needed to determine the least amount of silver nanoparticles that result in desirable antibacterial effect from LFS coatings either by reducing the number of flame sweeps or by modifying the initial concentration of silver nitrate precursor prior to flame deposition.

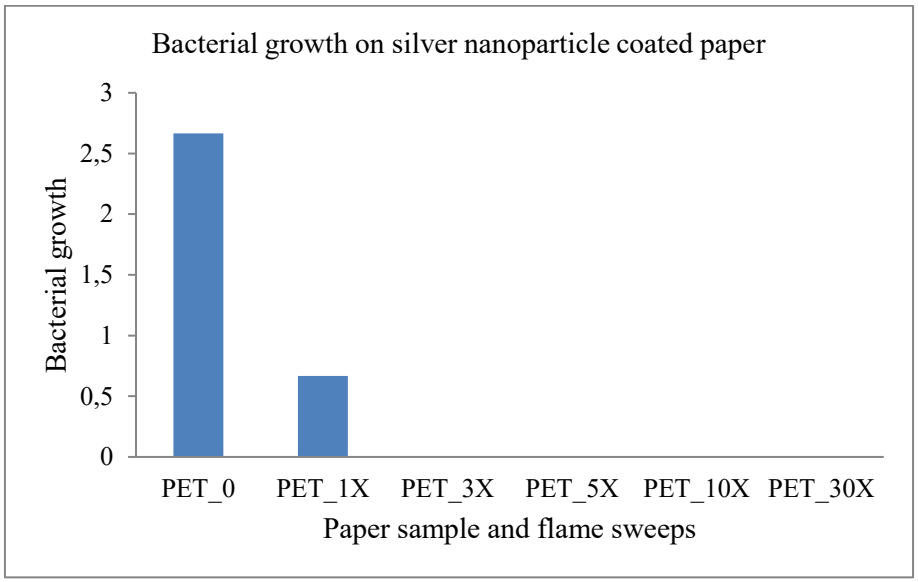


Figure 8: Antibacterial properties of silver nanoparticle coated paper against E. coli. Bacterial growth in the graph is given as 1, 2, and 3, corresponding to 10^3-10^4 CFU, 10^4-10^5 CFU, and $>10^5$ CFU, respectively. PET_0 is a reference sample with no silver nanoparticles.

3.3 Conclusions

Direct synthesis of silver nanoparticles onto paper substrate without subjecting the whole paper matrix to various chemical processes has been difficult to achieve over the years. In this study, we demonstrate a one-step deposition of silver nanoparticles on paper using a flame pyrolysis technique known as Liquid Flame Spray. While the LFS has been used to produce several types of nanoparticles, there are only few reports on the production of silver nanoparticles. The LFS process yields little or no effluents. Consequently, undesirable environmental impacts that result from effluents are considerably reduced. Nanoparticles are deposited onto the substrate in a dry process, which prevents alteration of the sample matrix. The LFS process also reduces restrictions for the substrate. Hence, it is possible to directly deposit silver nanoparticles onto large surface areas such as paper as shown in this study. The production process is flexible with

possibility for different metal and metal oxide nanoparticles simply by changing the liquid precursor used.

SEM and XPS results show that silver nanoparticles are successfully deposited on paper surface with an estimated average particle diameter of 30 nm. The amount of nanoparticles on the surface of paper can be effectively controlled by varying the number of times the substrate is passed through the nanoparticle producing flame. An advantage is also the up scalability of LFS to full industrial production scale utilizing roll-to-roll processing. The nanoparticles coated paper samples showed antibacterial properties against E. coli, which depended on the amount of nanoparticles on the paper. Nanoparticle adhesion to substrate is an issue that needs to be addressed, since the particle adhesion is mainly due to relatively weak van der Waals and electrostatic forces. Further studies will investigate various ways for improved adhesion or embedding of nanoparticles on surfaces. Potential applications of LFS generated silver nanoparticles include roll-to-roll fabrication of large area antibacterial surfaces that could be used, for example, in packaging or the textile industry. Production of antimicrobial filters is also possible using the technique presented in this study.

Acknowledgement

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