Advanced Powder Technology 28 (2017) 596–610

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Characterization and antibacterial properties of nanoboron powders and nanoboron powder coated textiles

Wazir Akbar [a], Mohamed Radzi Noor [b,d], Katarzyna Kowal [c,d], Tofail Syed [c,d], Tewfik Soulimane [b,d], G. Bahar Basim [a,e,*]

[a] Ozyegin University, Department of Mechanical Engineering, Nisantepe Mevki, Orman Sokak, No 34-36, 34794, Cekmekoy, Istanbul, Turkey
[b] Chemical and Environmental Sciences Department, University of Limerick, Ireland
[c] Department of Physics and Energy, University of Limerick, Ireland
[d] Materials and Surface Science Institute, University of Limerick, Ireland
[e] BioCapSOL Kimya, A.S., Teknopark Istanbul, Teknopark Blvd. No: 1, 34906, Pendik, Istanbul, Turkey

ARTICLE INFO

Article history:
Received 17 August 2016
Received in revised form 19 November 2016
Accepted 22 November 2016
Available online 18 December 2016

Keywords:
Nanoboron particle
Powder characterization
Antibacterial property
Nano-functionalized textiles

ABSTRACT

The antibacterial properties of boron-containing compounds are well known although there are limited studies available on the pure boron nanoparticles. In this paper, nanoboron particles are characterized in terms of their particle size, shape, stability and surface charge before and after their application onto textile surfaces to study their impact on bacterial activity. It was observed that the boron nanoparticles are effective in limiting the bacterial growth of both Gram-negative and positive species without requiring any stimulation to initiate the antibacterial action. In addition to the antibacterial functionality evaluation of the free boron nanoparticles, nanoboron coated textiles were also characterized and determined to change the wettability and surface charge of the textiles with a variable antimicrobial response to the different species. Consequently, we propose pure nanoboron as a new anti-bacterial agent that can function without external stimulation.

1. Introduction

The functionality of boron-containing compounds such as boric acid and its alkyl or aryl substitutes (borinic acid), borax, diazaborine and potassium tetraborate as antibacterial agents have been studied widely since they are commonly utilized as fertilizers, insecticides, detergent additives and buffers in pharmaceutical compounds [1–3]. As an example, potassium tetraborate nanoparticles applied to table grapes have shown a good antimicrobial activity and controlled gray mold formation caused by *Botrytis cinerea* both at room temperature and at 0 °C. The efficacy was positively correlated with the concentration of the solution [2]. High purity boron-nanoparticles, on the other hand, find applications in more advanced fields such as in semiconductor applications to produce p-type silicon and medicinal applications for neutron capture therapy and hence their synthesis and characterization became more important recently [4–6]. This study concentrates on characterization and anti-bacterial property evaluation of the boron nano-powders and the textiles finished with nanoboron powders in DI water and a standard finishing solution after their application as a coating.

Textiles have long been recognized as being prone to growth of microorganisms such as bacteria and fungi. These microorganisms may exist in the environment even at ambient conditions and can quickly grow when the suitable moisture, nutrient and temperature conditions are provided. The growth of microbes and bacteria on textiles during their use or storage not only degrades the performance of the textile itself but also negatively affects the public health. Most of the synthetic fibers, due to their high hydrophobicity, are more resistant to attacks by microorganisms than natural fibers [7]. The detrimental effects can be controlled by applying durable antimicrobial finishing to the textiles by using broad-spectrum biocides or by incorporating the biocide into synthetic fibers during extrusion [7,8]. However, while antimicrobial textiles provide the benefits of hygiene, odor control and protection of the fabric from microbial attacks, potential toxic breakdown products of the biocides are a concern environmentally as well as for the household. Most biocides used on the commercial textiles can develop bacterial resistance to the substances, which can lead to

* Corresponding author at: Ozyegin University, Department of Mechanical Engineering, Turkey.
E-mail address: Bahar.Basim@ozyegin.edu.tr (G.B. Basim).

http://dx.doi.org/10.1016/j.apt.2016.11.012
0921-8831/

increased resistance to certain antibiotics in clinical use [9]. Due to their small size and large surface, area antibacterial nanoparticles have also been investigated in this pursuit. Examples include zinc oxide (ZnO), carbon nanotubes (CNT), copper (Cu) and copper oxide (CuO), silver (Ag) materials and titanium dioxide (TiO_2) and nano silver [10,11].

One of the most critical uses of antibacterial textiles applies to medical applications. Hospital-acquired infections, known as nosocomial infections, are currently causing an epidemic-like condition affecting one in every ten patients [12]. The main route of infection is from the infected patient to healthcare professionals to an uninfected patient or visitor through inadvertent contacts with the surfaces of hands, furniture, walls, bed linens and upholstery. The death burdens from such infections are also high due to the ineffectiveness of the common broad-spectrum antibiotics including beta-lactam antibiotics. These infections are mostly caused by a limited number of bacterial pathogens such as methicillin/vanco mycin-resistant *Staphylococcus aureus* (MRSA/VRSA), methicillin-resistant *Staphylococcus epidermidis*, *Escherichia coli*, *Clostridium difficile*, vancomycin-resistant *Enterococci (VRE), Acinetobacter* sp. and *Pseudomonas aeruginosa* although MRSA and *E. coli* have been the most studied strains [13]. Accordingly, there is a need to either prevent bacterial attachment to these surfaces or *in situ* killing of the attached microbes. There are several commercially available antimicrobials such as triclosan, ammonium compounds, zinc pyrithione and silver.

Here we report the potential antimicrobial effect that can be obtained from nanoparticles of boron, termed as nanoboron. We have demonstrated that the solution of nanoboron particles has a high killing rate on both Gram-negative and positive bacteria while the killing rate is in particular effective on Gram-negative bacteria (*E. coli*) when applied on textiles under ambient conditions such as those in a typical hospital scenario.

2. Materials and methods

2.1. Preparation and characterization of the nanoboron particles and solutions

2.1.1. Preparation of nanoboron solutions

Boron nanoparticles with 99.7% purity and average reported particle size of 50 nm were obtained from NaBond Technologies Corporation, China. In order to apply the boron nanoparticles on the textile samples, solutions of the nanoboron powder were prepared at 0.002, 0.02 and 0.2 g/ 100 ml concentrations by dispersing the particles in deionized (DI) water as well as in the textile finishing solution. A standard textile finishing solution was prepared by using Setasil KF 1320 (amino functional silicon micro emulsion_cationic) Setas Chemicals, Serisoft 210 (non-ionic softener) Serboy Chemicals, Walline PE (polyethylene) Geochem Chemicals and acetic acid (Sigma- Aldrich for pH balance) at a total concentration of 2.7% (w/v) in DI water and measured pH of 3.52. In order to homogeneously disperse the nanoboron particles, the suspensions were prepared at pH 6 and kept on stirring continuously for 15 min. The prepared suspensions were tested for static stability, particle size and zeta potential after their uniform dispersion in DI water and the finishing solution.

2.1.2. Particle size and morphology analyses

The shape and size of the nanoboron powders were examined using SEM (Hitachi SU70) fitted with an EDS detector (Joel, Oxford Instruments). The powder sample was attached to the SEM stub using a double-sided carbon adhesive tape (Agar Scientific). A gold coating of 10 nm thickness was sputtered on the samples using a High Vacuum Sputter (Emitech K550), providing an optimal conductivity.

In order to analyze the particle size distribution of the powder sample measurements were performed via light scattering technique using Coulter LS-13320 Laser Diffraction Particle Size Analyzer (Beckman Coulter ALM-aqueous Liquid Module). In the process of particle size measurements, DI water at pH 6 was used as the background in the sampling cell to keep the particles stable during the measurement.

2.1.3. Particle crystalline structure analyses

In order to determine the crystalline structure of the nanoboron powders, X-ray diffraction (XRD) analyses were conducted by using a PANanalytical Epyrean diffractometer in a Bragg geometry using the CuKα radiation (λα1 = 1.5406 Å) in the reflection mode. Furthermore, nanoboron powders were examined using transmission electron microscopy, TEM (JEOL JEM-2100F) equipped in EDAX detector to visualize the crystal orientation after dispersing the powder in ethanol and exposing to ultrasound for homogeneous sampling on the TEM grid. The copper TEM grids with carbon film (Ted Pella) were dipped into the prepared solution and left to dry in air prior to analysis.

2.1.4. Surface charge determination of the nanoboron solutions

The zeta potential of the boron nanoparticle suspensions were measured by using Malvern Nano ZS Analyzer in DI water and in the textile finishing solution to determine the total electrical charge potential surrounding the particles to enhance their stability during their application on the textile surfaces.

2.2. Application and characterization of boron nanoparticles on textiles

2.2.1. Application of nanoboron solutions on textile samples

Solutions of boron nanoparticles were coated by dip-coating on a textile sample with 47 wt% polyester, 47 wt% viscose and 6 wt% spandex fiber composition. *Textile samples were initially treated with DI-water to remove any loose fibers to prevent deviation in the particle attachment analyses. They were kept at 36 °C for 24 h to dry followed by 36 h exposure to ambient to regain their natural moisture as a standard procedure after the prewash and nanoboron solution treatment.* The textile was chosen to be dark color to maintain the original color after the application of the nanoboron powder. Both suspensions prepared in DI water and in the finishing solution were coated by using the same methodology. The textile samples treated in DI water were dipped into 0.002, 0.02, 0.2 and 2 wt% nanoboron solutions in addition to the baseline DI water treatment without the particles. The samples treated in the finishing solution were treated only with 0.002, 0.02 and 0.2 wt% solution in addition to the pure finishing solution treatment since the 2 wt% solution treated textiles were observed to release particles in DI water treatment after the coating process. *In order to determine the amount of particle attachment in grams/m^2, the textiles were weighed with a high precision balance with 0.01 mg sensitivity after prewashing and drying in DI-water, and after the treatment with nanoboron solutions.*

2.2.2. Wettability measurements

To determine the surface hydrophobic nature of the textiles, contact angle measurements were performed on the baseline and coated textile samples by sessile-drop contact angle measurement technique by using KSV ATTENSION Theta Lite Optic Contact Angle Goniometer. Three readings were taken for each sample as a function of time intervals of 10 s up to 60 s.